ROMA NIR2025

LIGHT THROUGH CENTURIES

8-12 June 2025

Auditorium della Tecnica
(Rome, Italy)

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ABSTRACT BOOK



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PRECOURSES

P.01

PRACTICAL SAMPLING, NIR SPECTROSCOPY AND DATA ANALYTICS FOR PAT APPLICATIONS

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Workshop Synopsis:

Reliable analytical method development is the result of careful planning, qualified instrumentation and sound modelling. The practice of such development is described in guidance documents such as the international conference on harmonisation (ICH) Q2 [1] and American Standards for Testing and Materials (ASTM) [2]. There is one important aspect of near infrared (NIR) model development that is often overlooked; representative sampling. Fortunately, the theory of sampling (ToS) has provided many insights into how to solve the issues of poor sampling [3].

In particular, it has been shown that poor sampling can contribute errors between 10-50 times the total analytical error (TAE) of the analytical instrument [4]. The insertion of a probe into a process or the presentation of a specimen to a NIR spectrometer does not necessarily mean that a reliable result will be generated. With reference to regulated industries, guidance on 'Large N' sampling are provided by ASTM for ensuring that analytical predictions made by NIR analysers are not only reliable, they are representative. This is the sole reason why addressing sampling issues in the early stages is paramount in the development of analytical methods based on NIR (or other process analytical technology (PAT) system).

This workshop is presented by three world recognised experts in PAT all originating from the Pfizer Global PAT group. Brad, Phil and Steve will describe practical examples of the use of representative sampling systems along with NIR spectrometers for the development of robust, reliable and representative chemometrics models for use in at-line, online and inline applications. Participants to this workshop are encouraged to ask questions and group discussion is highly recommended.

References.

1.International Conference on Harmonisation; Pharmaceutical Development Q2(R1), I.E.W. Group, Editor. 2005: Europe.

2.ASTM Standard 2629 Standard Guide for Verification of Process Analytical Technology (PAT) Enabled Control Systems. 2020, ASTM International.

3.Esbensen, K., Introduction to the theory and practice of sampling. 2020, Chichester, West Sussex, UK: IM Publications Open. xiv, 323 pages: illustrations (chiefly colour).

4.Esbensen, K.H., et al., Adequacy and verifiability of pharmaceutical mixtures and dose units by variographic analysis (Theory of Sampling) - A call for a regulatory paradigm shift. International journal of pharmaceutics, 2016. 499 1-2: p. 156-174.

P.02

#NIR4 - HOW TO ANALYSE, STRUCTURE & HANDLE NIR RESULTS IN R RESILIENTLY

Zoltan Kovacs

Institute of Food Science and Technology, Hungarian University of Agriculture and Life Sciences (MATE), Budapest, HUNGARIA

Achieving reliable and adequate results from your NIR analysis efficiently begins long before the evaluation of the spectra. In addition to being an expert in various chemometric techniques, you must also know how to design experiments, structure, archive, and curate your NIR data to be effective in NIR applications. In this precourse, the major steps of NIR analysis will be covered with hands-on practical examples - from experimental design to saving and structuring the collected data as part of a larger NIR database - ensuring your data is instantly ready for effective data evaluation using the powerful R project and the aquaP2 package (Pollner & Kovacs, 2016). If you are interested in gaining practical skills in these areas, please ensure your computer is ready with R and aquaP2 installed, and join us for this exciting journey.

P.03

DETERMINING DETECTION LIMITS: KEY INSTRUMENT PARAMETERS AND SPECTRAL PROCESSING

Inagaki Tetsuya

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Near-infrared spectroscopy (NIRS) enables non-destructive, non-contact measurement of samples due to its inherently low molar absorptivity. However, the interpretation of detection limits in NIRS becomes complex as chemometric techniques are typically employed for analysis. Despite this complexity, as a spectroscopic method, the detection limit should fundamentally be determined by the molar absorptivity and noise contributions, including detector noise, photon noise, and modulation noise. This course will explore the concept of detection limits from this perspective. Additionally, understanding detection limits requires consideration of spectrometer configurations (e.g., Fourier transform vs. dispersive), spectral resolution, and wavelength interval. These aspects will be discussed in detail.

P.04

VARIABLES (FEATURES) SELECTION IN MULTIVARIATE CALIBRATION AND CLASSIFICATION MODELS USING NIR SPECTROSCOPY

Jean-Michel Roger

INRAE, Montpellier, FRANCE

This course will introduce participants to variable selection strategies in Near-Infrared (NIR) spectrometry, focusing on both the practical and interpretative motivations for reducing the number of spectral variables in multivariate calibration models. The session will begin with a discussion of the challenges inherent to NIR data - characterized by high dimensionality and strong collinearity - and will outline why variable selection can be useful.

Two major categories of variable selection objectives will be distinguished. The first concerns the optimization of calibration models when the number of available samples is limited, aiming to improve model robustness, reduce overfitting, and simplify final predictive tools. The second focuses on the identification of a small, relevant subset of variables to better understand the underlying physico-chemical or biological phenomena.

The first category will be extensively covered, presenting three main families of approaches:

- Filter methods, which select variables based on their individual statistical relationship with the target variable.
- Embedded methods, where variable selection occurs during model construction (e.g., variable importance measures in PLS).
- Wrapper methods, which iteratively assess variable subsets through predictive model performance.

The second category, less often addressed in the literature, will be illustrated through the presentation of the xx-CovSel family of methods, designed to identify a minimal set of variables explaining the covariance structure between spectral data and reference measurements. These methods are particularly well suited for exploratory analysis and the identification of spectral regions associated with specific chemical or structural characteristics.

By the end of the course, participants will have a clear understanding of the main strategies available for variable selection in NIR spectrometry, their appropriate applications, and the trade-offs involved.

AWARDS

A.01

"THE PRIDE AND THE PASSION BY NIRS": 36 YEARS OF DEDICATION TO LEARNING AND TEACHING IN NEAR INFRARED SPECTROSCOPY

Ana Garrido-Varo

Faculty of Agricultural and Forestry Engineering, University of Córdoba. ETSIAM, Campus Rabanales, Cordoba, SPAIN

This film, directed by the awardee, summarizes 36 years of dedication to learning, teaching, research, and technology transfer in Near Infrared Spectroscopy (NIRS). One of its main objectives is to share valuable insights and experiences from her R&D Group that are not typically published in indexed scientific journals but have had a significant impact on the advancement and funding of NIRS-related activities. The film aims to support industry professionals and laboratory practitioners in the challenging task of implementing NIRS successfully in real-world applications. Another key goal is to inspire young NIRS scientists and professionals to engage with that unique optical sensor- in combination with Machine Learning methods and Artificial Intelligence approaches- as pivotal in broader digital strategies to strengthen adaptive and resilient global food systems.

NIR sensors,Education,Research,Technology transfer,Machine Learning Artificial Intelligence, adaptive and resilient global food systems

A.02

UNMASKING THE PHANTOM: FROM MYSTERY TO MEANING

Marena Manley

Stellenbosch University, Stellenbosch, SOUTH AFRICA

Near-Infrared (NIR) spectroscopy was introduced to the wheat industry in South Africa in the late 1970s to early 1980s, following global trends in grain quality analysis. The Technicon InfraAlyzer was employed for rapid moisture and protein testing and particularly for quality grading and wheat cultivar evaluation. In the mid-1980's, as a first time NIR instrument user I was confronted by the 'Phantom in the Lab' a mysterious 'black box' where data went in and predictions came out. Soon, research began to unmask this Phantom and with the advent of chemometrics, the black box began to clear - first just a faint outline, then growing in transparency until it stood as a glass box. Understanding the 'why' behind the predictions - equally as important as the predictions themselves - became possible. Join me on a journey along the path of light and learning from those early days to where we stand today: with transparent, interpretable, integrated NIR science

OPENING CEREMONY

OL.01

BENVENUTI A NIR2025 - WARM GREETINGS FROM SISNIR

Monica Casale

University of Genoa, Department of Pharmacy, Genoa, ITALY

In her opening address, Prof. Monica Casale welcomes attendees to the conference on behalf of the Italian Society of NIR Spectroscopy (SISNIR), emphasizing the importance of collaboration, knowledge exchange, and interdisciplinary dialogue in the field of Near Infrared Spectroscopy.

She expresses her deepest gratitude to the members of the organizing committee, whose exceptional professionalism and meticulous planning have been instrumental to the successful realization of this event. Prof. Casale also warmly thanks SISNIR and its members for their continued commitment and active support of the Society's initiatives, which make gatherings like this possible. Further appreciation is extended to the sponsors for their generous support, to the invited speakers for their invaluable insights, and to all presenters, whose contributions enrich the scientific program and foster meaningful dialogue throughout the conference.
In closing, with sincere appreciation to all contributors and a special acknowledgment of the beauty of Rome as the host city, Prof. Casale invites participants to engage fully in both the scientific and social dimensions of the conference.

OL.02

THE HISTORY AND EVOLUTION OF NIR SPECTROSCOPY IN ITALY

Tiziana Cattaneo

CREA Research Centre for Engineering and Agro-Food Processing, Milan, ITALY

This presentation tells the story, the evolution and the growth of the NIR community in Italy. Starting from the first pioneers in the last century up to the current situation in terms of researchers, technicians, stakeholders, collaborators and disseminators.

The actions made for information; several schools dedicated to NIR technology and data processing; the involvement of instrumentation manufacturers; the cooperation of universities and research centres; the supervision of the Italian Society for NIR Spectroscopy have proved to be valid tools for a continuous growth and above all for the incessancy and increasingly widespread use of NIRS at both research and industrial level in our Country.

OL.03

THE PILLARS OF NEAR-INFRARED APPLICATIONS

Paolo Berzaghi

Vet School of the University of Padua, Padua, ITALY

A NIR application is a combination of multidisciplinary fields that involve knowledge on many different scientific areas. Any application stands on three fundamental "pillars": 1) Instrument/Sensor, 2) predicting model(s), 3) Sample preparation/presentation. Scientists and vendors are focused on the first two pillars, while sampling and sample presentation are often neglected and may be considered of lower scientific value. As scientist, we are committed to advance the performance of this technology, yet we have to make sure that transfer of knowledge to the industry also includes sample presentation and preparation. The presentation highlights some aspect of instrument trends and the importance of sample presentation.

KEYNOTE LECTURES

K.01

NIR SPECTROSCOPY ON MILK: FROM COMPOSITIONAL ANALYSIS WITH BENCHTOP INSTRUMENTS TO PHYSICAL PROPERTIES WITH SPATIALLY RESOLVED TECHNIQUES

Ben Aernouts

the Faculty of Engineering Technology of KU Leuven Campus Geel, Leuven, BELGIUM

As milk contains valuable information on the cow's metabolic status, regular analysis of the produced milk is an efficient way to monitor cow and udder health. Near-infrared (NIR) spectroscopy is a rapid, non-destructive and efficient technique that has proven valuable for on-line analysis of the raw milk composition. In the past 15 years, several prototypes for in-line milk analysis have been developed and validated, some of which are commercially available nowadays. Still, none of these sensors got ICAR certified so far, generally indicating that the accuracy, repeatability and reproducibility are insufficient. Based on our own 15-year experience, I will present and discuss the main challenges for in-line and on-farm milk analysers by reflecting results and sensor performances reported in the past against those obtained with our own research-prototypes for in-line milk analysis. These prototypes have been extensively tested at different dairy farms and under varying conditions over the past 8 years. Although the initial (calibration) accuracy of the prediction of milk fat, protein and lactose is largely depending on the signal-to-noise ratio of the sensor itself, the predictions afterwards are mainly subject to bias drift. Part of this comes from variation in milk temperature, which follows a seasonal pattern and can be accounted for by robust modelling. Additionally, unsupervised techniques and using bulk milk analyses results to correct for the bias can help and bring the sensor performance within the required specifications. Apart from predicting the milk composition from the NIR spectra, also the milk physical properties can be derived from the scattering present in the NIR spectra. To this end, my team develops and tests different spatially resolved NIR spectroscopy configurations to simultaneously measure the milk composition and fat globule size distribution in-line.

K.02

WHEN IMPERFECTION IS AN ADVANTAGE: NOISE-INDUCED STABILITY IN NIR BASED APPLICATIONS

Oxana Ye. Rodionova

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In mechanics, there is an interesting phenomenon called induced stability [1]. In short, it is a way of making an unstable system (such as an inverted pendulum) stable. To achieve this, small vibrations (actually noise) are added to the system. This little-known fact, however, is widely used in practice - for example, thanks to it we can ride a bicycle. In this talk, we will discuss how to use this principle for the practical implementation of the NIR spectroscopy.

There are many papers showing that NIR spectroscopy in combination with chemometrics is very promising for solving various practical problems. However, when it comes to the practical application for everyday use on multiple devices in different locations, numerous obstacles arise that hinder its implementation. It is generally accepted that various fluctuations are a source of disruption of stability and resilience, and therefore they must be somehow eliminated in order to make the system more perfect. Calibration transfer is a good example. On the contrary, based on the principle of induced stability, it is necessary to introduce into the system some controlled noise, the magnitude of which varies depending on the chosen goal.

The examples are taken from a real-life large-scale screening system, which is designed to detect counterfeit and substandard tablets and capsules using non-invasive NIR and chemometrics. In this framework, several levels of generalization can be considered depending on the ultimate goal. The most general task is to distinguish some specific remedy, say Amlodipine, from any other remedy that is not Amlodipine. In this case, we need to collect samples from as many Amlodipine manufactures as possible, ensuring the widest variability. At the second level, when we narrow the task and seek to distinguish Amlodipine from a particular manufacture from other manufacturers of the same remedy, we should collect samples from different batches of this manufacturer. At the third level, if the goal is to distinguish tablets from one particular batch from tablets from tablets from other batches, the training set should consists only of tablets from that specific batch.

It is obvious that at different levels, it is necessary to adjust the degree of variability of objects in the training sets. Less obviously, the NIR instrument and chemometric method should also be selected according to the level, as excessive accuracy may lead to less than ideal results.

Thus, in order to induce a stable, fit-for-purpose NIR system, it is necessary to adjust its degree of imperfection accordingly.

Reference

[1] Stephenson, A. (1908). XX. On induced stability. The London, Edinburgh, and Dublin Philosophical Magazine and Journal of Science, 15(86), 233-236. https://doi.org/10.1080/14786440809463763

K.03

UNLOCKING THE POTENTIAL OF SPECTRAL IMAGING THROUGH DEEP LEARNING

Junli Xu

University College Dublin, Dublin, IRELAND

This talk aims to showcase several case studies demonstrating how deep learning can enhance spectral imaging analysis. In the first case study, we present an innovative integration of deep learning and chemometrics for processing spectral images of fruits. The image processing tasks, such as object detection and recognition, are handled using deep learning, while the prediction of chemical properties is approached through chemometric modelling based on latent space analysis. The second case study focuses on leveraging deep learning to automate hyperspectral core scanning for geological applications. This approach integrates hyperspectral imaging with deep learning by introducing an automated analysis framework based on YOLOv8 (You Only Look Once), Segment Anything Model (SAM2), and Multi-Layer Perceptron (MLP). The framework is designed to identify and localize wooden boxes used for storing drill cores during hyperspectral image scanning, streamlining the analysis process. The third case study explores the application of deep learning for particle analysis in chemical imaging, specifically for microplastic detection. Deep learning models are employed to localize particles, automatically compute their morphological properties, and simultaneously identify polymer types, enhancing the accuracy and efficiency of microplastic analysis. In summary, spectral imaging is inherently data-rich, and its combination with deep learning significantly enhances its potential, making it a transformative tool across various applications.

K.04

EVALUATION OF AGRICULTURAL PRODUCTS USING HSI + 'X'

Inagaki Tetsuya

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NIR hyperspectral imaging (NIR-HSI) allows for the visualization of spatial distributions of chemical components in agricultural products. However, mere visualization does not fully utilize the potential of HSI. By integrating HSI with various analytical approaches, its effectiveness can be significantly enhanced. This presentation will introduce advanced methodologies that extend HSI applications, including reaction kinetics analysis with HSI, the combination of HSI with X-ray CT and finite element analysis, and the integration of HSI with convolutional neural networks (CNNs). These synergistic approaches will be demonstrated to highlight their potential in agricultural product evaluation.

FOOD & BEVERAGE 1

O.01.01

TARGETED AND NON-TARGETED APPROACHES FOR THE DETECTION OF PEANUT FRAGMENTS IN OTHER CHOPPED NUTS USING A HYPERSPECTRAL IMAGING SYSTEM

Miguel Vega-Castellote [1], María Teresa Sánchez [1], Moon S. Kim [2], Dolores Pérez-Marín [3]

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Unintentional allergen presence or cross-contamination in food products may arise during industrial production processes. The occurrence of peanut presence in other nut products arises in instances where various types of nuts are processed on the same food production line. This represents a significant food safety concern that must be addressed, given the potential health risks it poses to individuals with peanut allergies. In those cases, in which the nuts are chopped and marketed in small fragments, it is not possible to visually discern whether a given fragment is derived from peanuts or other types of nuts. As a result, there is a necessity to incorporate high-throughput methodologies into processing lines to facilitate the rapid and non-destructive differentiation of peanut fragments from small pieces of other nuts. Hyperspectral imaging technology integrates conventional imaging and spectroscopy methods, providing both spatial and spectral data from the examined product, which proves highly advantageous for assessing individual food items. The aim of this study was to evaluate a hyperspectral imaging system working in the 842-2532 nm range to identify small fragments of peanuts in chopped almonds, hazelnuts, and walnuts. To that end, two different methods were followed: A targeted and a non-targeted approach. For the former, Partial Least Squares Discriminant Analysis (PLS-DA) was used to develop discriminant models. For the non-targeted approach, the values of the spectral distances (calculated in principal components) of the peanut fragments tested against a standard sample of the other nuts, were used, following a methodology based on the Shewhart control charts. The targeted and non-targeted approaches classified 100 % and 97.7 % of the validation samples correctly, respectively. These results demonstrate that hyperspectral imaging technology, along with the proposed chemometric approaches, was a suitable alternative for identifying small peanut fragments in chopped almonds, hazelnuts, and walnuts.

ACKNOLEDGEMENTS: The authors gratefully acknowledge the United States Department of Agriculture (USDA) for the support offered to Miguel Vega-Castellote during his research stay from September to December 2023 and Spanish Ministry of Universities for the support offered to Miguel Vega-Castellote through the Training Programme for Academic Staff (FPU).

Keywords
Contaminant detection,Allergies,Food safety,Shortwave infrared,Control systems

O.01.02

NON-DESTRUCTIVE QUALITY ANALYSIS OF KOREAN FIR (ABIES KOREANA) USING FT-NIR SPECTROSCOPY

SangJun Lee [1], Fahri Reza Pahlawan Muhammad [2], Byoung-Kwan Cho [1,2]

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The Korean fir (Abies koreana), a critically endangered species native to Korea, is vital for ecological stability and cultural heritage. However, its population is severely declining due to climate change and environmental stress. This decline not only threatens biodiversity but also disrupts the ecological balance of forest ecosystems, where the Korean fir plays a key role. To support conservation and sustainable forest restoration, preserving high-quality seeds is essential. This study employed Fourier-transform Near-Infrared (FT-NIR) spectroscopy as a non-destructive method to evaluate qaulity of the Korean fir seed, ensuring the preservation of high-quality seeds.

FT-NIR analysis was conducted on seed samples categorized into quality-based groups: empty (E) vs. sound (S) and good (G) vs. not good (NG). Spectra were collected at four resolutions (4 nm, 16 nm, 32 nm, and 64 nm) within the 1100-2500 nm range. Balanced datasets were evaluated using PLS-DA, LDA, and SVM models.

FT-NIR achieved an average accuracy of 95.6% in detecting defective seeds (E vs. S), while the classification of G vs. NG reached 82.3% accuracy. Resolutions of 16 nm and 32 nm provided optimal performance, balancing precision and computational efficiency. Key chemical features, such as O-H and C-H bonds, significantly contributed to classification accuracy.

This study establishes FT-NIR as a reliable tool for non-destructive seed quality analysis, offering critical insights for conservation and forest restoration strategies. Future applications of FT-NIR could extend to other endangered tree species, supporting biodiversity preservation and sustainable resource management on a global scale.

Keywords
Fourier-transform Near-Infrared Spectroscopy,Nondestructive analysis,Seed quality,Tree seed

O.01.03

INVESTIGATION OF THE IMPACT OF FAT CONTENT IN GROUND BEEF ON PORK ADULTERATION DETECTION USING NIR SPECTROSCOPY

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KU Leuven, Leuven, BELGIUM

Ground meat, the main ingredient of various meat products, is vulnerable to adulteration with inferior or cheaper meat types. NIR spectroscopy has emerged as a rapid method for detecting adulterant (pork and chicken) in ground beef. However, most NIR spectroscopy studies used random samples or excluded fat from the meat, which can limit the applicability of the prediction models to real scenarios where the fat content varies across different samples. This research aims to study how fat content in ground meat can affect the accuracy of detecting pork adulteration using NIR spectroscopy. Three sample groups and two levels of fat content were considered: (1) ground beef with subcategories lean meat and lean meat mixed with 20% (w/w) beef fat; (2) ground pork with subcategories lean meat and lean meat mixed with 20% (w/w) pork fat; and (3) ground beef with 50% (w/w) pork adulteration with 4 subcategories being lean beef and lean pork, lean beef and lean pork with fat from beef and pork , lean beef and lean pork with 20% (w/w) beef fat, and lean beef and lean pork with 20% (w/w) pork fat. All samples were prepared in 15 replicates, resulting in 120 samples in total. Raw ground meat samples were measured in reflectance mode on a Bruker MPA FT-NIR instrument. Ground meat containing 20% (w/w) fat displayed peaks at 1728 nm, 1760 nm, 2310 nm, and 2345 nm, corresponding to C-H bonds. These peaks were found to be characteristic for pure fat and were absent in the lean meat spectra. Partial least square discriminant analysis (PLS-DA) was performed to distinguish adulterated pork-containing samples from non-adulterated pure ground beef samples. The PLS-DA accuracy of cross-validation trained on all samples showed a lower performance compare to a classifier trained on solely the lean samples. The addition of fat variation in the training set of the model can introduce additional complexity and may lower model performance, but may improve model robustness to differences fat variation.

ACKNOLEDGEMENTS: Researcher From the South scheme funded by KU Leuven

Keywords
Ground beef,pork adulteration,fat,NIR spectroscopy,PLS-DA

O.01.04

CLASSIFICATION OF HARD CANDIES WITH DIFFERENT FORMULAS BY USING HYPERSPECTRAL IMAGING COMBINING CHEMOMETRICS

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Confectionary industry focuses on hard candy production and their quality parameters. Citric acid plays a key role in controlling the acidic structure and flavor profile of the candy. Isomalt, helps prevent sugar crystallization, giving the product a clear and smooth texture. Additionally, the use of sugar alcohols offers lower calorie options when replacing sucrose and is beneficial for sugar-free candies[1]. The objective of this study is to explore Near Infrared-Hyperspectral Imaging (NIR-HSI) techniques, combined with chemometric modeling, can effectively predict hard candy samples in terms of citric acid concentration (%1.5, %2, %2.5), isomalt concentration (%92.5, %95, %97.5) and type of sugar alcohol (sorbitol, xylitol, erythritol) and enhance the quality of hard candy. NIR-HSI (Hyspex VNIR-1800) was employed in spectral range of 953-2500 nm. The analysis includes preprocessing steps like wavelength selection, Savitzky-Golay filter (15 windows, 2nd derivative), and normalization to improve data accuracy. In the modeling phase, k-Nearest Neighbors (kNN), Neural Networks, and Random Forest were employed to perform classification. Test set was randomly chosen to check the performance of each models on train set (3:7, test:train). Neural Network gave the highest average classification accuracy for citric acid concentration, isomalt concentration and type of sugar alcohol %95, %99 and %97 respectively for test set. On the other hand, kNN algorithm had the lowest average classification accuracy with %95, %92 %90. Results show that HSI can be utilized as a standard analysis method in hard candy production to not only determine the optimal formulation but also ensure the accurate identification and enhancement of quality parameters.

ACKNOLEDGEMENTS: Author gratefully acknowledge receiving funding from European Union's horizon 2020 research and innovation programme under grant agreement (#101008228)

Keywords
Hard Candy,NIR-Hyperspectral Imaging,Chemometrics,Quality Control

F.01.01

KIWIFRUIT UNDER STRESS: UNMASKING HIDDEN DAMAGE WITH HYPERSPECTRAL IMAGING

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The transition to Food Quality 4.0 represents change in how the agri-food industry ensures quality, safety and sustainability [1]. This concept exploits advanced, non-destructive technologies to meet the growing demand for efficient, real-time and data-driven quality assessment methods [2]. In this context, detecting the hidden effects of mechanical stress on fresh produce at an early stage is more than an economic issue, as it enables us to reduce food waste and optimise resource use.

Kiwifruit, a globally traded product, is particularly vulnerable to mechanical damage, which often remains invisible to the naked eye yet affects sensory and nutritional attributes [3]. These challenges highlight the need for non-destructive approaches to unmask and monitor such damage throughout the supply chain.

Mechanical damage was induced using a controlled setup, where a cylindrical metal weight (34.61 g) was dropped from a height of 1 m onto the base of each fruit. Three experimental conditions were established: undamaged samples served as controls, while other samples were subjected to a single or double weight drop to simulate different levels of mechanical stress.

Hyperspectral data were acquired with a short-wave infrared (SWIR) camera (Specim, Spectral Imaging Ltd., Oulu, Finland) at different time points to follow the evolution of mechanical damage over time. The spectral data were processed and analysed with HYPER-Tools 3 software [4].

The analysis of kiwifruits presented specific challenges due to their physical and chemical properties, including the brown colour of the peel, its opacity, and high-water content [5]. These factors can interfere with hyperspectral imaging analysis, necessitating additional preprocessing steps. Techniques such as derivative transformations and scattering removal were applied to improve the quality of the spectral data. Principal Component Analysis (PCA) was then conducted on the complete dataset, consisting of 56 NIR-HSI images. Based on these analyses, relevant wavelengths were identified to guide the development of a simplified multichannel system for mechanical damage detection.

Spectral analyses revealed clear patterns of moisture redistribution and tissue alteration, with spectral signatures evolving over time, particularly in the absorption band associated with water (around 1200 nm). These features were enhanced by applying a first derivative transformation prior to PCA. The exploratory analysis revealed that PC1 captured variations related to lighting conditions and fruit shape, whereas PC2 is associated with mechanical damage.

To quantify the extent of the damage, a threshold was applied to the PC2 scores, allowing the identification of damaged areas and the calculation of the percentage of affected tissue over time. Damaged tissues were absent in the control samples throughout the storage period, whereas the percentage damage in damaged kiwi samples increased over time.

The developed method has the potential not only to prevent damaged fruit from entering the fresh market, thus reducing waste and improving quality control, but also to facilitate alternative uses, contributing to a more sustainable and efficient food system.

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Keywords
kiwifruit damage,advanced methods,data analysis

F.01.02

PEA AND SOYBEAN PROTEIN DETECTION IN MINCED BEEF BY NEAR-INFRARED HYPERSPECTRAL IMAGING

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Vegetable proteins are introduced in meat products to boost certain properties, such as textural, nutritional and flavour profiles [1]. However, the minimal presence of these additives may be enough to create serious food allergies. Pea (PP) and soybean (SP) proteins are often added despite the labels not always declare the percentage of inclusion. In Spain, the content of vegetable protein in meat products is limited to a 3% [2]. Due to the difficulty to identify them, the objective of this study was to detect the pea and soybean protein addition in minced beef using near-infrared hyperspectral imaging (NIR-HSI).

For that purpose, a total of 120 beef patties of 20g were formulated. First, the meat pieces were minced with a domestic grinder, and afterwards, PP (n = 40) and SP (n = 40) were added into the grounded meat at proportions of 1%, 3%, 4%, and 6% w/w (no water added; 10 patties per formulation and protein). Pure beef samples were also considered (n = 40). All the samples were scanned using a hyperspectral imaging pushbroom system, composed of a transportation plate, an illumination unit (4 x 28W), and a Xeva-1.7- 320 camera (900 – 1,700nm; resolution of 320 x 256 pixels). Then, the images were stacked into 3-D hypercubes to perform black and white normalization. Dead pixels and spikes removal was also accomplished. The dataset was later imported into PLS_Toolbox 9.5. under MATLAB R2024a. Several pre-processing techniques were applied to the 2-D spectral matrix, including mean centering (MC), standard normal variate (SNV), multiplicative scatter correction (MSC) and, derivatives (1st and 2nd der), among others. Principal Component Analysis (PCA) was carried out to explore the spectral behaviour of the dataset while Partial Least Squares-Discriminant Analysis (PLS-DA) was used as a supervised classification approach. To guarantee the reliability of the models, the samples were divided into Train and Test in a ratio of 80:20.

From the PCA built with a combination of 1st der+SNV+MC and 4 LV, a clear separation among the 3 groups could be observed. Positive PC1 split the samples involving pea protein and the control samples vs. the soya patties. Also, PC2 was in charge of control (positive) and pea-based samples (negative) differentiation. For the discriminant analysis, two different strategies were carried out. First, the burgers were identified according to either the inclusion of protein or not. Secondly, the percentage of inclusion was considered. Focusing on the former, an excellent correctly classified (%CC) rate of samples according to the 3 main classifiers (control, PP, SP) was achieved (100 – 90%) in the Test. The PP and SP formulations were evaluated in 3 batches: (a) no protein added, (b) the contents below or equal to the legal limit (1% and 3%) and (c) patties above the regulation (4% and 6%). Overall accuracies of 100 – 80% were achieved for the different pre-processing methods. Furthermore, the VIP from PLS-DA and loadings of PCA exhibited the influence of protein addition in the beef burgers. This fact would explain the potential of NIR-HSI to classify the samples.

In summary, the appropriate discriminant rates achieved demonstrated how NIR-HSI could be implemented to pursue fraudulent practices and prove whether labeling is in compliance with the claims.

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ACKNOLEDGEMENTS: Funded by the Universidad Pública de Navarra through a 2022-PhD Scholarship.

Keywords
Meat,Vegetable protein,Hyperspectral imaging

F.01.03

ADVANCING BUTTER QUALITY ANALYSIS: A COMPARATIVE STUDY OF FOUR NEAR-INFRARED SPECTROMETERS ACROSS DIVERSE SPECTRAL RANGES

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This study evaluates the performance of four near-infrared (NIR) spectrometers—ISC, JDSU, FOSS, and Aurora—for analyzing key nutritional components (Moisture and fat content) in butter. Each spectrometer operates within a distinct spectral range: ISC (1100–1600 nm), JDSU (900–1650 nm), FOSS (850–2500 nm), and Aurora (950–1650 nm). These differences in spectral range provide a unique opportunity to compare the effectiveness and accuracy of each device for routine food quality assessments. Ninety-six butter samples were collected over a period of 12 months, at different plants located from the low land of the Po Valley, up to the higher altitude of the Alps. Each sample was scan in borosilicate glass and then in disposable plastic petri dishes. Spectra collected in plastic petri dishes showed distinct absorption peaks, overlapping those typical of fat showed The results for glass petri dishes showed variation in prediction accuracy across the instruments, particularly in relative prediction errors (RMSE/mean). Among the devices, FOSS and Aurora demonstrated the highest accuracy with RMSE around 0.5-0.6% and RSQ of 0.93. ISC and JDSU had lower performances that was similar between these two instruments, with RMSE around 1-1.15% and RSQ of 0.7-0.8. Despite the fact that spectra collected with plastic petri dishes had marked signal in the fat spectral region, calibration performances for Fat and Moisture had an increase in prediction error of 10-20% compared to the glass petri dishes. While the compact spectrometers (ISC and JDSU) exhibited reduced analytical sensitivity compared to the benchtop FOSS model, they performed sufficiently well for routine analysis of butter composition. These portable devices are cost-effective and practical for field-based or small-scale operations, where ease of use and portability are priorities. Aurora, being versatile in its spectral range, offers a middle ground between accuracy and practicality. On the sample presentation, the use of disposable plastic dishes greatly simplify routine work compared having to wash and reuse glass dishes, which, even with the slight increase of error, makes it easier to implement in routine analysis.

ACKNOLEDGEMENTS: We anknolege the great help of Dr. Federica Rela and Dr. Massimo Mirisola of LabCNX for their great support

Keywords
Butter composition,portable instrument,Laboratory instrument,sample presentation

FOOD & BEVERAGE 2

O.02.05

CLASSIFICATION OF MILK POWDERS UNDER DIFFERENT PRODUCTION AND STORAGE CONDITIONS USING NEAR-INFRARED SPECTROSCOPY

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Ensuring the quality of milk powders is critical due to their wide use and nutritional significance. Caking, a problem arising from factors such as moisture absorption, amorphous content transitions, and fat crystallization, significantly impacts flowability and quality. This study investigates caking tendencies in milk powders produced using various methods, including demineralized whey powder (DPST), whole milk powder (YT), and skim milk powder (YST), produced under varying flow rates (L/h), tank retention times (12 h and 8 h), and stored at different relative humidity levels. Near-Infrared (NIR) spectroscopy (NIRFlex N-500) was employed in the spectral range of 1100–2500 nm as a rapid, cost-effective and non-destructive solution for comprehensive quality assessment [1]. Preprocessing of NIR spectra involved mean centering and Savitzky-Golay smoothing with the first derivative. Chemometric techniques were applied, and classification models using k-Nearest Neighbors (kNN), Support Vector Machines (SVM), and Artificial Neural Networks (ANN) were developed. All models achieved high classification accuracy. The models were built using 10-fold cross-validation on the training set, and the SVM model achieved the highest performance with 97% accuracy on an external test dataset, underscoring the robustness of NIR spectroscopy combined with advanced data processing in identifying milk powder variations. Findings of this study demonstrate the potential of NIR spectroscopy as a reliable and efficient method for monitoring production and storage conditions, enabling enhanced quality control and product standardization in the dairy industry.

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ACKNOLEDGEMENTS: This research was supported by the TUBITAK 1001 project titled Toz Gidalarda Keklesmeyi Incelemek Icin Farkli Molekuler Yaklasimlar (ToKeMol) (Project No: 217O089), which focuses on the application of molecular techniques to investigate caking behavior in powdered foods.

Keywords
Milk powder,caking,Near-Infrared Spectroscopy,chemometrics,quality control

O.02.06

CONTROL OF HIGH MOISTURE EXTRUDATES TEXTURE IN THE COOLING DIE OF AN EXTRUDER USING NEAR-INFRARED SPECTROSCOPY

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Meat analogues are a popular, sustainable alternative to animal meat. During High moisture extrusion process (HMEP), alternative protein sources are submitted to high temperatures and pressures, resulting in high moisture extrudates (HMEs) with a texture that mimics animal meat [1]. Industries have used off-line analysis to determine the texture of HMEs, but these do not allow continuous process evaluation. In-line analysis is gaining interest as it could enable real-time monitoring, control, and optimization of HMEP.

This study aims to evaluate the feasibility of using Near-Infrared Spectroscopy (NIRS) coupled with conventional and machine learning algorithms to monitor HMEP within the cooling die using a NIR probe and improve the final HMEs' texture.

Soybean-based HMEs were produced using a laboratory twin-screw extruder at different cooling die temperatures (10 and 30 °C) and flow rates (10, 12.5, 16, 19.5, and 22 g/min). Temperatures and near-infrared spectra were acquired during the process. HME anisotropy was determined using instrumental texture analysis. Partial least squares regressions (PLSR) and convolutional neural networks (CNN) were used to predict HMEs anisotropy. Spectra were pre-processed and the impact of temperature variations in the cooling die was reduced using different filtering techniques.

Results showed that Orthogonal Signal Correction (OSC) [2] yielded the best results for eliminating temperature effects. Smoothing the spectra, on the other hand, seems to eliminate important information, as it reduces the predictive ability of the anisotropic index models. This could be because the scattering effects that allow the prediction of anisotropy are eliminated when the spectra are smoothed [3]. CNN improved the anisotropic index prediction compared to PLSR due to its capacity to find non-linear correlations [4] in the spectra associated with the impact of scattering, with a RMSEP of 0.0202, a R2 of 0.91, and a bias of -0.0011 compared to the PLSR model's RMSEP of 0.0343, a R of 0.86, and a bias of -0.0040.

In conclusion, these results show that NIRS can be used in the meat analogue industry to monitor and control the texture of HMEs in-line. The effects of temperature can be filtered using OSC pre-processing, but smoothing should be avoided because it seems to eliminate relevant information related to the scattering of light in the sample.

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Keywords
Meat-analogues,Alternative proteins,Process analytical technologie,Machine learning

O.02.07

RAPID, NON-DESTRUCTIVE PREDICTION OF RIPENESS OF PINK LADY APPLES BY USING NEAR-INFRARED SPECTROSCOPIC METHODS TO MONITOR FIRMNESS, SUGAR CONTENT, JUICINESS AND ACIDITY

Amandine Arnal [1], Léa Volmerange [3], Jean Brustel [4], Céline Verdier [1], Sylvain Gerbaud [1], Marielle Pages [4], Cecile Levasseur [2]

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Apples are one of the most widely consumed fruits in the world and are available year-round. In France, Pink Lady production has increased despite stable global production in recent years.

To meet consumer expectations in terms of quality, apples must be at optimum ripeness. Traditional destructive methods are currently used to measure physicochemical parameters. To avoid such destructive measurements, it has been shown in the literature that near-infrared (NIR) spectroscopy can predict fructose and glucose content in apple juice, as well as firmness, titratable acidity and sugar content in Fuji. The present study demonstrates the relevance of the MicroNIR spectrometer to address agricultural sustainability concerns. This compact device is easy to use in the field and allows non-destructive monitoring of the physicochemical characteristics of Pink Lady. The device acquires NIR spectra from different areas of apples, followed by standard analyses to assess these characteristics. Results indicate no impact on measurements across different quarters of the apple, though there is a slight impact between the median zone and the poles. Firmness is predictable with a 77 N threshold (using partial least square regression), and juiciness prediction is reliable, though a larger database could improve the model. Predictions for sugar content and acidity still need improvement, which would confirm the MicroNIR device's potential for assessing Pink Lady apple ripeness in the field.

ACKNOLEDGEMENTS: We would like thank ANRT (Association Nationale de la Recherche et de la Technologie) France (grant Cifre n° 1362/2023) for the funding.

We would like to thank all our colleagues who helped prepare the samples.

Special thanks to Marie Homs for her help in planning the protocols, for her expertise on the instruments and for her assistance with standard method analyses. Their invaluable contribution enabled us to build the database from which these results were obtained.

Keywords
NIRS,pink lady,ripeness,sustainable technique,PROBE

FS.02.01

ADULTERATION TESTING IN MILK POWDERS USING THE CONFORMITY TEST WITH A HANDHELD NEOSPECTRA SCANNER

Marcal Plans Pujolras, Adham Hesham, Maid Said

Büchi (Switzerland)

Screening for the presence of an adulterant in a material is an essential task for numerous businesses. Screening methods for a certain adulterant could fail as new adulterants could be used to pass the established screening method. Detecting untargeted adulterants in a known material is a real challenge. The spectral fingerprint of a material is a strong tool that can be used in this application. For example, dairy products are among the most widely consumed goods worldwide, making milk one of the most adulterated foods. Although urea (carbamide, CO(NH2)2) is a natural constituent of milk and is rich in nitrogen, it may also be used as an adulterant to mask the presence of bulk-added materials in milk powders. Since nitrogen is used to estimate the protein content of milk, urea is commonly employed as an adulterant.

The proposed algorithm compares a test sample to a predefined database of unadulterated samples and calculates the Conformity Index (CI). The CI is determined for each wavenumber by computing the confidence interval of the spectra in the database. The Conformity Index serves as a measure of compliance with the database, expressed in standard deviation units. This algorithm has two main advantages: i) no modeling is required, making the Conformity app a plug-and-play solution for novice users; ii) it does not assume specific spectral regions of interest, allowing untargeted analysis.

This study aims to analyze the limit of detection of the Conformity Index using NeoSpectra Scanner data for detecting urea adulteration in milk powder. A total of five milk powder samples were obtained from different commercial providers. The samples were divided into two main groups: reference samples (unadulterated) and test samples (adulterated with 0.15%, 0.6%, 1.25%, 2.5%, and 5% urea). Spectra were collected using five instruments, with one instrument designated for calibration and the remaining four used for testing. All measurements were conducted in reflection mode using NeoSpectra's powder kit. Each sample was measured four times, with each measurement lasting 3 seconds, and the results were averaged for analysis.

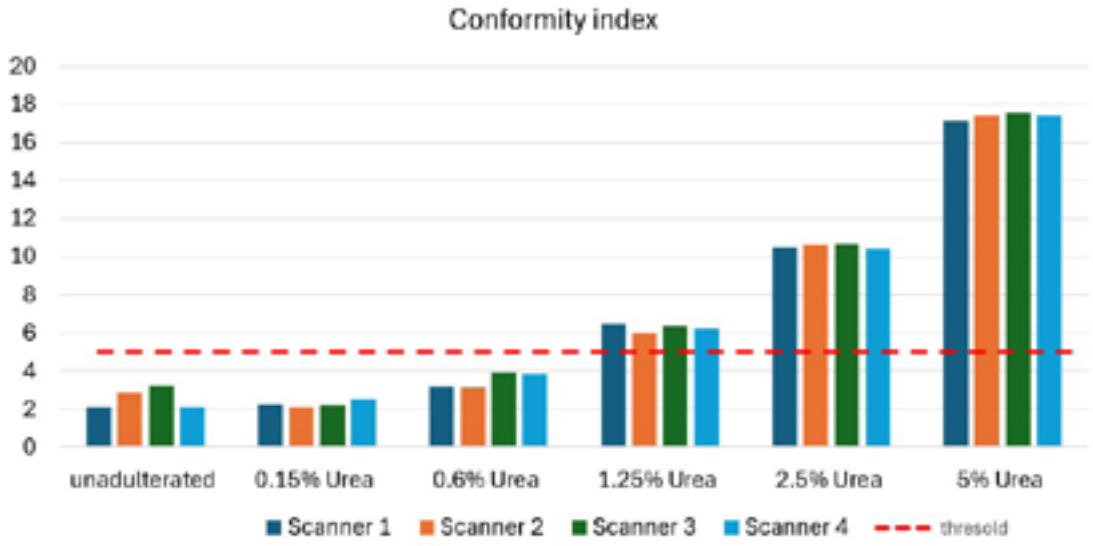


Figure 1: conformity index of urea adulterated milk powder samples.

The CI has demonstrated the ability to detect urea adulteration at 1.25% in milk powder. The standard CI threshold is set at 5 to avoid false positives. However, the threshold can be lowered to 4 to increase sensitivity, which may result in more false positives. The combination of the NeoSpectra Scanner and the Conformity Index algorithm has shown good sensitivity in detecting urea adulteration in milk powders.

ACKNOLEDGEMENTS: We would like to express our heartfelt gratitude to the company (Si-Ware) vice presidents (Dr. Mostafa Medhat, and Ahmed Badr) - for their invaluable contributions, expertise, and dedication throughout this project. Your relentless pursuit of innovation and excellence has been instrumental in driving success.

Keywords
fingerprint,Economic MotivatedAdulteration,NIR,Conformity Test

FS.02.02

ADVANCES AND APPLICATIONS OF MWIR HYPERSPECTRAL CAMERAS

Mathieu Marmion

Specim, Spectral Imaging Ltd, Oulu, FINLAND

The presentation will discuss the various advances and applications of the state of the art Specim MWIR hyperspectral camera, providing valuable insight into the capabilities of these solutions.

The capabilities of MWIR hyperspectral camera range significantly impact both research and industry, as they can detect materials that are undetectable by other traditional wavelengths up to 2500 nm.

The MWIR spectral range excels at detecting, for example, hydrocarbons, minerals, oil and glue contamination on different types of surfaces, and can even sort black plastics. Also, in this presentation, utilization hints related to this specific thermal spectral range will be provided.

The Specim MWIR hyperspectral cameras utilize push-broom imaging spectrograph, with transmission grating and on-axis optics. The camera is equipped with thermally stabilized optics and a cryogenically cooled MCT detector with a Stirling cooler.

ACKNOLEDGEMENTS: I would like to thank Minna Törmälä from Specim for her precious help in making this abstract

Keywords
MWIR,Hyperspectral,Sorting,Contaminants,Geology

FS.02.03

INNOVATIVE APPROACH TO POINT AND IMAGING SPECTROSCOPY USING A NEW COMMON-PATH INTERFEROMETER

Marta Ghirardello

NIREOS s.r.l., Milan, Italy

Point and imaging spectroscopy are powerful techniques used in the analysis of materials and biological samples to study the interaction of light with matter. Both techniques are widely used in fields such as material science, environmental analysis, medical diagnostics, and remote sensing, offering valuable insights into the molecular and structural characteristics of diverse materials. In this work we present an innovative approach to perform these measurements, based on the use of Fourier-Transform. In detail, we introduce our common-path interferometric technology, used in our TRICLOPS spectrometer and HERA hyperspectral cameras, and explore the different possible applications, from time and frequency resolved spectroscopy to hyperspectral imaging.

F.02.04

IDENTIFICATION OF BUTTER CHEESE ADULTERATION USING PORTABLE NIR SPECTROMETERS

Douglas Barbin [1], Adriano Lima [1], Monica Gonçalves [2], Helena Godoy [1], Maria Lucimar Medeiros [1]

[1] *University of Campinas, Campinas, BRAZIL,* [2] *Federal University of Campina Grande, Pombal, BRAZIL*

Food products with protected designation of origin or geographical identification have high value and are adulteration prone. Brazil is seeking to add value to its artisanal cheeses, thus its authentication is necessary. Due to a considerable lower price compared to benchtop instruments, handheld NIR spectrometers have potential for quality control in food industry. In this work, it was used a handheld NIR spectrometer as a non-destructive and low-cost alternative to identify adulteration in authentic and intentionally adulterated cheeses produced in the laboratory (70 samples) and commercial cheeses (12 samples).

NIR spectra were acquired at room temperature, using a portable spectrometer (DLPR NIRscanTM Nano, Texas Instruments, USA, 900-1700 nm). Fatty acids were determined to obtain the lipid profile of authentic cheese (non-adulterated with vegetable oil) and to verify the authenticity of commercial cheeses.

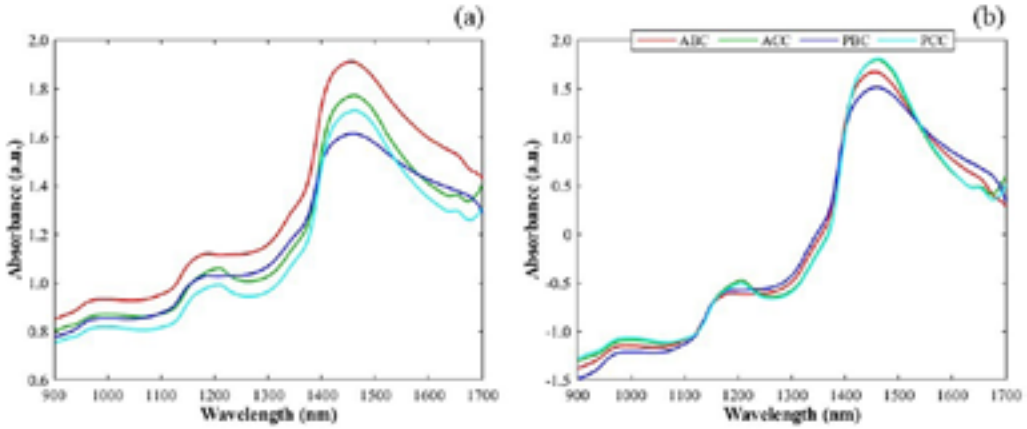
Partial least squares regression (PLSR) models were constructed with the full spectrum and optimal wavelengths to estimate the percentage of adulteration in butter cheese. The spectral dataset (referring to laboratory butter cheeses) was divided into training (70% of the data) and test (remaining 30%) using the KS algorithm (Kennard and Stone, 1969). The number of latent variables (LVs) was selected based on the lowest mean squared error of cross validation (leave-one-out) (RMSECV). Model performance and robustness were evaluated by coefficient of determination (R2) and RMSE for calibration (R_C^2, RMSEC), cross-validation (R_CV^2, RMSECV) and prediction (R_P^2, RMSEP). In addition, the ratio of the standard deviation of prediction data set to RMSEP (RPD) was calculated.

The mean absorbance spectra of authentic cheeses (Fig. 1) were similar to those previously collected in samples of cheese (Ozturk et al., 2022) and differed from adulterated cheeses only in the intensity of absorbance in specific spectral regions, especially at 970, 1130 - 1240, 1400, 1450, 1510 and 1660 nm.

PLSR models were constructed to predict of the level of soybean oil adulteration in butter cheese. In general, the PLSR models showed excellent performance to quantify the level of adulterant, with prediction errors (RMSEP) lower than 17% and high values of R2 and RPD. Models based on selected wavelengths (informative region and iPLS) performed similarly or better than models constructed with the full spectrum. The best model was obtained with the informative region (1050 – 1350 nm), without pre-processing, where an RMSEP of 7.202, R2 of 0.941 and RPD of 4.45 was obtained in the external validation. In addition, the pre-processing methods did not provide significant improvements in the predictive capacity of the models in relation to that obtained with the raw data, suggesting that both multiplicative and additive effects were minimal during spectral acquisition.

These results demonstrate that NIR spectra can be used to identify the replacement of authentic fat by soybean oil in butter cheese and that the developed models can be used to identify adulteration in external samples with good performance.

Fig. 1. Mean spectra of cheeses (PBC – Laboratory pure butter cheese, ABC – Laboratory adulterated butter cheese, PCC – Pure commercial butter cheese and ACC – Adulterated commercial butter cheese) (a) raw, pre-processed with (b) Savitzky-Golay smoothing combined with SNV.



ACKNOLEDGEMENTS: This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001 and São Paulo Research Foundation (FAPESP) (project number 2015/24351-2). Maria Lucimar da Silva Medeiros acknowledges scholarship funding from CNPq (300145/2022-5). Prof. Douglas Fernandes Barbin is CNPq research fellow (308260/2021-0).

Keywords
chemometrics,dairy,fraud,PLS,fatty acid profile

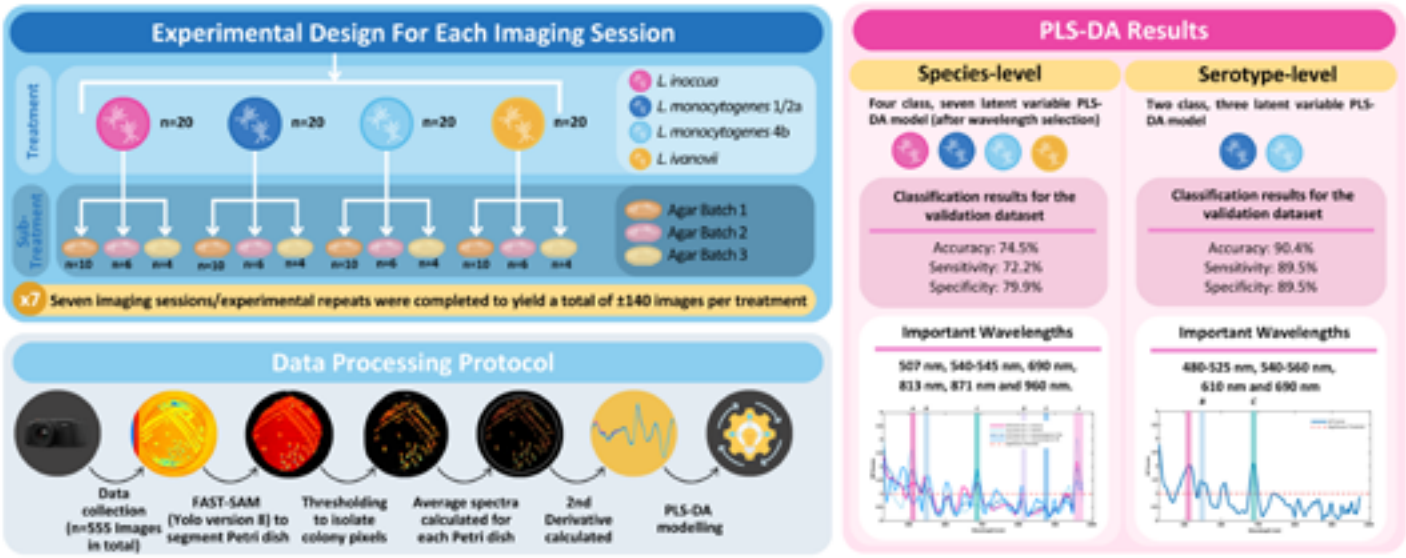
F.02.05

DISTINCTION BETWEEN LISTERIA SPECIES AND LISTERIA MONOCYTOGENES SEROTYPES WITH A HANDHELD, VNIR HYPERSPECTRAL IMAGING CAMERA

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Stellenbosch University, Department of Food Science, Stellenbosch, SOUTH AFRICA

A handheld, visible near-infrared hyperspectral imaging camera was used to differentiate between Listeria species (L. monocytogenes, L. ivanovii and L. innocua) and two L. monocytogenes serotypes: 1/2a and 4b. The results indicated that a portable hyperspectral imaging camera could effectively be used for pathogen detection and distinction applications, despite lower spectral resolution and higher sensitivity to noise. Five hundred and fifty-five hyperspectral images were collected from pure cultures on agar plates throughout seven sampling campaigns. Sixty-five percent of samples/isolates (n=±90 samples per class) were used to train Partial Least Squares Discriminant Analysis (PLS-DA) models and 35% of samples (n=±50 samples per class) were used for validation. An overall classification accuracy (validation) of 74.5% was achieved with a reduced wavelength model to distinguish between different Listeria isolates (L. monocytogenes 1/2a, L. monocytogenes 4b, L. ivanovii and L. innocua). Remarkably, L. monocytogenes serotypes 1/2a and 4b could be differentiated with an exceptional classification accuracy (90%) with a two-class PLS-DA model. Variable Importance in Projection (VIP) score plots were used to characterise valuable wavelengths in the visible range that were significant for discriminant analysis, for example, 690 nm was significant for the classification of L. innocua, whilst 871 nm was associated with L. ivanovii. Notably, all the significant absorption regions used to differentiate between the L. monocytogenes serotypes were in the visible range (480-525 nm, 540-560 nm, 610 nm and 690 nm) despite the colony morphology being virtually identical for these organisms on brain heart infusion agar.



ACKNOLEDGEMENTS: This work is based on the research supported by the National Research Foundation of South Africa (Grant Number: 137998).

The authors would also like to acknowledge the Harry Crossley Foundation for financial support to the student Jana Schreuder

Keywords
Portable hyperspectral imaging,Pathogen Detection,VNIR,PLS-DA,Wavelength Selection

F.02.06

ADULTERATION OF PAPRIKA SPICES: COMPARATIVE MULTISPECTRAL QUANTIFICATION OF AZO DYES BY NIRS, VIS-NIRS AND LIBS

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Institute of Analytical Chemistry and Radiochemistry, University of Innsbruck, Innsbruck, AUSTRIA

Food fraud and adulteration of herbs and spices remains a subject of great importance in analytical research. Paprika spice (Capsicum annuum L.) is particularly susceptible to falsification due to the high market value of premium quality paprika from specific protected designations of origin. Common adulteration methods include the addition of various red dyes or natural colorants to mask inferior quality and extend the shelf-life appearance. Azo dyes are the most common red food colorants found in adulterated paprika spice. So far, limited attention has been given to addressing this issue by using near-infrared (NIR) and visible-NIR (Vis-NIR) spectroscopy. The latter, in particular, offers a unique potential for the authentication of paprika containing different colorants, as the VIS spectral region is highly indicative to colored additives. However, current research in spice authentication did not step beyond standard analytical techniques. This study aims to achieve multispectral quantitative authentication of paprika spice. Spectral analysis was performed using a full-range handheld Vis-NIR (350–2,500 nm), a high-performance benchtop FT-NIR (1,000–2,500 nm) spectrometer, and laser-induced breakdown spectroscopy (LIBS). We evaluated three different azo dyes: two food colorants, Allura Red (E129) and Ponceau 4R (E123), and one non-food colorant, Orange II, which is potentially carcinogenic. Seventeen colorant levels ranging from 0.1% to 6% were assessed and divided into calibration and independent test sets prior to regression analysis. Results demonstrated that both FT-NIR and Vis-NIR are highly effective tools for the authentication of paprika spice, yielding comparable RMSEP values of 0.2-0.3% and R^2 values of 0.96-0.99. Notably, results derived by the handheld Vis-NIR spectrometer outperformed the benchtop FT-NIR spectrometer, providing similar regression performances but with more easily interpretable Partial Least Squares Regression (PLSR) models. LIBS analysis also delivered suitable regression models, albeit with inferior regression performance (RMSEP of ca. 0.7% and R^2 of ca. 0.86).

Keywords
near-infrared spectroscopy,paprika spice authentication,comparative multispectral quan,azo dyes

THEORY & FUNDAMENTALS

O.03.08

ANALYSIS OF NIR SPECTRA OF WATER BY COMPARING WITH THE CORRESPONDING IR SPECTRA

Yukihiro Ozaki [1], Mika Ishigaki [2]

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NIR spectra of water has long been investigated, however, they have not always been fully investigated. Although NIR bands at around 6900 and 5235 cm-1 are assigned to the combinations of v1+v3 and v2+v3 of water, respectively, detailed analysis of each band has not been explored. NIR spectra and their second derivative of water in the 9000-5500 cm-1 region consist of two bands at around 7090 and 6715 cm-1 due to weakly hydrogen-bonded (WHB) water species and strongly hydrogen-bonded (SHB) water species, respectively. It can be seen from the spectra that the former increases with the temperature increase while the latter decreases.

In the IR spectra in the 4000-3000 cm-1 region of water have two peaks at around 3400 and 3200 cm-1 ascribed to the OH stretching mode of WHB water species and SHB water species, respectively. Both SHB and WHB bands contain contributions from v1 and v3 modes, but it is difficult to resolve the contributions from both modes. As in the case of the NIR spectra the band due to the SHB species decreases with the increase in the temperature while that of the WHB species increases. Frequencies of both bands increase by about 10 wavenumber in the temperature increase from 20 to 60 degrees. On the other hand, the frequencies of the SHB and WHB bands in the NIR spectra shift only a few wavenumbers. It is rather strange point. We need deeper analysis of both IR and NIR spectra of water. One of the purposes of the present study is to explore the correlation between the NIR and IR spectra of water using two-dimensional correlation spectroscopy (2D-COS), which is powerful in investigating correlation between the bands.

Another important point in the NIR spectra is a board foot in the 6500-6000 cm-1 region. There may be one broad band there but its assignment is not uncertain. Another important point is comparison of the two water bands at 6900 and 5235 cm-1. Their band shapes are similar but the assignments are different. Nobody has tried to find correlation of these two bands. Thus, we also tried 2D-COS for the two regions.

Keywords
NIR spectra,IR spectra,Water,Hydrogen bonding,combination mode

O.03.09

HYDROGEN BOND STRUCTURE OF WATER IN CONCENTRATED SALT ELECTROLYTES AND IONIC LIQUID

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High-concentration aqueous salt electrolyte solutions have attracted attention as safe and inexpensive new electrolytes for secondary batteries. These electrolytes have different electrochemical stability and ionic conductivity from conventional aqueous electrolytes. Ionic liquids have also attracted attention as non-flammable electrolytes, but the control of their physical properties by adding small amounts of water attracts attention. In both cases, the factors that control the properties include the destruction of the hydrogen bond network of water and the resulting weak hydrogen bonds.

The vibrational transitions of overtones observed by near-infrared spectroscopy make it easier to study weak hydrogen bonds in detail than other vibrational spectroscopy methods. In this study, we used near-infrared spectroscopy to study the weak hydrogen bonds of water in high-concentration aqueous salt electrolyte solutions and water in ionic liquids.

Local hydrogen bonding environments of water in high-concentration Li-salt aqueous solutions were investigated using NIR spectroscopy. Absorption spectra of nu1+nu3 mode of water were measured for LiTFSI and Li(TFSI/BETI)aqueous solutions in the salt concentration ranges of 0.6-19 and 20-28 m.

As for the water in the ionic liquid, solutions were prepared in which the ionic liquid contained 0.1M water. The water/ionic liquid ratio was 0.5, 1, or 2, and the hydrogen bond state of the water was examined by near-infrared spectroscopy.

ACKNOLEDGEMENTS: This work was supported by Grant-in-Aid Scientific Research (C) (KAKENHI) from the Japan Society for the Promotion of Science (JSPS) grant number 21K05013.

Keywords
hydrate melt,ionic liquid,weak hydrogen bonding,electrolytes,multi curve resolution

O.03.10

THE ASSESSMENT OF THE GRAVITY EFFECT ON THE HYDROGEN BOND NETWORK OF WATER

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The water molecules form a complex network of hydrogen bonds, which render anomalous properties such as a high boiling point in spite of having low molecular weight, and the density of water reaches its maximum at 4 °C. Although the water molecule is one of the simplest and the most common molecules, the reasons behind the anomalous properties have not been fully elucidated yet. Furthermore, the era is expected to come soon when human beings venture into space in the near future. In low- or zero-gravity environment outside of the earth, it is expected that intermolecular interactions differ from those on the earth. Furthermore, the hydrogen bond network of water molecules will also be significantly different from that on Earth. Thus, the time has come when we need to seriously think about whether a living organism that contains a lot of water can maintain its healthy body functions under such conditions. Therefore, we aimed to study the dynamics of water molecules in a low-gravity/weightless environment.

NIR spectroscopy is a valuable method to analyze aqueous solutions. A band shift due to the formation of hydrogen bonds is much larger in the NIR region compared with that in IR region, of which feature can be cited as a strong point to investigate the variations in hydrogen bond network of water. In this presentation, the study of the assessment of gravity effect on the hydrogen bond network of water via NIR spectroscopy is introduced. The clinostat allows the sample to be rotated in three dimensions, distributing gravity evenly and create microgravity environment. By analyzing the NIR bands of aqueous solutions measured in this microgravity environment, the effect of gravity on the hydrogen bond network of water was evaluated. The results showed that the water behaves more freely water, and the hydrogen bond network changed towards disconnected. Further investigation is expected to determine how such differences in water dynamics affect the functions of living organisms.

ACKNOLEDGEMENTS: This work was supported by Japan Aerospace Exploration Agency (JAXA), RFP10(13)-05.

Keywords
NIR spectroscopy,hydrogen bond network of water,gravity

O.03.11

NEAR-INFRARED SPECTROSCOPY AS A TOOL FOR EXAMINATION OF STATE OF WATER IN ORGANIC SOLVENTS

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Water is the most common chemical compound on the Earth, but its unusual properties are not fully elucidated. In particular, the interaction between water and semi-hydrophobic molecules needs more attention. Even traces of water may significantly modify the properties of organic solvents. Hence, examinations of the interactions between water and the solvents are of great importance for science and technology. This presentation provides insight into the state and interaction of water in a series of aliphatic ketones [1], alcohols [2] and binary mixture of solvents. [3] Due to bulky aliphatic part, water has a limited solubility in all studied solvents, hence our interest was focused on the saturated solutions. Selection of solvents of different size and structure permitted us to examine the influence of various structural motives on behaviour of water in the mixtures.

As a research tool, we selected NIR spectroscopy since it provides highly specific information on the state of water at a molecular level. The concentration and temperature-dependent NIR spectra were recorded on Thermo Scientific Nicolet iS50 spectrometer in variable-temperature quartz cells (Hellma) of pathlengths from 1 to 5 mm. The spectra were analysed using Omnic, Grams/AI and Matlab/PLS-Toolbox software. For analysis, we applied the classical methods, two-dimensional correlation analysis and the chemometric methods. The water content in the saturated solutions was determined by Karl Fischer titration.

Our results reveal that the solubility of water in ketones and alcohols depends mainly on accessibility of the functional groups (C=O, O-H), while the other factors are less important. Conformational flexibility of aliphatic chains allows for effective shielding of the C=O or O-H groups in the linear compounds, and is the main reason of poor solubility of water in these solvents. On the other hand, the compounds with the cyclic aliphatic chains have limited conformational freedom, and as a result, the functional groups are less protected against the interaction with molecules of water. Our results evidences that the state of water strongly depends on the solubility. Due to poor solubility and low water content, in solvents with linear structure, molecules of water are involved mostly in solvent-water interactions, while the water-water interactions are of minor importance. In contrast, better solubility of water in the cyclic compounds enables creation of clusters, where the molecules of water are in water-like environment. A quantitative analysis of NIR spectra has shown that the molar absorptivity of the v2+v3 combination band from water has a constant value (290 [dm3*mol-1*cm-1]) in a series of aliphatic ketones [1], alcohols [2] and esters. [4] Since this band is located in the spectral region free from absorption of the other bands (5300-5100 cm-1), its intensity one can determine with a high accuracy. Therefore, the v2+v3 combination band has a great potential for qualitative and quantitative studies of water in organic solvents.

Keywords: NIR spectroscopy, hydrogen bonding, water, organic solvents, solubility.

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[3, 4] Unpublished results.

ACKNOLEDGEMENTS: This work was supported by statutory fund from University of Wroclaw.

Keywords
NIR spectroscopy,hydrogen bonding,water,organic solvents,solubility

F.03.07

UNRAVELLING LIGHT-MATTER INTERACTION OF COMPLEX FOOD PRODUCTS TO IMPROVE THE ROBUSTNESS OF NEAR-INFRARED SPECTRAL MEASUREMENTS

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SINTEF and Nofima have previously developed novel NIR instrument prototypes for non-contact, non-invasive, sub-surface quality measurements of complex food products based on chemical composition, including the benchtop high-performance SmartSensor and the more compact and portable MiniSmartSensor. The interactance geometry in these instruments relies on a certain degree of light scattering to transfer light from the field of illumination to the field of detection. In some applications, however, the NIR spectral variation for complex, structured, and highly scattering samples is dominated by physical properties rather than the chemical information. Such variations are commonly removed from the spectra through pre-processing, to emphasize the chemical information for analysis and minimize signal interference caused by production processes or environmental conditions. However, there are examples where physical variation correlates with the quality variable of interest and should not be removed, as doing so would reduce the accuracy of the prediction model.

Care must be taken to ensure that the inclusion of signal changes due to physical variations in the sample leads to robust and reliable prediction models. It is important to have a fundamental understanding of how the sample's physical properties affect the measurements, how its optical properties relate to quality attributes, the mechanisms causing correlations with the variable of interest, and light-matter interaction. This helps us determine the limitations of the model, which is important for trustworthiness in commercial applications. It also provides a starting point for developing models that incorporate physical information in a robust way.

In our talk, we will present our approach for understanding NIR spectral measurements in complex, structured, highly scattering samples, using dried salt-cured cod (clipfish) as a case study. We measured the sample's bulk optical properties (BOP), i.e. the scattering and absorption properties, using a double-integrating-sphere (DIS) setup and the inverse adding-doubling (IAD) algorithm. These lab measurements were compared with industry-suitable NIR interactance measurements to understand which variations are caused by the bulk optical properties and which can be explained by other influences. The corresponding light propagation model helped identify limitations and improve the robustness of the prediction model. Specifically, we investigated the effects and robustness of various optical geometries, gained an understanding of the mechanisms that cause correlations with quality attributes, and explored how sample properties of interest affected the signals and prediction model accuracy in relation to unwanted sources of variation. The results demonstrate how a fundamental understanding can improve the commercial robustness of spectroscopic measurement solutions for complex samples.

Keywords
Fundamentals,Robust instrumentation,Complex samples,Optical properties,Optical sampling

F.03.08

CONCENTRATION-DEPENDENT NEAR-INFRARED SPECTRA OF BINARY LIQUID MIXTURES INCLUDING WATER

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Concentration-dependent near-infrared spectra of binary liquid mixtures were automatically measured by using a spectrometer combined with a gradient mixer for liquid chromatography. A total of 101 spectra was measured during the mixing process of the binary system from 0-100 vol% every 1 vol% increments. In the present study, the effect of aprotic organic solvent addition for the first overtone of the O-H stretching of water was investigated. A higher wavenumber shift of the first overtone of the O-H stretching was observed for the addition of acetonitrile to water, while a lower wavenumber shift was observed for the addition of pyridine. It should be noted that no spectral contribution of the aprotic organic solvent was observed in the first overtone of the O-H stretching region. The spectral waveform variation of the first overtone of the O-H stretching was analyzed by means of multivariate curve resolution (MCR) assuming two contributions, one from water molecules hydrated by the other water molecules and the other from those solvated by the aprotic organic solvent molecules.

ACKNOLEDGEMENTS: This work was supported by JSPS KAKENHI Grant Number JP24K08531.

Keywords
binary liquid mixtures,water,aprotic organic solvent,concentration dependence,multivariate curve resolution

F.03.09

FINDING NEAR-INFRARED SPECTRAL PATTERNS CHARACTERISTIC OF GLYCOLYSIS - A COMPARISON OF HUMAN AND YEAST

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NARO, Tsukuba, JAPAN

[Introduction and Object] NIR spectroscopy has been expected to be used for non-invasive monitoring applications of living organisms. We have tried non-invasive human blood glucose measurement by NIR spectroscopy and identified wavelengths linked to blood glucose levels [1]. However, these wavelengths are not due to glucose itself and are probably due to metabolites related to glycolysis. In this study, we focus on glycolysis of yeast extracts to simplify the metabolic pathway. The yeast extract is not alive, but glycolysis proceeds by enzymes and, more interestingly, the concentrations of metabolites and coenzymes show oscillatory responses. The glycolytic oscillation of yeast extract has been extensively studied using mathematical models, and the phase relationships among the key metabolites have been estimated [2]. This study uses this phase relationship to identify the NIR spectral patterns of metabolite groups in typical glycolysis.

[Materials and Methods] Baker's yeast (Saccharomyces cerevisiae) was crushed and extracted with water. Transmittance spectra of the extract were acquired alternately for one sample using a VIS-NIR spectrometer (XDS near-infrared Rapid Liquid Analyser, FOSS) and a UV spectrometer (HR4000CG UV NIR, Ocean Optics). The extract was measured every 90 seconds for 2 hours immediately after the addition of trehalose. The temperature was maintained at 25.0 °C during the measurements. A part of the extract was used to track the reaction by a 1H-NMR spectrometer (AVNEO 800, Bruker) every 65 seconds.

[Results and conclusions] The 1H-NMR results showed approximately 16.4-minute periodic oscillations in the concentrations of ATP, ADP, and Glucose/Glucose-6-phosphate. Furthermore, phase differences between metabolites were generally consistent with previous studies. The major changes in the NIR spectra are monotonic, representing ethanol production from trehalose. To extract the small metabolite signal about the glycolytic oscillations hidden in the major signal, the difference spectra, A[t]-A[t-1], were calculated for each time step. In addition, MCR-ALS (Multivariate curve resolution- Alternating Least Square) was applied in the 750-1050 nm range and two oscillating components were identified. The scores of the resolved components oscillated with a period of about 18.5 min, which is consistent with the period of NADH observed at 340 nm. The loading of component 1 was strongly influenced by water with a peak at 970 nm. This reflects the hydrolysis of ATP to obtain energy for glycolysis. The number of molecules involved in the hydrolysis was estimated using extended molar absorption [3]. The loading of component 2 showed peaks at the wavelengths found in the human blood glucose experiments. Based on these results, we may have identified universal spectral patterns of glycolysis across species.

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ACKNOLEDGEMENTS: This work was supported by JSPS KAKENHI Grant Number JP23K26687.

Keywords
Yeast extract,Glycolytic oscillation,Metabolites

AQUAPHOTOMICS

O.04.12

20TH ANNIVERSARY OF AQUAPHOTOMICS

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Aquaphotomics, introduced in 2005 at the 12th NIRS International Conference in New Zealand, explores the crucial role of water in Near-Infrared Spectroscopy (NIRS). Water has been long neglected as the water absorbance is very strong and other molecules of interest absorbance is comparatively low in this wavelength range. Despite water's strong absorbance overshadowing other signals, pioneers like T. Hirschfeld, M. Iwamoto, and Y. Ozaki noted NIRS could detect components lacking NIR absorbance, raising questions about water's role.

While using NIRS for disease diagnostics in mammals, it was very easy to identify the sick animals, but very difficult to develop a model for milk protein measurement. It became clear that water is related to disease and water's impact was key, revealing its immense significance and providing complementary insights into NIRS. In many biological and aqueous systems experiments repeatedly observed high variations at specific water bands under the 1st, 2nd and 3rd overtones. In Aquaphotomics they were linked to different water molecular structures, aligning with Infrared Spectroscopy findings and presented as spectral patterns (aquagrams) related to respective water functionalities.

In Aquaphotomics, consecutive spectral measurements of the same sample are used to activate water's molecular network. Light frequencies alter water's structure, and their scanning sequence impacts spectral data and model accuracy. New type of spectrophotometer was developed highlighting these findings.

Environmental changes like temperature, humidity and pH introduce perturbations, to which water is highly sensitive, enabling correlations between absorbance (OD) and the logarithm of these changes. As Giuseppe Vitiello explains, linearity in log-log plots indicates self-similar fractal structures and system coherence. Thus, Aquaphotomics showed that NIRS is a powerful tool for studying biological systems, including brain dynamics and consciousness.

Join us in celebrating 20 years of Aquaphotomics at the 5th Aquaphotomics Conference, May 17–20, 2025, Kobe University, Japan.

ACKNOLEDGEMENTS: References: G. Vitiello, New Mathematics and Natural Computation 5, 245 (2009); Phys. Lett. A 376, 2527 (2012)

Keywords
water molecular structure,water spectral pattern,aquagram,biological and aqueous systems,new spectrophootometers

O.04.13

MONITORING ALTERATION IN WATER STRUCTURE OF FRUIT JUICES INDUCED BY ADDED PLANT EXTARCTS: AN AQUAPHOTOMICS EXPLORATION

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Fruit juice fortification with fruit extracts is a process designed to enhance the nutritional value of juices by incorporating extracts from various fruits into a base juice. This addition enriches the final product with a broad spectrum of bioactive compounds, including antioxidants, vitamins, and minerals, which offer potential health benefits. However, even small amounts of plant extract can significantly alter the juice's physicochemical, as well as its functional properties, including taste and bioavailability. As a result, it is crucial to adopt a comprehensive and controlled approach to monitor and manage fortification, ensuring that the final product maintains its desired sensory qualities and nutritional benefits. In modern quality assurance, non-targeted, fingerprint-based techniques (e.g., chromatography, spectroscopy, artificial sensors, -omics disciplines) are increasingly being used. These methods provide detailed analytical profiles of samples without requiring extensive preparation, and in many cases, they can be non-destructive and even contactless. One of the most important representatives of these techniques is near infrared spectroscopy. Due to the naturally high water content of fruit juices, the application of the aquaphotomics methodology is justified, as it well reflects the chemical, biological, and physical differences. This study aimed to combine near infrared spectroscopy with aquaphotomics to indirectly explore water structural alterations of stone fruit juices induced by enrichment with plant extracts. For this purpose, various fruit extracts and their mixtures were dosed in 0-2.5 g/100 mL to fruit juices made from concentrates. Spectra were acquired using a desktop near infrared spectrometer operating in transmission mode (NIRflex N-500, Büchi, Flawil, Switzerland). Data were analysed through the lens of aquaphotomics, focusing on the 1300-1600 nm wavelength range. The analyses procedures involved the calculation of difference spectra; principal component analysis-based linear discriminant analysis to classify according to fruit extracts and their dosage levels; partial least squares regression to predict extract concentration. Clear trends of separation were observed in the classification results according to the type of juice enrichment. The concentration prediction typically resulted in very accurate correlation (R^2 close to 1). As part of the chemometric modelling, variables that best described the variance in the data were also identified. The 15 most frequently occurring wavelengths determined in this way showed a clear relationship with the different water species, characteristic of fruit juices. The differences discovered by using aquaphotomics can be linked to the bioavailability of fruit juices depending on type and degree of enrichment.

ACKNOLEDGEMENTS: The study was supported by the Doctoral School of Food Science of the Hungarian University of Agriculture and Life Sciences; the Cooperatice Doctoral Programme (KDP-2023), the University Research Scolarship Programme (EKÖP-MATE/2024/25/K), the 2023-1.2.4-TÉT-2023-00068 Program of the Ministry for Innovation and Technology (Hungary) from the source of the National Research, and the CEEPUS mobility grant.

Keywords
fruit juice,plant extract,enrichment,authentication,aquaphotomics

F.04.10

STUDY ON HYALURONIC ACID AQUAPHOTOMICS-FROM ONE DIMENSION TO TWO DIMENSION ANALYSIS

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1 Introduction

Hyaluronic acid (HA) is widely used in cosmetics owing to its unique hygroscopic properties. Depending on the molecular weight, it can pass through different layers of the skin by interacting with water. However, there is still no research, especially on non-invasive methods, on the interaction between HA and water structures in the skin. Near-infrared (NIR) spectroscopy has the advantages of being fast, non-destructive, and capable of real-time monitoring. Aquaphotomics based on NIR was introduced to obtain water information, using water as a label-free probe to reflect the interaction between water and solutes. In this study, we developed a fast and nondestructive method for visualizing skin water species based on aquaphotomics to elucidate the mechanisms by which HA affects water species.

2 Materials and methods

Nine HA samples with different relative molecular weights were selected. The back skin of the Bama pig was used to build the skin model. Active water absorption bands on the skin were identified using chemometric methods. Fig. 1 illustrated the comprehensive experimental framework for the visualization of water species in the skin model.

3 Results and discussion

To compare the intensity difference of water species within the same group and between different groups in a 2D view, the HSI was processed with the characteristic wavelength assigned in Fig.2. The intensity differences of five water species, S0 (1415 nm), S1 (1446 nm), S2 (1464 nm), S3 (1476 nm), and S4 (1486 nm), on the skin model after the application of different HA solutions.

Fig. 3 illustrated the absorption intensity of five water species on the skin model. The application of different HA solutions caused changes in the distribution of the water species on the skin. In contrast to other water species studies, this research achieved an in-situ analysis without damaging skin tissue. NIR-HSI combined with chemometric analysis could directly extract the distribution and relative intensity of water species in the skin. A1 and A9 showed the strongest signals for the water species. A1 and A9 showed the strongest signals for the water species. This might be because HA1 had the smallest Mr and the best permeability, which led to HA1 entering different layers of the skin to exert hydration during the experiment. However, the Mr of HA9 was high, and after the sugar chain increased to a certain length, the molecule formed a rigid spatial structure in the aqueous solution. At that time, the solvation of water was reduced, effectively reducing the loss of these water species. We could conclude that, in future design endeavors for HA products, it is possible to consider the combination of HA tetraosaccharides and HA at 600~700kDa to enhance the abundance of water species in skin and reduce free water loss. With the help of water species distribution, it could help cosmetics formulators optimize their HA formulations to realize their best hygroscopic properties.

4 Conclusions

In summary, we developed a visual analysis method to characterize the mechanism by which HA affects water species. In addition, the change rule of water species in the skin was visualized by converting 1D aquaphotomics to 2D aquaphotomics, which could help us quickly and nondestructively understand the interactions between biomacromolecules and water in biological systems.

ACKNOLEDGEMENTS: This work was supported by the Key R&D Program (Science and Technology Demonstration Project) program of Shandong Province, China [No. 2022SFGC0104] and Shandong Provincial Natural Science Foundation, China [No. ZR2022MB026].

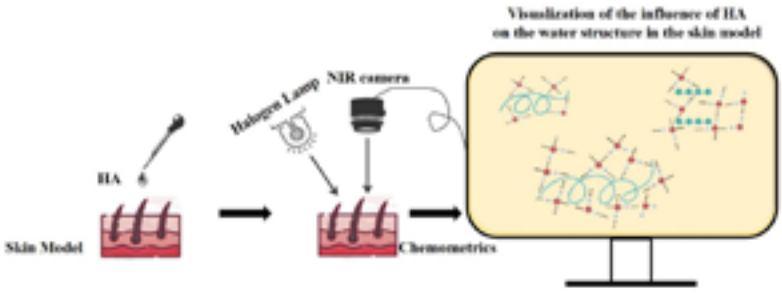


Figure 1 Experimental flow of Aquaphotomics analysis of skin model

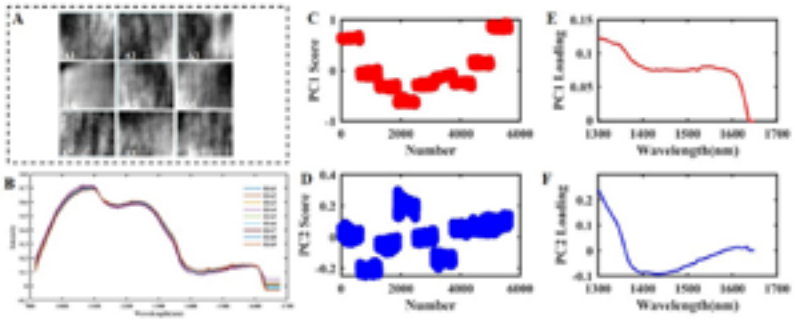


Figure 2 Aquaphotomics analysis of skin model

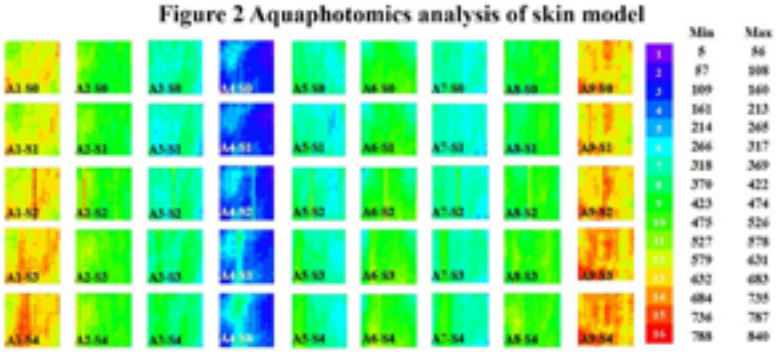


Figure 3 The distribution of five water species on the skin after application of HA1-HA9 aqueous solution

Keywords
Hyaluronic acid,Aquaphotomics,Hydration,Analysis of images,NIR

F.04.11

NON-DESTRUCTIVE NEAR INFRARED SPECTROSCOPY AND AQUAPHOTOMICS FOR MOISTURE QUANTIFICATION OF ALLOGENEIC BIOMATERIALS

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Recent advancements in tissue engineering, bioengineering, and 3D printing technologies are paving the way for unique biomaterials in regenerative medicine that closely resemble natural human tissues and organs [1]. These innovative biomaterials have found wide applications in orthopedics, traumatology, and dentistry. Lyophilized allogeneic biomaterials are tissues and organs obtained from one donor and transplanted to another patient of the same biological species. They represent some of the most promising solutions for restoring defects in supportive and connective tissues [2]. To ensure their safety and biocompatibility for patients, it is crucial to carefully monitor the quality of these materials at every stage of production and during storage.

The moisture content in bioimplants has a significant impact on the preservation of biologically active substances, mechanical strength, and durability of the samples [3]. Consequently, the moisture level is an important quality indicator. Among various moisture measurement methods, thermogravimetric analysis is the most frequently used for assessing the moisture content of biomaterials, providing relatively accurate quantitative results [4]. However, this technique is time-consuming and destructive. Therefore, a new express method is required for fast and non-destructive moisture monitoring in allogeneic biomaterials, preferably through the packaging's protective film.

Near-infrared (NIR) spectroscopy allows rapid, non-invasive, and cost-effective analysis of various processes and products. NIR absorption spectra contain rich information about the water structure, especially in the first overtone region of water (1300-1600 nm). In this regard, NIR spectroscopy is well established for analyzing the moisture content of food products [5]. However, to the best of our knowledge, there are no engineering solutions based on NIR spectroscopy for moisture quantification in allogeneic bioimplants.

In the present work we evaluate the performance of NIR spectroscopy for moisture determination of allogenic biomaterials. Mineralized (dry) and demineralized (wet) bioimplants "Lioplast"® from human spongiosa prepared according to a unique technology at the Research Institute of Biotechnology "BioTech" (Samara State Medical University, Samara, Russia) [2] were analyzed. Two spectrometers were tested and compared: a short-wave fiber-optic NIR Quest (Ocean Optics, USA) and a long-wave NIR portable Phazir Polychromix (Polychromix, USA). Classification models were built to distinguish between the dry and wet samples. Then, the samples were studied during the drying process or with stepwise addition of water, and partial least-squares (PLS) regression models were developed to predict moisture content. Aquaphotomics was utilized to identify changes in the water matrix during the drying process of the samples.

Our results confirm the high prospectively of NIR spectroscopy for moisture determination of bioimplants with high prediction accuracy without destroying the sample. Additionally, the use of aquaphotomics facilitates the assessment of protein conditions in samples, including the structural changes of collagen during the drying process.

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ACKNOLEDGEMENTS: This work was financially supported by the ITMO Fellowship Program.

Keywords
regenerative medicine,allogeneic biomaterials,moisture content,NIR spectroscopy,aquaphotomics

F.04.12

NIR AQUAPHOTOMICS FOR UNDERSTANDING WATER COHERENCE

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In this aquaphotomic study we offer and discuss refined definitions of the concepts of "water coherence" and "water coherent domain", based on NIR monitoring conducted on various waters by FOSS 6500 (NIRSystems, Denmark). The notion of water coherence has so far been referring to molecular spatial coherence in regards to the structural organization and correlated behavior of water molecules, primarily resulting from the extensive hydrogen bonding that occurs between them. In liquid water, this network is considered to lead to the formation of coherent domains, where water molecules maintain a stable arrangement over time, allowing for collective behaviors that can influence the overall characteristics of the liquid. However, these phenomena are also attributable to some water molecular conformations with less hydrogen bonding, such as primary water solvation shells, quasi-free water molecules, as well as several water ions, including hydronium and superoxide. Temperature manipulations on liquid water show that strongly-bounded water molecules respond to applied thermal changes similarly as, and in opposite direction of, less strongly-bounded water molecules. As one of the primary components for hydrogen bonding, temperature has been seen least impactful at spectral isosbestic points formed between spectra of water with varying temperature values. The observation suggests for a non-coherent domain in respect to external conditions where water is most unaffected through its phase transitioning. In the 1st water overtone at 1300-1600nm region, wavelength assignment of the only isosbestic point at 1444nm demonstrating thermal resistivity is the water dimer, with good "flexibility" to environmental changes described by its wide array of possible stable isomers able to absorb and transform energy into new isomeric configurations. Our study proposes the existence of multiple coherent and non-coherent domains in the near-infrared region through utilization of the aquaphotomics method.

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Keywords
NIR Aquaphotomics,water,coherence,molecular dynamics,hydrogen bonding

INSTRUMENTATION, PHARMA & BIOTECH

O.05.14

NEW OPPORTUNITIES WITH INDUSTRIAL NON-CONTACT SUB-SURFACE NIR MEASUREMENTS

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Representative sampling is often a limitation with NIR spectroscopic methods for heterogeneous foods. Reflection measurements are effective when the sample surface represents the target composition, such as in fat determination of ground meat batches, where an average value can be recorded over a large volume at the grinder outlet. NIRS technology, based on high-speed hyperspectral imaging combined with so-called interaction measurements (spatially offset spectroscopy), enables sampling across the entire width of a conveyor belt and to a depth of about 5-10 mm into the sample. This approach allows thorough sampling and has been industrialized for in-line determination of fat in meat trimmings [1] and protein in chicken fillets [2].

Imaging is not always necessary and can waste valuable photons in noise-limited applications such as sub-surface measurements. NIR "point"-spectroscopy in interactance mode is widely used for assessing fruit quality, but the instruments often require physical contact to avoid noise from ambient light. At Nofima and SINTEF Digital, we have recently developed and evaluated novel NIR sensors that allow rapid, non-contact sub-surface NIR measurements. This technology offers rich opportunities for in-depth sampling in industrial processes, exemplified by monitoring dry matter in potatoes [3], core temperature in sausages [4], in-field assessment of sugar in strawberries [5], and meat content in king crabs [6].

The novel NIR sensors have a unique combination of four features:

1) The ability to measure in depth and suppress signals from the surface.

2) The ability to measure without physical contact, even in ambient light.

3) The ability to easily evaluate different illumination geometries to find the best for different products.

4) They are designed to work well under industrial conditions, allowing rapid transition from lab to industry and evaluation under realistic conditions.

Results from two recent studies will be presented:

1) Non-contact measurement of total soluble solids (TSS) in cherry tomatoes moving on a conveyor belt, with typical standard errors of prediction ranging from 0.38% to 0.52%.

2) Industrial monitoring of core temperature in fish cakes, achieving an acceptable accuracy (RMSEP = 1.4 °C).

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ACKNOLEDGEMENTS: This work was funded by the Research Council of Norway through the projects SFI Digital Food Quality along with The Agricultural and Food Industry Research Funds through the projects Precision Food Production.

Keywords
sampling,in-line,sub-surface,NIR spectroscopy,foods

O.05.15

PROTOCOL FOR EVALUATING THE PERFORMANCE OF NEAR-INFRARED SPECTROMETERS

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Over the last decades, an increasing number of near-infrared (NIR) spectrometers have become commercially available, varying in size, spectral range, design and price [1]. While these devices represent new analytical opportunities, assessing their performance and determining their feasibility can be challenging without the experience gained from long-term use.

To address this problem, a protocol was developed to objectively evaluate the performance of reflectance NIR spectrometers and assess the feasibility of transferring calibration models to a new device. This protocol focuses on practical applications and is conducted using products routinely analyzed in the agro-food area.

The protocol includes the evaluation of key parameters such as accuracy, short-, medium- and long-term repeatability, the ability to analyze various sample types and the potential for model development. Measurements are carried out in a controlled laboratory environment (22 ± 3°C) with minimal external variability (e.g. humidity, light, etc.) and following the manufacturer's recommended setup. These optimal analytical conditions ensure that each instrument operates at its maximum potential, while routine use (e.g. field measurements) is expected to reduce performance [2].

The evaluation covers several weeks, and each measurement is performed at least in duplicate, nonconsecutively. For each measurement, the signal-to-noise ratio and root mean square error between repetitions are calculated. First, a homogeneous rare earth sample with six nominal absorption peaks in the NIR region is measured 10 consecutive times to assess the device accuracy and short-term repeatability. A standardization box containing 40 sealed cups of diverse products with varying spectral signatures is then analyzed to assess medium-term repeatability and evaluate transferability with other instruments.

The ability to measure different matrices is evaluated by analyzing a set of wheat grains and a set of soil. While most spectrometers can easily measure wheat flour, measuring whole grains can be challenging, as handheld spectrometers may struggle to generate a sufficiently strong beam to penetrate intact grains [3]. The ability to perform calibration modelling is evaluated by measuring four sets of 20 feed samples with reference values over a four-week period, measuring one set per week. Calibration models are developed to predict protein, fat, fiber and ash by cross-validation and leave-one-week-out cross validation to ensure device stability over time. In addition, throughout the evaluation period, a sealed control cell is measured each morning, and the predicted protein value is used to monitor instrument stability over time.

Although this protocol does not guarantee absolute performance, it provides valuable information on the capabilities of the spectrometers and highlights potential applications. The aim is not to compare spectrometers, as there are internal variabilities, but to establish a baseline understanding of their expected performance. The features of this protocol will be presented, as well as its application on different instruments.

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ACKNOLEDGEMENTS: The authors would like to thank the various CRA-W teams, i.e. the NIR, Fieldpro, and DRIM of the Quality and Authentication of Agricultural Products Department for carrying out the analyses and assisting in the management of the data.

Keywords
Spectrometer evaluation,Protocol,Handheld, and benchtop

O.05.16

ADVANCING SAMPLING STRATEGIES USING LOW-COST MINIATURISED NEAR-INFRARED SPECTROMETER

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Effective sample presentation and scan collection strategies are crucial for obtaining valuable spectra of intact samples and for developing a chemometric model capable of assessing the quality and/or safety of food products. This work evaluates different sampling and scanning procedures to analyse olive oil for human consumption using a miniaturized, low-cost, and easy-to-use near-infrared spectroscopy (NIRS) system. These portable instruments are often constrained by their narrow scanning window and wavelength range, which can sometimes be a limitation for their usability in analysing raw samples [1]. The core of the proposed NIR spectroscopy measurement system is Texas Instruments' NIRscan Nano evaluation module (EVM) spectrometer [2]. The food raw material selected to be analysed is olive oil. To evaluate the feasibility of the prototype to detect frauds/adulterations in olive oil, three sampling/scanning strategies were evaluated, two of them using transmittance as a scan mode, and combining static measurements in a quartz cuvette and dynamic measurements in which the sample was passed through a flow cuvette. The third form of measurement was performed in reflectance mode with a fibre optic probe.

The final objective of the research is not just to check the efficiency of the proposed system. It is also to determine the characteristics of the measurement system, and how to improve them for olive oil security control. Results have shown differences when using static and dynamic transmittance mode compared to fibre optic transmission measurements. Coefficient of determination values of 0.76 for transmission measurements and 0.68 for probe measurements were obtained. Transmittance measurements make it possible to discriminate between pure and adulterated oils.

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ACKNOLEDGEMENTS: The authors would like to acknowledge the Ministry of Science and Innovation for the project Ref. PID2022-142323NB-I00 and PID2023-149221-B-I00RB-I00 funded by MCIN/AEI/10.13039/501100011033/ and by "ERDF A way of making Europe. Candela Melendreras acknowledges her PhD grant (BP22-115) from the Asturias Regional Government (Spain).

Keywords
miniaturized spectrometer,sampling strategies,low-cost devices

O.05.17

INTERPRETABILITY IN NIR SPECTROSCOPY: QUANTIFICATION OF PARACETAMOL IN PHARMACEUTICAL BLENDS AIDED BY VARIABLE SELECTION AND SPECTRAL SIMULATIONS

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Near-infrared (NIR) spectroscopy combined with multivariate data analysis forms a highly successful analytical framework. However, one of the major challenges within it is the interpretability of the resulting models, as the spectra often feature complex overlapping overtones and combination bands. These spectral complexities can obscure the connection between the observed data and the underlying molecular structure of the sample.

This study addresses these challenges by quantifying paracetamol in a pharmaceutical blend containing also caffeine and cellulose using Partial Least Squares Regression (PLS-R) while applying several variable selection methods to enhance both model performance and interpretability. Five variable selection techniques were systematically evaluated: Variable Importance in Projection (VIP), Competitive Adaptive Reweighted Sampling (CARS), Moving Window Partial Least Squares (MW-PLS), Monte Carlo Uninformative Variable Elimination (MCUVE), and Random Frog (RF). The best performance was achieved with the RF method using Savitzky-Golay (SG) second derivative pretreatment, resulting in R^2 values of 0.968, 0.958, and 0.960 for calibration (CAL), cross-validation (CV), and test-set validation (TSV), respectively. Notably, the RF method enabled a reduction in model complexity from the original 8 LVs to just 2 LVs.

In addition to improving predictive performance, this study emphasizes the interpretability of the selected variables. Both analyte-specific and matrix-related spectral bands that contributed to the model were successfully identified and interpreted. The selected variables were linked to specific molecular transitions in the paracetamol spectrum using anharmonic quantum chemical simulations, which also provided insights into the matrix contributions to the model—an aspect often difficult to interpret in conventional NIR models. By integrating variable selection techniques with theoretical simulations, this work demonstrates how the problem of transparency and interpretability in NIR spectroscopy can be addressed, with particular benefits for method development in sensitive and regulatory-driven environments such as pharmaceutical analysis.

Keywords
variable selection,spectra simulation,interpretability,paracetamol,pharmaceutical

F.05.13

NON-DESTRUCTIVE DRUG QUANTIFICATION OF MEDICINES PRODUCED BY PHARMACEUTICAL 3D PRINTING IN A HOSPITAL FOR A CLINICAL TRIAL USING A MINIATURISED NIR SPECTROMETER

Anna Kirstine Jørgensen [1], Antoine Dowek [2], Lucas Denis [2,3], Jun Jie Ong [1], Maxime Annereau [2], André Rieutord [2], Maryam Parhizkar [1], Alvaro Goyanes [1,4,5,6], Abdul Basit [1,5]

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Introduction:
A clinical trial with personalised medicines containing tamoxifen produced by pharmaceutical 3D printing (3DP) is being conducted by Gustave Roussy Cancer Campus [1]. 3DP is an enabling technology for personalised medicines and for small-scale manufacturing [2]. Standard quality control (QC) methods are unsuitable for the production scales offered by 3DP due to their inherent destructiveness [3]. Non-destructive methods such as near infrared (NIR) spectroscopy has been reported capable of verifying various QC attributes of 3D printed, personalised medicines.
This work investigated the feasibility of using a miniaturised NIR spectrometer for dose verification in the medicines produced through a pharmaceutical 3D printer in the Clinical Pharmacy Department of the hospital for the preparation of the clinical trial through two methods of spectra acquisition.

Methods:
Nine calibration formulations spanning from 20.0 - 40.0% w/w tamoxifen citrate (TC) dispersed in polyethylene glycol (PEG) 4000 at 80 C were prepared to predict TC conc. in the medicines produced in the hospital pharmacy (30.0% w/w nominal). Four green/white (cap/body) size 0 gelatine capsules per calibration conc. were filled with ca. 100 - 200 mg formulation through the semisolid extrusion printhead heated to 80 C in the M3DIMAKER2 pharmaceutical 3D printer. Six capsules from the hospital pharmacy were used for model prediction.
NIR spectra were collected using a MicroNIR 1700ES spectrometer with a tablet probe (8 mm collection optic). Open capsules were scanned into from above (collection optic ca. 0.5 mm from capsule shell, 10 scans per capsule). Spectra were preprocessed and partial least squares regression (PLSR) models developed using python. A paired t test was conducted on the determined tamoxifen dose in the medicines produced in the hospital by NIR and reference HPLC method.

Results:
TC quantification from NIR spectra acquired from above into the open capsules resulted in highly linear and more robust calibration models. The spectra were preprocessed with detrend method and second derivation through Savitzky-Golay method (filter width of 19, second order polynomial) before a PLSR model with six latent variables was developed. The resulting model displayed high linearity and goodness of fit (coefficient of determination (R^2) and root mean squared error of calibration (RMSEC)) along with robustness upon 10-fold cross validation (CV) (R^2 = 0.953, RMSEC = 1.373% w/w, R^2 CV = 0.911) (Figure 1).
The prediction accuracy of the model, evaluated by the root means square error of prediction (RMSEP), was 1.427% w/w for capsules produced in the hospital pharmacy, corresponding to a relative error of 4.7%. No statistical difference (p = 0.2603) was found for predicted tamoxifen dose in the medicines produced in the hospital pharmacy through the developed NIR model and the reference HPLC method. The out-of-specification dose unit (according to ± 10% U.S. pharmacopoeia limits) was accurately identified by the NIR predicted TC conc. and fill weight (Figure 2).

Conclusions:
A highly predictive model was developed for employment at the hospital pharmacy for non-destructive tamoxifen dose verification in the medicines produced via a pharmaceutical 3D printer for the clinical trial. No statistical differences were found for mean doses predicted by the NIR model and HPLC method.

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Figure 1.

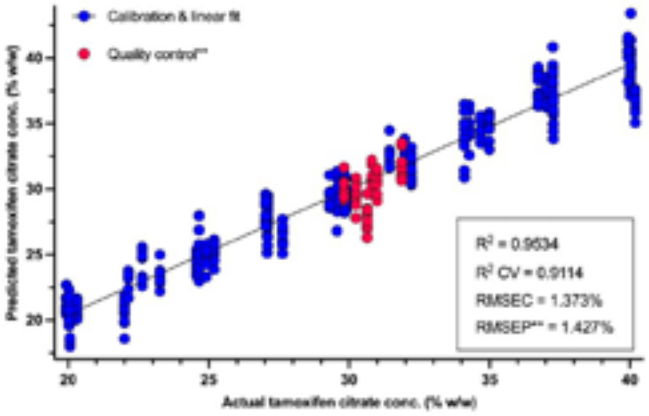
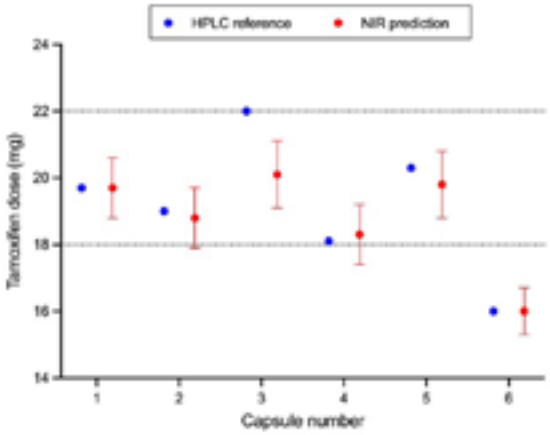


Figure 2.



ACKNOLEDGEMENTS: A.K.J. acknowledges the U.K. Engineering and Physical Sciences Research Council (EPSRC) (EP/S023054/1) for their funding.

Keywords
Non-destructive quantification,3D printed pharmaceuticals,Handheld spectrometers,Quality control,Hospital pharmacy

F.05.14

NIR SPECTROSCOPY IN PERSPECTIVE - A PROVEN ANALYTICAL TOOL WITH EXPANDING APPLICATIONS

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Near-infrared (NIR) spectroscopy has undergone a long path of development, evolving into a valued and widely used analytical technique. Despite reaching this level of maturity, NIR spectroscopy continues to demonstrate remarkable adaptability to new challenges and emerging fields. Its flexibility is evident in recent technological advances, such as the miniaturization of spectrometers, which has enabled portable, on-site analysis. Handheld devices are becoming crucial in environments where real-time data is essential, particularly in fields like environmental monitoring and phytopharmaceutical quality control.

Additionally, breakthroughs in spectral data analysis, including advanced methods for interpreting complex spectral features, have further extended the capabilities of NIR spectroscopy. Quantum chemical simulations now provide a more precise understanding of absorption bands and their manifestation in chemometric models, enhancing our ability to extract valuable analytical information from NIR spectra. Combined with the continuous refinement of multivariate methods, these advancements have pushed the boundaries of NIR spectroscopy, enabling more targeted and efficient analyses across various disciplines.

The continued evolution of NIR spectroscopy is evident in both applied and fundamental research. This discussion will focus on its growing importance in immediate applications like phytopharmaceutical analysis and food authentication, as well as its potential to address emerging challenges, such as the detection of microplastics in environmental samples. Moreover, the increasing synergy between NIR spectroscopy and other analytical techniques is a significant trend, with NIR being effectively combined with other spectroscopic methods, both molecular and atomic such LIBS, paving the way for new and expanded applications.

Keywords
NIR spectroscopy,miniaturization,phytopharmaceuticals,analytical applications

F.05.15

ESTIMATION OF PARACETAMOL IN INTACT TABLETS BY USE OF NEAR INFRARED SPECTROSCOPY AND ARTIFICIAL NEURAL NETWORK

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Paracetamol, or acetaminophen, is among the most widely consumed drugs worldwide. Assays for intact paracetamol tablets face challenges, as standard production batches lack sufficient variation for reliable calibration equations. To address this, tablets with underdosed and overdosed analytes must be specifically prepared.

The US-Pharmacopoeia recommends HPLC-UV for paracetamol content analysis in solid samples. While HPLC is accurate and precise, it is limited by high operational costs, time-intensive protocols, and extensive sample preparation. In contrast, Near-Infrared Spectroscopy (NIRS) offers rapid, non-destructive methods for quality control and counterfeit detection. Recent studies emphasize its application in quantifying active pharmaceutical ingredients, with machine learning and nonlinear regression techniques like Gaussian Process Regression enhancing accuracy. Despite challenges with spectral variability and model robustness, NIRS shows potential, especially in resource-constrained settings [1-3].

This study combined NIRS with Artificial Neural Networks (ANN) to quantify paracetamol content in tablets. A case study analyzed common over-the-counter brands in India, including paracetamol and diphenhydramine hydrochloride powders. Pretreated spectra using transformations such as Standard Normal Variate (SNV) and Savitzky-Golay filtering enhanced data quality.

Representative samples were prepared by mixing paracetamol powder (IP grade, >99% purity) in varying quantities (400-1200 mg) with excipients. Purity was verified using HPLC (IP-based method). NIR spectra were acquired with a Metaspeq spectrometer (Ayudyog, Kolkata) in diffuse reflectance mode, covering 900–1,700 nm. Scans averaged 16 repetitions per sample, with preprocessing steps like scatter correction and smoothing were applied.

The spectrometer produced 256 spectral points per sample, generating a total of 256 × 480 × 16 data points across 480 samples with 16 replicates each. PCA reduced dimensionality before ANN analysis. The ANN model comprised an input layer, two hidden layers, and one output layer, with relu activation functions and ADAM optimization. The training set's mean squared error was 0.0031, and the test data set showed an average prediction deviation of 6.77%.

This study demonstrates the feasibility of NIRS combined with ANN for assessing paracetamol content in tablets. The approach is adaptable for quality control and content uniformity in tablet production lines, providing a fast and non-destructive alternative to traditional methods.

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Keywords
Paracetamol,Portable NIR,ANN,PCA

INDUSTRY & PAT

O.06.18

AUTOMATED CERAMIC-GLASS SEPARATION USING HYPERSPECTRAL IMAGING: A SUSTAINABLE SOLUTION FOR RECYCLING

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Ceramics present a significant challenge in the glass recycling industry, disrupting processes and compromising product quality. Their similar appearance and properties to glass limit reliable detection and automation. Existing methods, such as UV-based imaging, rely on narrow spectral ranges like 365 nm, often resulting in false positives for tinted or impure glass. Spectrometers provide precise analysis but lack spatial data capture, limiting their industrial applicability [1][2].

This study introduces a first approach to automating ceramic-glass separation using hyperspectral imaging (HSI). The developed workflow includes HSI capture, segmentation, preprocessing, dimensionality reduction, classification and evaluation [3]. Experiments involved a diverse dataset of ceramic and glass samples, including additional data to replicate real-world conditions. All these experiments were performed using the Newtec Buteo hyperspectral camera [4], capable of capturing a broad spectral range (430-1700 nm) across 900 spectral bands.

Preprocessing was performed using various techniques, including Min-Max normalization, Savitzky-Golay smoothing, and spatial filtering [5][6]. Min-Max normalization was finally selected for its computational efficiency and preservation of spectral integrity. LDA was employed for dimensionality reduction, identifying key wavelength range (450–570 nm and 1000–1200 nm) [7]. Among classifiers evaluated (SVM, KNN, RF, etc.), SVM demonstrated the best performance [8].

Two prediction studies were developed: i) object-based predictions, achieving up to 97.46% accuracy using segmentation methods such as MobileSAM [9] and Background Substraction [10]; and ii) continuous (line-by-line) predictions, with execution times of 0.0806 seconds per line, which makes it a solution with great potential for real-time environments (high throughput production lines).

The results demonstrate that the proposed system performs well in both detection ratio and processing speed. The proposed HSI-based approach aims to provide a sustainable solution for improving recycling processes in industrial contexts and represents a first step toward automating ceramic-glass separation, overcoming traditional method limitations.

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ACKNOLEDGEMENTS: We would like to thank Elkartek for providing the economic framework for this work, and José Manuel Amigo for facilitating access to the necessary equipment.

Keywords
hyperspectral imaging,hyperspectral processing,ceramic-glass separation,sustainable recycling,industrial applications

O.06.19

NEAR-INFRARED SPECTROSCOPY AS PAT MONITORING TOOL FOR THE DEVELOPMENT OF BIOPHARMACEUTICAL PRODUCT & PROCESSES

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During the fill and finish manufacturing process of a biotech products, freeze-drying is crucial for long-term stability, as it removes most of the water through sublimation, converting the ice into vapor, under low pressure and temperature. Residual moisture (RM) is a key Critical Quality Attribute to monitor in order to assess the product quality. The method currently used to measure RM in freeze-dried products is the Karl-Fischer titration, which has several drawbacks: it is destructive, resulting in economic loss. It is also time-consuming, requiring sample handling and instrument calibration before each analytical run. In line with the regulatory recommendations for the implementation of Process Analytical Technology (PAT), Near-Infrared Spectroscopy represents a powerful alternative tool for the evaluation of the RM in freeze-dried products. However, this spectroscopic technique generates large amounts of data, leading to the need for multivariate data analysis to extract useful information from the spectra.

At-line NIR-based application

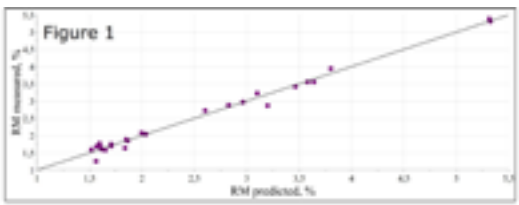
A primary challenge in model development within a pharmaceutical company is the limited availability of Drug Products and the significant costs associated with their use for modelling. Therefore, if a model developed using Placebo data effectively predicts RM in the Drug Product, this could greatly facilitate the integration of modelling techniques into process design and validation activities. In this context, a Partial Least Squares (PLS) regression model was developed on a surrogate product and tested on a real Drug Product to reduce the experimental effort and costs associated with model development.

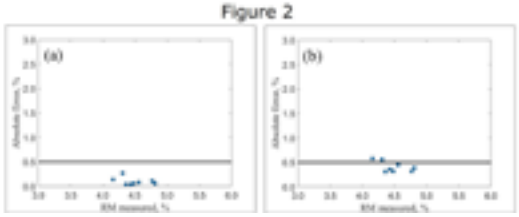
A 6% w trehalose in water was used as a surrogate product since the Drug Product primarily contained trehalose as excipient. Two different wavenumber ranges were explored. For model performance, parity plots are presented, showing RM measured by Karl-Fischer against RM predicted by the model, as reported in Figure 1. Additionally, the Root Mean Squared Error was calculated for both the calibration and cross-validation sets (RMSEC and RMSECV). The similarity between RMSEC (0.129%) and RMSECV (0.136%) underscores the strong performance of the developed model.

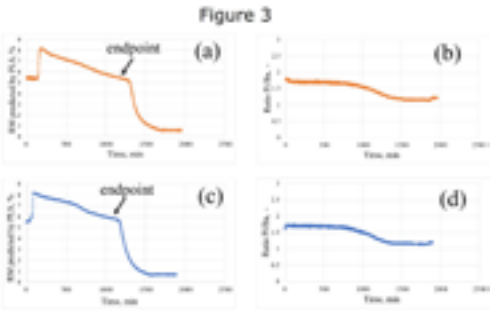
The developed PLS models were used to predict ten different samples of the Drug Product after they underwent the freeze-drying cycle. The absolute error plot is presented for two different wavenumber ranges: one specific for the water peak and the other one specific for the full wavenumber ranges. Additionally, a threshold of 0.5% was established, considering the analytical variability of Karl-Fischer method. Focusing on the water absorption bands while excluding product-specific peaks yields more favourable results, as shown in Figure 2a. This is a significant achievement, demonstrating that the model developed with a surrogate product can accurately predict the RM values of a real Drug Product.

In-line NIR-based application

The PLS model was used to estimate the RM trend during the freeze-drying process by inserting the NIR probe in the freeze-dryer chamber. Additionally, the endpoint of primary drying phase was calculated and compared with the one measured by Pirani-Baratron approach for two freeze-drying cycles. The results obtained are summarized in Figure 3.







ACKNOLEDGEMENTS: I would like to thank my supervisors, Davide Fissore and Nunzia Falco, for their constant support. I also want to thank my colleagues, Daniele Mari, Caterina Sapienza, and Michele Dimattia, for their experimental support.

Keywords
PAT,Multivariate Data Analysis,Monoclonal Antibody,Freeze-drying,Process Development

O.06.20

THE UNTAPPED POTENTIAL OF NEAR-INFRARED SPECTROSCOPY FOR INLINE PROCESS MONITORING OF WATER QUALITY INTENDED FOR REUSE

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University of Copenhagen, Copenhagen, DENMARK

Fresh water is a limited resource in growing demand as the global population increases. Freshwater scarcity has already reached a level in which two-thirds of the global population (4.0 billion people) live under conditions of severe water scarcity at least 1 month of the year (Mekonnen & Hoekstra, 2016). At the same time water is the main ingredient in most food products and an important part of food manufacturing processes such as soaking, washing, rinsing, fluming, blanching, scalding, heating, pasteurizing, chilling, cooling, steam production, as an ingredient, and for general cleaning, sanitation and disinfection purposes. The food industry is dependent on water for cleaning and sanitation and is a major consumer of tap water. It is thus of prime importance for the food industry to implement a sustainable water use that is compatible with the health of the people, communities and ecosystems where the industry operate.

Rapid real-time monitoring of water quality can help facilitate the reuse of industrial process water. Near-infrared spectroscopy is a well-established tool for process monitoring within the pharma and agri-food industries. Due to the prize, speed, accuracy and the green analytics technology of this method (Czaja & Engelsen, 2025), there is an industrial interest in exploiting the possibilities of applying near-infrared spectroscopy to solve water related production issues related to sustainable water use and reuse(Holst, van den Berg, & Engelsen, 2014).

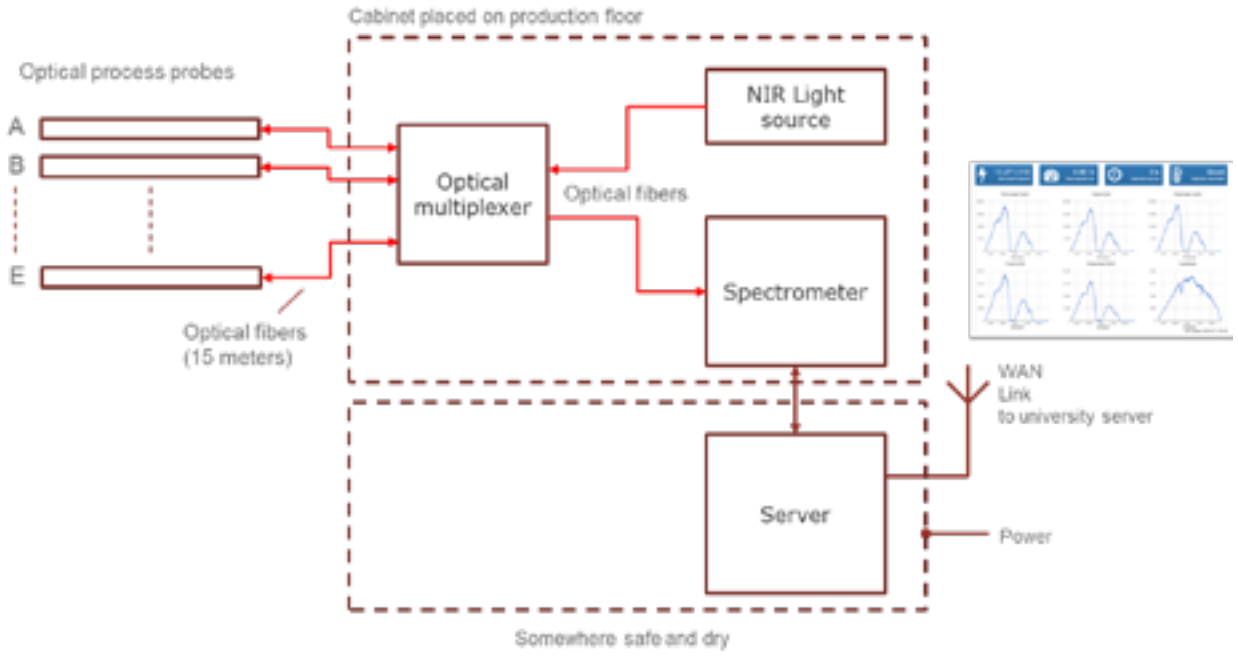
This paper demonstrates the application of NIRS technology to monitor a reverse osmosis cleaning system for whey water intended for reuse. We show the result obtained from approximately one million NIR spectra recorded over the production for half a year – and discuss possibilities and limitations. Digitalization and data technology are critical components for safe circular water reuse and for monitoring the water savings.

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Keywords
water reuse,NIRS,process monitoring,whey water,circular economy

OS.06.01

ROADMAP FOR THE IMPLEMENTATION OF A PAT PROJECT IN THE FIELD OF MOLECULAR SPECTROSCOPY - AN INDUSTRIAL CASE STUDY

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Hellma GmbH & Co. KG, Muellheim, GERMANY

In today's competitive world, the implementation of Process Analytical Technology (PAT) has become increasingly crucial across all industries. In any case, whether for cost reduction, quality improvement, risk mitigation, or regulatory compliance, the use of PAT provides a direct insight into the production process. An important component in this regard is molecular vibrational spectroscopy. With non-destructive molecular spectroscopy the possible applications in food, chemicals, or pharmaceuticals, from the laboratory to industrial facilities are countless. The vast range of technological options, challenges, and regulatory requirements makes the planning and implementation of a PAT project highly complex. Critical success factors extend beyond the choice of hardware and include aspects of system integration, process adaptation, and the operational environment. This presentation outlines a comprehensive roadmap for the successful realization of a PAT project, illustrated with insights from a real-world industrial case study.

ACKNOLEDGEMENTS: Hellma Solutions Teams of Experts

Keywords
PAT project,Molecular Spectroscopy,Production Process Monitoring,Industry

F.06.16

EMBEDDED NEAR INFRARED SPECTROSCOPY FOR PHARMACEUTICAL CONTINUOUS MANUFACTURING

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Novartis Pharma A.G., Basel, SWITZERLAND

Continuous Manufacturing (CM) of pharmaceutical drug products represents a paradigm shift from traditional batch manufacturing processes due to its potential for enhanced manufacturing flexibility and efficiency. In CM, all process units are seamlessly integrated, allowing for a continuous flow of materials and real-time adjustments. Process Analytical Technology (PAT) is part of the control strategy in CM, enables consistent product quality through continuous monitoring and offers potential for advanced control. Within this framework, Near Infrared Spectroscopy (NIRS) plays a pivotal role, particularly in the control and monitoring of the continuous pharmaceutical manufacturing (Figure 1).

The integration of Near Infrared Spectroscopy (NIRS) in the pharmaceutical continuous manufacturing line represents a significant advancement in the pharmaceutical industry. The spectrometers are directly controlled by the automation system without additional computers in order to create a robust PAT control system. Three NIRS instruments were integrated into the process: one spectrometer placed after drying to monitor blend uniformity and water content, and two within the tablet press to guarantee both blend and content uniformity (Figure 2).

NIRS evaluates five parameters: blend uniformity after fluid bed drying (FBD), water content (loss on drying) post-FBD, blend uniformity in the tablet press feed frame, tablet uniformity synonymous with content uniformity, and identity. These parameters are crucial for controlling and releasing the product during the continuous manufacturing process.

The calibration and validation of the five models were successfully accomplished by implementing a comprehensive approach. The process involved the selection of appropriate preprocessing techniques, determining the optimal number of principal components (PC), and conducting thorough validation in terms of linearity. The performance metrics such as Root Mean Square Error of Calibration (RMSEC), Root Mean Square Error of Cross-Validation (RMECV), and Root Mean Square Error of Prediction (RMSEP) were evaluated and will be presented in detail. This rigorous calibration and validation process ensures the robustness and reliability of the NIRS models, which are important for their effective application in real-time quality control within the continuous manufacturing process.

The PAT equipment and models will serve multiple purposes, such as real-time production line monitoring to detect disturbances. They will also be used to analyze residence time distribution for better process comprehension and will be integrated within the manufacturing line's automation and actively control the manufacturing line.

This study demonstrates the application of NIRS for real-time control and monitoring of the continuous manufacturing process. The results confirm that embedded NIRS is an effective Process Analytical Technology (PAT) tool for continuous manufacturing, enabling real-time quality control and facilitating opportunities for Real Time Release.

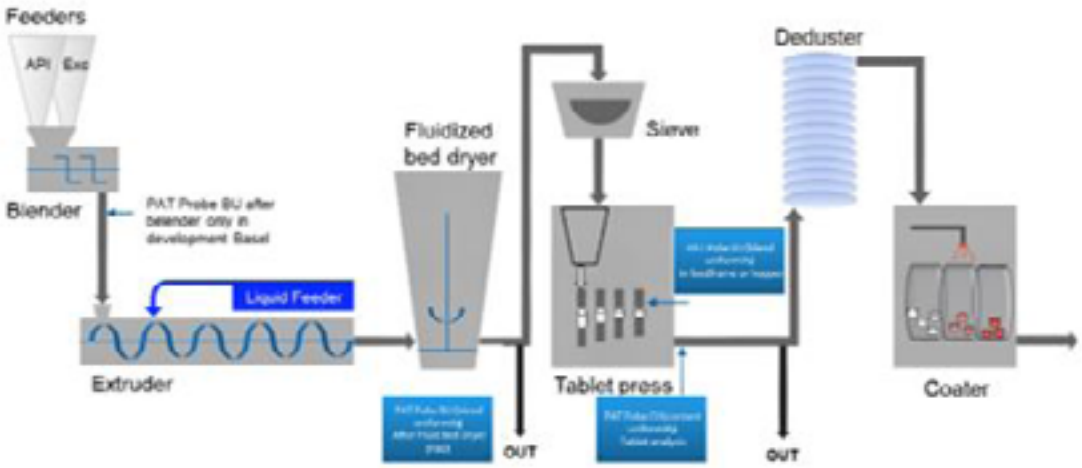


Figure 1. Continuous manufacturing line and PAT sensors

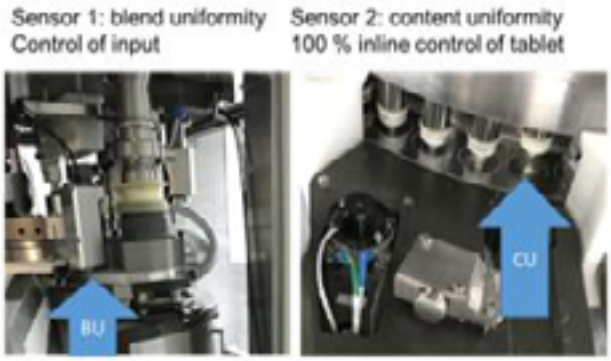


Figure 2. Tablet press with two NIR probes: Blend Uniformity of powder and Content Uniformity of tablet

Keywords
Process Analytical Technology,Embedded spectroscopy,Tablet press,Blend uniformity,Pharmaceutical

F.06.17

USING COLLABORATIVE ROBOTS TO AUTOMATE NEAR-INFRARED SPECTROSCOPY MEASUREMENT OF WOOD SAMPLES

Edit Földvári-Nagy [1,2], Aminata Dia [3], Jakub Sandak [1,2]

[1] *InnoRenew CoE, Izola, SLOVENIA,* [2] *University of Primorska, Koper, SLOVENIA,* [3] *CESI Graduate School of Engineering, Puteaux, FRANCE*

Reliable evaluation of biological-origin materials with NIR spectroscopy requires special routines to mitigate natural heterogeneity and variability within and between samples. Common strategies include increasing the number of samples or measurement points and homogenizing the sample by rotating or moving it during the scan. However, working with heterogeneous sample sets and large-sized samples can be cumbersome and challenging to perform manually. A significant limitation of manual scanning is the risk of human error in reading/typing sample codes or improper sample presentation. To address these challenges, we developed various robotic systems to support operators in the routine measurement of near-infrared spectra on different frequently studied wood sample topologies. A key aspect of the project was to reduce the time technicians spend on manual tasks, allowing them to allocate their efforts more efficiently.

The first example of the automated solution is the routine determination of wood sorption isotherms. This time-consuming experiment requires wood samples to be conditioned, weighed, and measured multiple times during conditioning cycles. A collaborative robot (ABB YuMi) was selected as the core of the prototype system. The YuMi robot consists of two seven-axis robotic arms integrated into a single system. The robot was fully synchronized with add-on measurement components, including a laboratory balance, metrological video camera, and portable near-infrared spectrometer (NIRONE 2.2, Spectral Engines). The robot arms transported samples between predefined workstations, while coordination tasks for the measurement sequence were performed by communicating with a PC supervising all hardware components.

The second robotic system was built using the ABB GoFa collaborative robot, equipped with a custom 3D-printed tool holder for the fibre optic NIR probe. The robot was integrated with the FT-NIR multi-purpose analyser MPA II (Bruker Optics). Custom control software was coded using ABB RobotStudio, and the HTTP protocol was used to trigger NIR spectra measurements in OPUS (Bruker Optics). The system was designed to automatically process a set of eight wood samples in the form of disks cut from harvested trees. The measurement routine included reading the barcode corresponding to the sample code and visually determining the disk's radius. Spectra were acquired at 5 mm intervals from the pith to the bark, and reference background images were taken before each sample to ensure the highest data quality. During the measurement campaign, 1022 wooden discs were measured, resulting in the collection of more than 26,000 FT-NIR spectra. This extensive dataset is key to linking phenotypic and genotypic data, modelling the effects of genetic variation within tree populations, and ultimately gaining a deeper understanding of the ecological resilience of trees. It is estimated that the manual measurement of the same sample set would require more than 450 hours of intensive labour, not counting sample handling nor code reading/typing operations. This automation not only significantly speeds up the work process, freeing up human resources, but also guarantees precise spatially-resolved measurements performed by the robot.

ACKNOLEDGEMENTS: Part of this work has been developed within project "Spectroscopic Methods for Rapid Phenotyping of Trees Reflecting their Ecological Resilience" (acronym DENDRO-SPEC), co-financed by the National Science Centre Poland (NCN) No. UMO-2021/43/I/NZ9/02809 and the Slovenian Research Agency (ARRS) N4-0274 and project SINTETIC receiving funding from the European Union's Horizon Europe research and innovation programme under the grant agreement No 101082051.

Keywords
automation,collaborative robot,wood properties,moisture content,wood phenotyping

F.06.18

RAPID AND RELIABLE ANALYSIS OF QUALITY PARAMETERS OF CRUDE OIL UNIT CHARGE USING NIR SPECTROSCOPY AND CHEMOMETRIC MODELING

Selahattin Senturk [1], Ayten Ekin Mese Sezen [1], Melike Duvanoglu [1], Durmus Ozdemir [2]

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Crude distillation units, the first step in crude oil refining, operate as fractional distillation columns to separate petroleum fractions based on their boiling points. These units process crude oils as blends rather than individually, with compositions adjusted to maximize refinery profitability. Accurately determining the quality of these blends is essential for optimizing unit efficiency, enhancing distillate yield, and ensuring high-quality production. Critical parameters such as API gravity, asphaltene, and sulfur content are typically analyzed in laboratories according to ASTM standards. However, these traditional methods are time-consuming, costly, and require skilled personnel, which can delay decisions and increase operational costs.

Implementing rapid and reliable quality prediction methods addresses these challenges by enabling real-time adjustments to crude blends and process parameters. This approach minimizes uncertainties associated with crude oil variability, significantly improving distillation unit performance and overall refinery efficiency. By reducing analysis time and resource requirements, it allows operators to optimize production processes, maximize yield, and lower costs, representing a critical advancement in modern refining operations.

This study demonstrates the rapid and reliable analysis of critical quality parameters in crude oil unit charges by using Near-Infrared (NIR) spectroscopy in reflectance mode combined with chemometric modeling. Over a two-year period, an extensive dataset of NIR spectroscopic measurements was gathered from more than 10 distinct crude oil sources and their combinations. Obtained spectral data were treated with different data preprocessing techniques and two different multivariate calibration approaches which are Partial Least Squares (PLS) and Genetic Inverse Least Squares (GILS) algorithms were interpret this complex data and assess crude oil quality.

The developed chemometric models were validated using independent data set and the performance of the models were evaluated according to root mean squared of prediction (RMSEP) values as well as reproducibility values reported on ASTM standards. Prediction performances of PLS and GILS algorithms were also compared as well as an ensemble model of two algorithms. The results demonstrated that GILS algorithm and ensemble models exhibit high precision, with approximately 80% success in predicting critical quality parameters such as API, total sulfur content, and asphaltene content of crude oil samples. This study shows that utilizing NIR spectroscopy combined with chemometric modeling for the rapid and reliable analysis of crude oil quality parameters represents a transformative advancement in refinery operations.

Keywords
NIR Spectroscopy,Petroleum Refining,Crude Oil Quality Analysis,Chemometric Modeling,Genetic Inverse Least Squares

CHEMOMETRICS

OS.07.02

NEW INNOVATIVE INSTRUMENTS AND CONCEPTS FOR FT-NIR ANALYSIS

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Bruker Optics GmbH & Co. KG, Ettlingen, GERMANY

Spectroscopy, particularly Near-Infrared (NIR) Spectroscopy, is a well-established and essential tool for quality control. However, there is always room for improvement and new concepts or designs. This presentation will highlight two recent advancements in the field:

1. MOVE-T Liquid Analyzer: This instrument is designed for the quality control of raw milk, liquid finished products, intermediates, and premixes, as well as plant-based milk drinks. It integrates a homogenizer and a peristaltic pump feeding a quartz flow cell, enhancing the precision and efficiency of liquid analysis.

2. BEAM Single-Point FT-NIR Spectrometer: The BEAM is a single-point spectrometer that leverages the full power of FT-NIR spectroscopy for in-process control. It combines the full NIR spectral range capability and x-axis stability with a compact design suitable for direct mounting, eliminating the need for fiber optics.

This talk will discuss the technical innovations and practical benefits of these instruments, supported by real-world examples.

Keywords
FT-NIR,process control,milk analysis

O.07.21

OPTIMIZING NIR CALIBRATION MODELS WITH SEMI-AUTOML AND FUSION MODELS

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Eigenvector Research, Inc., Manson, USA

Near-infrared Spectroscopy (NIR) is used extensively in monitoring and control systems in a wide variety of industrial applications. This includes oil and gas refining, food and beverage, pharmaceutical and bio-pharmaceutical Process Analytical Technology (PAT) applications and many others. There are many calibration models required as part of these NIR applications. Many of these applications require occasional to frequent updating of the calibration models because of changes to process inputs or output targets. The number of calibration models required has created a heavy load for the available analysts. Thus, there is a need for tools which increase the efficiency of the model creation process.

We present here a semi-automated machine learning (semi-AutoML) framework for generating, evaluating, and optimizing ensembles of predictive models is presented. We refer to this system as "Diviner." (Divine—to discover or locate something by intuition, insight or supernatural means.) Unlike fully automated approaches, Diviner's semi-AutoML balances efficiency with expert-driven input, providing greater transparency and flexibility than traditional AutoML pipelines. By incorporating user knowledge and domain-specific insights, this framework helps overcome challenges in interpretability, customizability, and oversight that often arise when model selection is fully automated.

Instead of identifying just one "best" model, Diviner creates a diverse set of candidate models. These models differ in preprocessing methods, variable selections, model hyper-parameters, and underlying regression algorithms. They are evaluated using cross-validation error, predictive accuracy, and overfitting metrics. This diversity is critical for effective model fusion.

To harness this diversity, our approach employs a model fusion technique—also known as voting regression. By aggregating predictions from multiple calibrated models through methods such as mean or median, it leverages complementary strengths and reduces individual model weaknesses, enhancing overall predictive robustness.

Building on these foundations, Diviner systematically explores various ensemble configurations to identify the optimal combination of models. It considers factors like ensemble size and model diversity while monitoring performance and potential overfitting. The result is a more transparent and resilient modeling solution that surpasses the accuracy and reliability of any single model in the ensemble, and reduces the risk of selecting a single sub-optimal model.

The Diviner approach for creating models and optimizing ensembles is demonstrated with several NIR calibration problems. Results show that ensembles typically outperform any single model.

By uniting semi-AutoML principles, model fusion techniques, and systematic search strategies, Diviner fosters a collaborative, knowledge-driven approach to improving predictive accuracy, enhancing robustness, and providing a more insightful machine learning workflow, from which the analyst is likely to learn about the system under consideration.

Keywords
calibration,machine learning,automated,ensemble,optimization

O.07.22

PRELIMINARY PREDICTION OF MILK HEAT STABILITY USING NIR, MIR AND RAMAN SPECTROSCOPY COUPLED WITH CHEMOMETRICS: A COMPARISON BETWEEN TRADITIONAL PLS AND NON-LINEAR MODELS

Áine Ní Fhuaráin [1], Jiani Luo [1], Kexin Zhang [2], Ming Zhao [1], Jeremiah Sheehan [2], James O'Mahony [3], Tom O'Callaghan [3], Arlene McGrath [2], Jonathan Magan [2], Mark Fenelon [2], Norah O'Shea [2], Colm O'Donnell [1], Aoife Gowen [1]

[1] *UCD School of Biosystems and Food Engineering, University College Dublin, Dublin, IRELAND,* [2] *Teagasc Food Research Centre, Moorepark, Cork, IRELAND,* [3] *School of Food and Nutritional Sciences, University College Cork, Cork, IRELAND,* [4] *Dairy Processing Technology Centre, Limerick, IRELAND*

Predicting the heat stability of milk using spectroscopy is vital for an agile approach to dairy processing and quality control. Predicting the functional properties of milk using Near-Infrared (NIR), Mid-Infrared (MIR) and Raman spectroscopy is rapid and nondestructive, and could allow milk product segregation in real-time. Using spectroscopy would also result in a decreased reliance on laborious wet chemistry, reducing costs and waste.[1] In this study, 1,821 samples from 7 Irish dairy processors had oil bath heat stability reference measurements coupled with NIR spectra taken on an MPA instrument (Bruker, MA, USA) from 2022-2024, MIR spectra taken on a DairySpec instrument (Bentley, Chaska, Minnesota, USA) from 2021-2024 and Raman spectra using an i-Raman Plus 785S (Metrohm, Herisau Switerland) portable instrument from 2022-2024. Spectral noise was removed and various pretreatments trialled, such as Standard Normal Variate (SNV). Principal Component Analysis (PCA) was performed to explore seasonal trends in the data. Regression models were created using modelling methods including traditional Partial Least Squares (PLS) and non-linear modelling such as neural networks. Prediction was assessed using metrics such as Ratio of Prediction to Deviation (RPD) and Root Mean Square Error of Cross Validation (RMSECV). An external test set of every fifth week in the dataset is used to validate the model performance. These models show potential to be implemented into industry in the future, with reasonable errors achieved.

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ACKNOLEDGEMENTS: This work has been supported by the Dairy Processing Technology Centre funded through Enterprise Ireland. Grant Agreement Number: TC-2020-0028

Keywords
Chemometrics,Food & Beverage,Industry & PAT

O.07.23

QUANTIFICATION OF QUATERNARY MIXTURES OF MICROPLASTIC BY USING AN ULTRA-COMPACT NEAR-INFRARED SPECTROMETER

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Due to the enormous production and mismanagement, the issue of plastic waste represents a critical and urgent social, economic, and environmental challenge. Microplastics (MPs) have emerged as a significant focus of research, primarily because of their pervasive presence and potential risks to ecosystems and human health. One of the major challenges in addressing MPs is the ability to conduct extensive environmental monitoring. Among the available technologies and analytical techniques for monitoring MPs contamination, portable near infrared (NIR) spectrometers [1] have gained popularity in recent years. These devices enable fast, on-site data acquisition. However, multivariate data analysis methods are necessary to process the spectral information collected, particularly when the goal is to classify or quantify different polymers [2][3].

This study focuses on developing a method for quantifying four types of MPs: polypropylene (PP), polyethylene (PE), polystyrene (PS), and polyethylene terephthalate (PET). Everyday objects were used to produce MPs: starting materials were first characterized using mid-Infrared spectroscopy, then mechanically fragmented in a laboratory setting to mimic the environmental degradation process, producing realistic MP-sized particles. A total of 72 samples were prepared, including pure samples, as well as ternary and quaternary mixtures. For each sample, MPs were distributed on a metal plate, and 10 individual acquisitions were performed randomly, by using a compact NIR spectrometer working in the range 908–1676 nm.

Principal Component Analysis (PCA) was initially carried out to identify outliers based on Q-residuals and Hotelling's T^2 metrics. The dataset was then split into training and test sets (80/20%) and different regression strategies for predicting MPs were evaluated: four individual Partial Least Squares (PLS1) models versus a single PLS2 model, along with various pre-processing methods, including baseline correction, second derivative, Standard Normal Variate (SNV), and their combinations.

Regression parameters, such as R^2 and root mean square error (RMSE) for calibration, cross-validation, and prediction, were compared to identify the best strategy for quantifying quaternary samples. The best results were obtained using PLS2 with second derivative and SNV pre-processing, achieving an R^2 of 0.94 in prediction and a prediction error of approximately 4.8%. Comparable results were also obtained with the PLS1 models. The selected model was subsequently applied to quantify MPs in real samples collected from various beaches in Sardinia, Italy. The promising integration of Chemometrics with portable NIR devices highlights the potential for developing further multivariate models to include a broader range of MP polymers.

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Keywords
near-infrared spectroscopy,microplastics,chemometrics,multivariate regression

O.07.24

QUANTIFICATION OF TOTAL PETROLEUM HYDROCARBON CONTENT IN SOILS USING HANDHELD NIR: DEVELOPEMENT OF A LOCALLY WEIGHTED PARTIAL LEAST SQUARES GLOBAL, SITE AND INSTRUMENT-INDEPENDENT CALIBRATION MODEL

Marina De Gea Neves [1], Barry M. Wise [2], Frank Pfeifer [1], Heinz W. Siesler [1]

[1] *Department of Physical Chemistry, University Duisburg-Essen, Essen, GERMANY,* [2] *Eigenvector Research Inc., Manson, USA*

Soil contamination by Total Petroleum Hydrocarbons (TPH) represents a significant environmental concern, necessitating the development of rapid and reliable quantification methods. Achieving predictive accuracies with errors below <1500 ppm remains a key challenge. In this context, handheld near-infrared (NIR) spectroscopy, in combination with advanced chemometric techniques, has emerged as a promising tool for on-site soil analysis. This presentation focuses on two main objectives: First, it demonstrates the development of site-independent calibrations using a Locally Weighted Partial Least Squares (LW-PLS) model, enabling the creation of a global calibration framework based on 314 soil samples collected from three different continents. The first step was conducted previously and published in [1]. Second, the study expands the existing global calibration by incorporating additional spectral data from selected soil samples, analyzed with eight additional NIR scanners of the same model, resulting in a total of 458 samples. This expansion led to the development of a LW-PLS calibration that accounts for instrumental variability, ensuring that the calibration is independent of fluctuations associated with this particular NIR scanner type.

This research lays the foundation for the establishment of a global network of field offices, equipped with miniaturized NIR spectrometers of the same type, and utilizing a standardized LW-PLS calibration model. This network will facilitate consistent on-site measurements of TPH content in soils, ensuring comparability of results across diverse geographical locations and instrument types, with absolute errors maintained below 1500 ppm. In doing so, it fosters a more cohesive and standardized approach to the global monitoring of soil contamination.

[1] T. Miao, N. Sihota, F. Pfeifer, C. McDaniel, M. De Gea Neves, and H. W. Siesler;

Rapid Determination of the Total Petroleum Hydrocarbon Content of Soils by

Handheld Fourier Transform Near-Infrared Spectroscopy; Anal. Chem. 2023, 95.

ACKNOLEDGEMENTS: The authors gratefully acknowledge the University of Duisburg-Essen for providing the infrastructure and support, as well as the collaboration with Barry Wise.

Keywords
TPH,Handheld NIR,LW-PLS,Soil,Global calibration

SO.07.03

MODEL TRANSFERABILITY ACROSS A PORTABLE NEAR-INFRARED SPECTROMETER FLEET

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trinamiX GmbH, Ludwigshafen, GERMANY

Portable near-infrared (NIR) spectrometers are increasingly being adopted for on-site quality analysis across diverse applications. A significant challenge for the broader acceptance of these portable devices lies in minimizing instrument-to-instrument variations within a large fleet of (affordable) spectrometers. In this study, we present findings on the transferability of chemometric models within a fleet of over 50 portable grating spectrometers (trinamiX PAL Two; wavelength range: 1300 nm – 2350 nm; spectral resolution: 50 nm) and between this fleet and a benchtop spectrometer. We emphasize the critical role and interdependence of optical design, high-precision spectral calibration, and robust chemometric model training in achieving reliable results. We report on cross-device accuracy for both classification and regression applications, demonstrating the effectiveness of our approach.

Keywords
Portable NIR spectrometer,on-site quality analysis,transferability of models

F.07.19

UNVEILING THE COMPLEXITIES OF NIR SPECTROSCOPY WITH PORTABLE SENSORS: MANAGING VARIABILITY, ERRORS, AND DATA OPTIMIZATION

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The rapid evolution of miniaturized near-infrared (NIR) spectroscopy has unlocked new possibilities across diverse application fields, driven by its portability, speed, and minimal sample preparation requirements. However, with the growing adoption of these compact instruments, critical challenges related to data reliability, variability, and measurement errors have emerged. While significant attention has been given to the development of models for classification, quantification, and exploratory purposes, there remains a pressing need to address the underlying complexities of data quality. In this presentation, we will highlight the importance of understanding and managing multivariate errors and variability in spectroscopic data, which is essential to improving analytical robustness and predictive performance.

We will introduce a comprehensive approach that combines advanced error analysis workflows, allowing for a more informed and data-driven selection of preprocessing techniques. By examining how instrumental variability and sample characteristics influence measurement errors, this methodology provides valuable insights into the relationship between acquisition conditions and data structure. Visual tools, such as covariance and correlation matrices, will be presented as a straightforward and intuitive framework, allowing for the easy interpretation of complex data structures and enabling the identification of the most effective preprocessing pipelines tailored to specific analytical objectives.

This approach emphasizes the need to treat miniaturized NIR spectrometers as unique instruments, each requiring customized calibration and error management solutions. By systematically addressing variability and uncertainty, we aim to pave the way for more reliable and transferable applications, establishing a foundation for future advancements in real-time and on-site spectroscopic analysis. The strategies we propose not only refine current analytical practices but also foster the broader adoption of miniaturized NIR spectroscopy in fields where accuracy and reliability are crucial.

ACKNOLEDGEMENTS: BG and MM acknowledge the financial support from Progetto di Ricerca di Interesse Nazionale PRIN 2022 "Innovative multi analytical Characterisation of the influence of pAtina-coating inteRaction on anti-corrosive propErties–InCARE" (Project code: 2022895PTX), funded by the European Union–Next Generation EU, M4.C2.1.1, CUP D53D23009030006

JR acknowledge the financial support from the Spanish Ministry of Science, Innovation and Universities (MICIU), the State Research Agency (AEI), and the European Regional Development

Fund (ERDF), EU: PID2022-136649OB-I00

Keywords
portable sensors,multivariate error,data preprocessing,error covariance matrices,correlation error

F.07.20

HOW NIR SPECTROSCOPY COMBINED WITH CHEMOMETRICS CAN IMPROVE RMID AND COMPLIANCE VERIFICATION OF RAW MATERIALS IN TYRE INDUSTRY

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The correct identification of raw materials plays a key role in the quality control of the tyre industry production chain. Especially when dealing with a large number of raw materials having different chemical compositions, and with analysis performed by several operators in different plants, it is important to perform raw material identification (RMID) being as more accurate and automated as possible. In this context, near-infrared (NIR) spectroscopy is one of the most suitable techniques, as it is non-destructive, user-friendly and timesaving.

In the present study, performed in collaboration with Pirelli Tyre S.p.A., FT-NIR spectra (Perkin Elmer, Frontier NIR, 10000 – 4000 cm-1) were acquired for most of the raw materials used for tyre production in two of the main Pirelli Tyre's factories.

Exploratory analysis by means of principal component analysis (PCA) was performed on the data with the aim to investigate data structures and groupings within samples. Due to the huge amount of data collected, it was necessary to automate the exploratory analysis through the creation of a dedicated routine in the Matlab ® (Version 2022b, The MathWorks, Inc.) environment. This automation strategy includes incoming controls for each spectrum acquired, with the aim of detecting anomalous data and for the transformation of the measurement scale (from reflectance to pseudo-absorbance). If there are measurement replicates, the average spectrum is then calculated. Supervised classification strategies were then applied on this data, in order to define the class membership of raw material analyzed, in terms of both material specification and supplier.

With the aim to apply this strategy for the routine analysis performed in the context of quality assurance, a report including all steps of data import and processing is then generated.

A further improvement of this RMID protocol will be the development of predictive models in order to estimate chemical and physical characteristics of the raw material directly during the acceptance phase.

Once developed and validated, the proposed strategy will be implemented in a graphical user interface (GUI) created in the Python environment for an immediate application in the industrial production chain; its usage will not require the operator to have previous knowledge about spectroscopy and chemometrics.

Keywords
Raw Material Identification,Compliance verification,Tyre industry,Chemometrics

F.07.21

COMPOSITIONAL DATA METHODS APPLIED TO NEAR-INFRARED SPECTROSCOPY. AN EXAMPLE FOR IN FIELD PREDICTION OF SOIL ORGANIC CARBON

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Near infrared spectroscopy (NIRS) methods often use models settled as multivariate correlations between the spectra and the content of a component from a matrix, expressed as parts from it. These last data, constrained within a scale, belongs to a 'simplex' space, and there is codependence among the parts they represent. The name in Mathematics of these data is 'Compositional Data' (CoDa). Karl Pearson was the first to notice spurious correlations from indices used in measuring biological organs. These data have a compositional nature, which means they carry only relative information. However, the classical multivariate analysis methods are for the real space, which values are not within limits, independent, and comparable across samples. Thus, the special features of CoDa make conventional statistical and chemometrics methods prone to produce several distortions when used with them. Integrating specific mathematical treatments for CoDa into NIRS methods have been first time introduced for measuring the nutritional components of several food products.

Here are presented a different example for in field measuring soil organic carbon with an analogous approach. Soil organic carbon content (SOC) has an important role in modulating atmospheric CO2. Soil moisture (SM) changes supposes an important challenge to VisNIRS measuring SOC. The CoDa methods can integrate SOC prediction along with the other soil parts considered, including SM. Different soil compositional approaches were evaluated within EJP-Soil ProbeField Project (https://ejpsoil.eu/soil-research/probefield). ProbeField focused on proximal sensing techniques for SOC measurement. Different CoDa approaches for 6, 5, 4, 3, and 2 soil parts were assessed based on isometric logarithmic coordinates (ILR). The soil components considered were soil organic matter (SOM), SM, soil inorganic carbon (SIC), the textural fractions clay (C) and silt (S), and the rest of soil mass (Other). SOM was calculated from SOC by applying the conversion factor 1.724. The sand content was considered in all the approaches together the soil part 'Other'. The results provided correlation coefficients of the predictions swinging from r = 0.74 with the traditional PLS calibration to predict SOM, to r = 0.87 with the 4-Parts 'Clay' CoDa approach, which was the best. Therefore, the results highlighted the CoDa method evaluated improved the SOM predictions regarding the traditional PLS.

ACKNOLEDGEMENTS: The authors acknowledge to the European Joint Programme Soil the (EJP Soil) and to the Spanish National Research Council (CSIC) the funding allocated to the project ProbeField (A novel protocol for robust in field monitoring of carbon stock and soil fertility based on proximal sensors and existing soil spectral libraries), within the framework of which was developed the research presented in this study.

Keywords
CoDa,Compositional data,SOC,Soil organic carbon,VISNIR

IMAGING & REMOTE SENSING

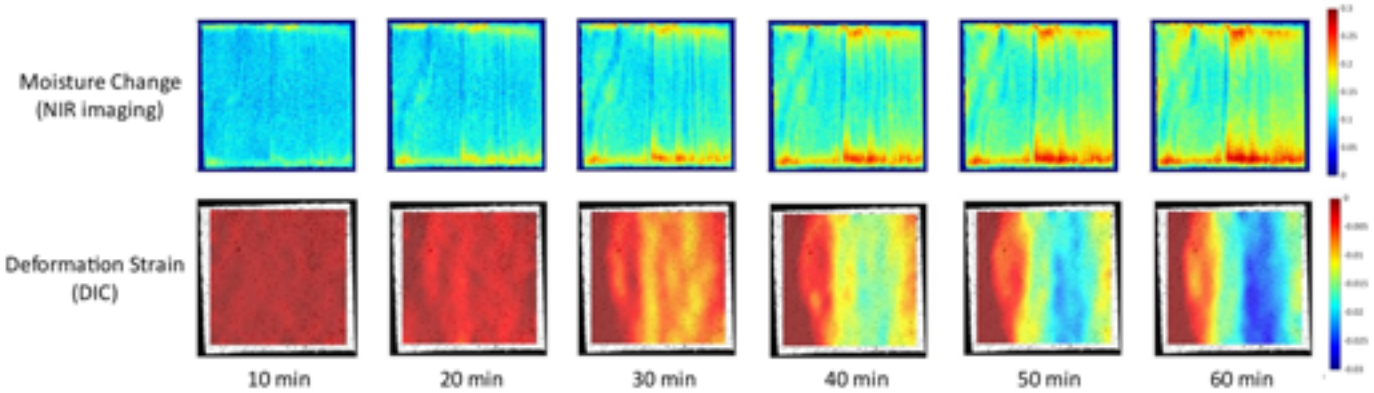
O.08.25

MONITORING SYSTEM FOR WOOD DRYING DEFORMATION AND MOISTURE CHANGE BASED ON DIC METHOD AND NIR IMAGING (970 NM)

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The pivotal role of wood in promoting a sustainable future is underscored by its renewable properties, carbon sequestration capabilities, and versatility across applications such as construction, furniture, and energy production. Proper drying of wood before use is essential. However, the uneven moisture distribution during the drying process can lead to deformation, negatively impacting durability and mechanical properties and significantly increasing energy consumption. Thus, to precisely control the drying process, improve drying quality, and reduce drying losses, it is crucial to monitor the drying process with a non-destructive method and investigate the relationship between moisture and deformation. We proposed a system that utilizes digital image correlation (DIC) and near-infrared (NIR) imaging at 970 nm to monitor deformation and moisture migration during wood drying simultaneously. To achieve this, ink visible only under visible light was applied to the wood surface to create an artificial random pattern. These patterns enable accurate measurement of wood deformation during drying without interfering with the NIR imaging. Our findings reveal the intricate details of wood's anisotropic nature and moisture motivation behaviors within and between annual rings. These insights lay a crucial foundation for advanced modeling, simulations, and predictions, ultimately facilitating the optimization of drying schedules and enhancing wood drying quality.



ACKNOLEDGEMENTS: This work was financially supported by JST SPRING, Grant Number JPMJSP2125. Zeng WP would like to take this opportunity to thank the "THERS Make New Standards Program for the Next Generation Researchers". This study is also supported by JSPS (KAKENHI, No. 24K09016 and 23K23670) and Emachu Wood Promotion Foundation.

Keywords
Wood drying,DIC,NIR imaging,Moisture,Non-destructive testing

O.08.26

EARLY DETECTION OF APPLE BRUISES AND PUNCTURES USING MULTISPECTRAL IMAGING INTERATED WITH DEEP LEARNING

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External defects on fruit surfaces vary in severity, ranging from superficial blemishes to those significantly affecting internal quality. Surface defects such as stem punctures and bruises, often caused during harvesting and distribution, can lead to secondary infections and overall quality degradation. Early detection and sorting of these defects are therefore critical for maintaining fruit quality. Recent advancements in deep learning have enabled automated sorting of defective fruits, improving quality assessment processes. However, detecting apple bruises remains challenging with RGB imaging due to variations in peel color. Near-Infrared (NIR) imaging has proven highly effective in overcoming these limitations, enabling precise identification of bruises. In this study, a 3-CMOS multispectral imaging system covering the 400–1000 nm range, combined with LED illumination, was used to capture RGB and NIR images of apples with various defects. Deep learning models were applied to successfully identify bruises, including those not clearly visible in RGB images. The results demonstrated that NIR imaging was more effective than RGB imaging in detecting both bruises and punctures, confirming its superiority for identifying subtle surface defects. Our findings highlight the advantages of multispectral imaging in detecting apple surface defects. The integration of multispectral imaging and deep learning offers a reliable solution for early defect detection and sorting, enhancing fruit quality management and reducing post-harvest losses.

ACKNOLEDGEMENTS: This work was supported by Korea Institute of Planning and Evaluation for Technology in Food, Agriculture, Forestry (IPET) through (Smart Agri Products Flow Storage Technology Development Program), funded by Ministry of Agriculture, Food and Rural Affairs (MAFRA) (No.322051052HD020)

Keywords
Multispectral Imaging,Deep Learning,Apple,External Defect Detection

O.08.27

NIR-HYPERSPECTRAL IMAGING FOR AUTOMATED IDENTIFICATION OF PUNCTURES ON PEARS: FROM IMAGE ANNOTATION TO PIXEL-LEVEL CLASSIFICATION

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Brown Marmorated Stink Bug (BMSB) has recently emerged as an invasive insect pest of global importance [1]. Indeed, BMSB punctures on pears determine the occurrence of internal damages to the fruit pulp, which are not visible to the naked eye. If not promptly detected, these damages may promote the spread of pathogens compromising the freshness and marketability of sound fruits during storage.

To face this issue, Near Infrared Hyperspectral Imaging can be employed during post-harvest sorting as an automated method for early detection of BMSB damaged pears.

In this study, organic pear fruits of two varieties (cv. Williams and cv. Abate Fétel) were collected during summer 2022 and summer 2023. About half of the fruits were exposed to BMSB for one week before harvesting, while the remainder were used as control samples. The hyperspectral images were acquired in the 1156-1674 nm range at eight subsequent times for six weeks from the day of harvesting. In total, about 2000 hyperspectral images were acquired for each variety.

For the development of classification models able to discriminate damaged areas it is firstly necessary to annotate the hyperspectral images to have the ground truth. This task was very challenging due to the subtle spectral differences between sound and punctured regions and to the huge amount of acquired images. To face the issue, an innovative method based on hyperspectrograms, an image data dimensionality reduction method, and image-level classification coupled with spatial features selection was developed to automatically and objectively annotate punctured areas [2, 3]. This method allowed the correct annotation of punctures in the original images' domain on 89.7% and 80.8% of punctured fruits of cv. Williams and cv. Abate Fétel, respectively.

Thanks to this preliminary annotation step, a library of representative spectra belonging to both punctured areas and sound fruits was built for the development of supervised pixel-level classification models and to select the most relevant spectral regions for discrimination. To this concern, sparse Soft Partial Least Squares Discriminant Analysis (s-Soft PLS-DA) algorithm [4, 5] led to satisfactory results both in cross-validation and external validation. Finally, the s-Soft PLS-DA models were applied to the acquired hyperspectral images and the corresponding prediction images were used to perform the final fruit-level class assignment to sound or punctured classes. Furthermore, the spectral regions selected by s-Soft PLS-DA can be viewed as a first step toward the development of multispectral imaging devices to be implemented in post-harvest sorting systems for automated identification of internal damages on pears.

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ACKNOLEDGEMENTS: HALY.ID project, ERA-NET Cofund ICT-AGRI-FOOD, with funding provided by national sources (Ministero delle politiche agricole e forestali, MIPAAF) and co-funding by the European Union's Horizon 2020 research and innovation program, Grant Agreement number 862671.

Keywords
Hyperspectral imaging,NIR,punctures detection,BMSB,pear

O.08.28

DETECTION OF PESTICIDE RESIDUES IN TABLE GRAPES BY MEAN OF HYPERSPECTRAL IMAGING

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The detection of pesticide residue levels is critical for food safety assessment. The traditional detection methods are time-consuming and laborious while non-destructive detection makes the process rapid and ensures quality. This research focuses on the time-series hyperspectral imaging of red grapes to classify pesticide residue levels with the passage of days. A combined solution of two pesticides, "Flint" (Bayer) and "Switch" (Syngenta) was used in a ratio of 1:5.33 in water; the initial solution was then diluted into the required low levels of concentration. The pesticide solution was applied in the lab, by spray, simulating the field conditions. Five concentrations were prepared diluting the initial solution 100 % (T1), by 80% (T2), 60% (T3), 40% (T4) and 20% (T5). After the spray, the grapes were stored at 0 ? C. The hyperspectral images were taken with the hyperspectral line-scan scanner (Version 1.4, DV srl, Padova, Italy) with two sensors, in the visible near-infrared (Vis-NIR) range from 400 to 1000 nm and near-infrared (NIR)from 900 to 1700 nm. The hyperspectral images were taken 24 hours, 8 days, 15 days and 19 days after treatment. For each day 10 grape berries were taken from each treatment so 200 samples were imaged. After that berries were frozen for further chemical analysis of residues. For classification, the data set was divided into five classes according to treatments, 100% (class1), 80% (class2), 60%(class3), 40% (class4) and 20% (class5). The partial least square discriminant analysis (PLSDA) method was used for each sampling. The Vis-NIR spectra didn't discriminate well among classes and showed accuracy for most classes lower than 70%, while the NIR spectra showed better discrimination for most classes. The cross-validation (CV) accuracy for treatments after 24 hours was lower than 70% while for the remaining days the average CV accuracies for T1, T2, T3, T4 and T5, were 98%, 88%, 75%, 70 and 94%, respectively. Pesticide analysis is still running but the results show that the pesticide residue could be detected at very low concentrations, even after 19 days from the application, suggesting that prediction may also be achievable. This research could enhance the non-destructive detection of pesticide levels on grapes and the regression models for the degradation of pesticide residue over time would help the quality assessment for industries and the market.

Keywords
pesticide residue,hyperspectral imaging,grapes quality assessment,non-destructive detection,Vis-NIR and NIR spectroscopy

F.08.22

HYPERSPECTRAL IMAGING FOR FIRE PERFORMANCE CLASSIFICATION OF WOOD FAÇADES

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The use of wood as a sustainable building material requires reliable fire safety assessment, particularly for façade applications where external conditions can compromise the efficacy of fire-retardant treatments[1]. While European standards classify construction products into Euroclasses based on reaction to fire performance, current testing methods such as cone calorimeter, though informative, are destructive and do not enable continuous monitoring. This study introduces hyperspectral imaging as a non-invasive approach for classifying wood's Euroclass ratings under simulated façade conditions. Following established classification criteria[2,3], we evaluated fire-retardant-treated wood samples targeting specific Euroclass thresholds: Class B (peak heat release rate <=100 kW/m^2 for >=900s, time of ignition >=40s), Class C (<=180 kW/m^2, >=30), and Class D (<=250 kW/m^2, >=15). Using short-wave near-infrared hyperspectral imaging combined with partial least squares discriminant analysis, we developed a predictive model correlating spectral information to these Euroclass assignments. Our methodology included chemical modification of wood samples using commercial fire-retardant reagents, followed by simulated outdoor exposure through water leaching and drying cycles. Continuous mass loss measurements quantified reagent leaching, while mass loss calorimetry assessed fire protection effectiveness. The developed model successfully classified wood samples into their respective Euroclasses using non-destructive imaging techniques, demonstrating the potential for real-time monitoring of fire-retardant treatment effectiveness in wooden façades. This digital solution advances sustainable building practices by enabling continuous assessment of fire safety performance without destructive testing, contributing to improved fire safety management in wooden structures.

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ACKNOLEDGEMENTS: I would like to express my sincere gratitude to the staff members of the Mechanical Workshop at NMNU for their invaluable technical support and expertise throughout this work. I am also deeply grateful to the staff members at NIBIO for their cooperation, technical assistance, and insightful suggestions that significantly contributed to this research.

Keywords
Hyperspectral imaging,classification,wood façades,fire-retardant treatment

F.08.23

DETECTION AND IDENTIFICATION OF MICROPLASTICS IN SEA SALT BY SWIR-HSI

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Microplastics represent more than 80% of the waste present in the seas, becoming one of the main problems today [1]. These not only affects to marine ecosystems, but also these have a significant and indirect impact on the humans, since these plastic fragments accumulate in species such as mussels, fish, and other marine animals that are consumed by humans, creating a potential health risk.

This is one of the reasons because they have been categorised as the 10th greatest threat in the World Economic Forum's Global Risks Report 2024 [2]. To meet this challenge, different international organizations have promoted action plans to monitoring, mitigate and prevent these pollutants [3]. Aligned with these initiatives, the presented study aims to develop analytical methods based on the application of hyperspectral imaging (HSI) together to machine learning methods to monitor the presence of these pollutants in the Mediterranean Sea, specifically in the region of Almeria (Spain). For this purpose, salts sampled from solar saltworks instead of Mediterranean seawater were analysed, since solar saltworks act as a high-scale natural 'pre-concentrators' of solid pollutants in water, including microplastics, thus salts can be considered as an indicator of their presence in marine environment.

SWIR-HSI was applied in reflectance mode in spectral range (900 to 1700 nm).Three kinds of materials were considered: (i) salts from inland saltworks, which have been considered as 'blank salt', i.e. microplastics-free, due to its origin; (ii) salts from coastal saltworks considered as polluted salts; (iii) standard plastic materials which were selected according to the plastic most commonly found in Mediterranean Sea (PE, PET, PS, PP, PVC) [4]. All of them were crushed to reach microplastics size and to homogenise particle size. Samples of above-mentioned materials were analysed and as a result, a hyperspectral image of each one was obtained. Then, the region of interest (ROI) of each sample was selected. As exploratory analysis, principal component analysis (PCA) was performed using the ROI average spectrum of blank salt and standard materials samples. In addition, partial least square discriminant analysis (PLS-DA) was applied to build a classification model capable of discriminate between ROI pixel-spectral which contain salt or any kind of microplastic. Once the model was trained and validated it was applied to detection and identification of microplastics in salts from coastal saltworks.

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ACKNOLEDGEMENTS: AMJC acknowledges the Grant (RYC2021-031993-I) funded by MICIU/AEI/501100011033 and 'European Union NextGeneration EU/PRTR'.

Funding: Grant PCM-00042, funded by Consejería de Universidad, Investigación e Innovación and Gobierno de España and Unión Europea – NextGenerationEU.

Keywords
HSI,Environmental analysis,microplastics,Chemometrics

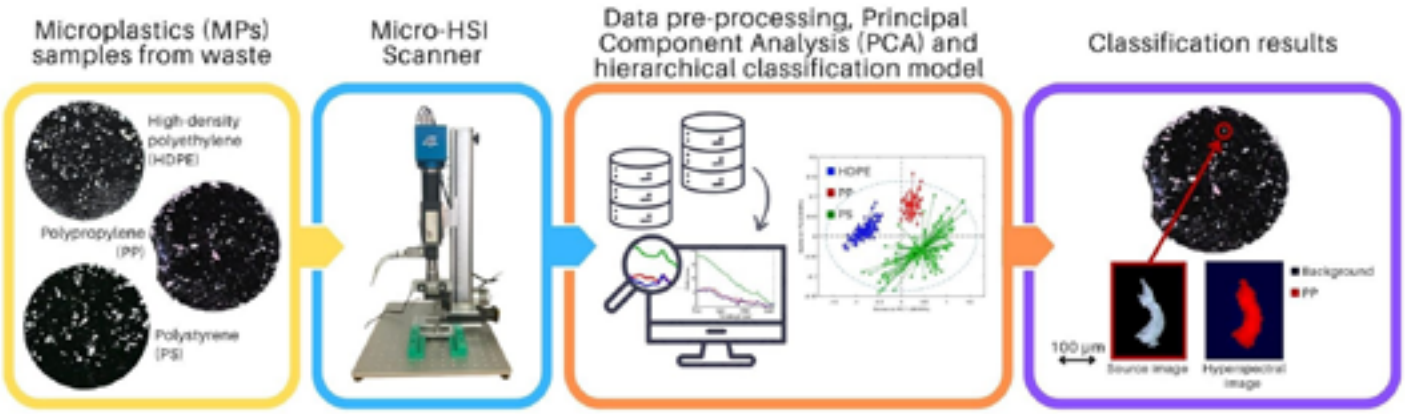
F.08.24

IDENTIFICATION AND CLASSIFICATION OF MICROPLASTICS BY A MICRO-HYPERSPECTRAL IMAGING SCANNER PROTOTYPE

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Plastic pollution has emerged as a critical global issue, exerting significant ecological impacts on both aquatic and terrestrial ecosystems. However, the detection of microplastics (MPs), i.e., particles smaller than 5 mm, remains a complex task due to their microscopic size and diverse chemical compositions. Hyperspectral imaging (HSI) has recently gained attention as a powerful technique to address this challenge, offering precise differentiation of MPs by leveraging their distinct spectral signatures. Moreover, HSI integrates imaging with spectral analysis, enabling rapid identification and significantly reducing processing times compared to conventional spectroscopic methods. In this study, a novel approach for the rapid detection and classification of MPs using a custom-built near-infrared (NIR: 1000–1650 nm) micro-spectral scanner is proposed. The innovative micro-HSI spectral system, specifically designed for MPs recognition, combines hyperspectral technology with microscopy to achieve a spatial resolution of 10 µm/pixel. The scanner's sample holder is mounted on a motorized 2-axes system that moves in the X and Y directions, enabling the scanning of areas up to 20 x 26 mm. The scanning process involves parallel acquisitions with a 3.37 mm field of view (FOV) of the IR camera, which are automatically mosaicked by the acquisition software. To evaluate the performance of the micro-HSI device, reference samples were prepared by grinding three common polymer types typically found in marine and terrestrial environments: polystyrene (PS), polypropylene (PP), and high-density polyethylene (HDPE). The obtained MPs, ranging in size from 1 mm to 30 µm, were collected on nine adhesive carbon tabs (three tabs per polymer type) with a diameter of 1 cm. Hyperspectral images of the samples were acquired and pre-processed to enhance spectral features and reduce noise caused by light scattering and MPs morphology and then evaluated using Principal Component Analysis (PCA), with particular attention to spectral variations as particle size decreased. Based on the features identified by PCA, a hierarchical classification approach was developed to automatically and rapidly identify MPs, achieving accurate classification for particles greater than 100–150 µm, confirming the device's ability to detect a wide range of MP sizes with high precision. The results demonstrated that the innovative micro-HSI system, when combined with a dedicated classification models, enabled significantly faster analysis of MPs compared to conventional techniques such as micro-Fourier Transform Infrared Spectroscopy (micro-FTIR). Additionally, the system's configuration supports automated scanning and mosaicking, further reducing analysis time. This advancement can be a starter point to improve the micro-HSI system for efficient, high-resolution MPs analysis, paving the way for broader applications in environmental monitoring and plastic pollution research.



ACKNOLEDGEMENTS: The research was carried out in the framework of the Enlarged Partnerships supported under the National Recovery and Resilience Plan (NRRP), Mission 4, Component 2, Investment 1.3 funded by the European Union–NextGenerationEU (RETURN project "Multi-risk science for resilient communities under a changing climate", no. PE00000005, CUP B53C22004020002.

Keywords
Hyperspectral imaging, Near-infrared spectroscopy, Micro-spectral scanner, Microplastics identification, Environmental monitoring

FUTURE TRENDS

O.09.29

CHARACTERIZING AND QUANTIFYING ANISOTROPY IN MOZZARELLA CHEESE BY SPATIALLY OFFSET SHORTWAVE NIR SPECTROSCOPY

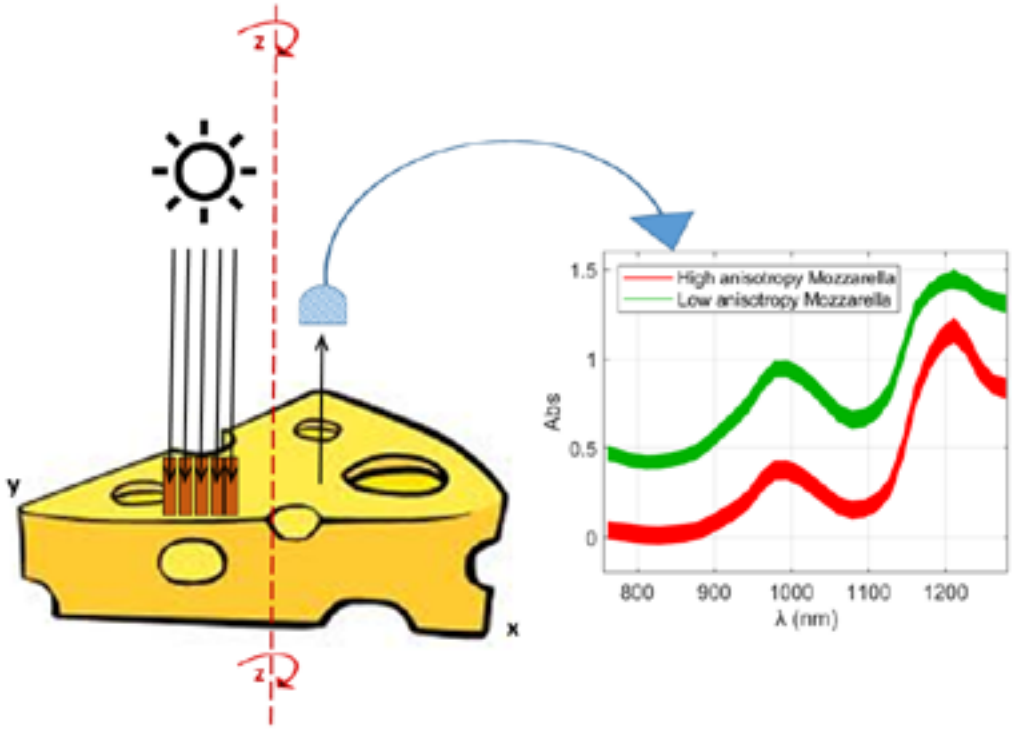
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Cheese is a widely consumed dairy product, valued for its diverse attributes shaped by biochemical and mechanical processes, such as protein and lipid hydrolysis, fermentation, and maturation, which contribute to various cheese types with distinct properties. Mozzarella cheese is distinguished by its unique functional properties, i.e. meltability and stretchability, which are essential for culinary applications such as pizza and baked dishes. These properties are attributed to the pasta-filata process. During this process, coagulated milk particles are heated in a cooker-stretcher, causing partial melting and increased flowability of the proteins. These partially melted proteins undergo reorganization due to shear forces, leading to an anisotropic structure in mozzarella. This anisotropic structure refers to the alignment of molecular structures along specific directions, while an isotropic structure is characterized by the absence of such directional preference. Anisotropy in mozzarella is evident and extensively studied, influencing its functional and sensory properties. Various techniques have been developed to characterize anisotropy, but most are destructive, non-reproducible, slow, and unsuitable for continuous monitoring [1].

This study investigates the anisotropy of a set of 19 mozzarella samples, using a prototype short-wave NIR instrument in a non-contact, spatially offset interactance mode [2]. In a previous study, this sample set, which was manufactured with varying process parameters and additives has been analyzed for anisotropy using fluorescence anisotropy, X-ray tomography, oblique incidence reflectometry, and functionality tests [3]. NIR measurements were performed along two axes – parallel and perpendicular to the fiber direction – in five replicates. Mozzarella samples exhibiting minimal anisotropy showed no significant differences between the two orientations, leading to overlapping scores in a principal component analysis (PCA) of the NIR spectra. Conversely, mozzarella with pronounced anisotropy displayed substantial spectral offset differences between the two axes, resulting in distinct separation in the PCA score plot. In addition, the spatially offset NIR spectra were measured by rotating the mozzarella samples during data acquisition (Figure 1). Mozzarella with high anisotropy typically exhibits a defined fiber direction. As a result, when the cheese is rotated, in two orientations (i.e. 0° and 180°), the same axis is aligned with the light source, while at the perpendicular points (i.e. 90° and 270°), fewer fibers are aligned with the light source. This produces a characteristic sinusoidal variation in intensity when rotating the sample during measurement. The sine waves in intensity were further analyzed as polar plots, to examine the shapes and consistency. This analysis revealed that the mozzarella samples with the highest anisotropy exhibited a relatively constant radius in the polar plot, whereas the mozzarella samples with the lowest anisotropy displayed a varying radius. A similar pattern was observed for an isotropic cheese, which showed a more irregular radius distribution. Furthermore, the sinusoidal attributes were quantified using sine model fitting, with the anisotropic cheese showing the best fit and less residual compared to the isotropic cheese.

This study presents a novel method for quantifying anisotropy using spatially offset NIR, linking observed differences to elongated protein structures in anisotropic cheeses. It is especially valuable for those characterizing and replicating food structures.



ACKNOLEDGEMENTS: The authors gratefully acknowledge Innovation Fund Denmark for their support through the Exchequer project grant and thank the Arla Innovation Centre for providing the cheese samples.

Keywords
Mozzarella cheese, Anisotropy, Isotropy, Cheese functionality, Spatially Offset Shortwave NIR

O.09.30

REBUILDING SCIENCE-BASED NIR SPECTROSCOPY BY FOOD METABOLOMICS

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Nondestructive near-infrared spectroscopy (NIRS) has begun to predict subjective sensory indicators such as sweetness, umami, and freshness of fruits and vegetables. However, NIRS is often performed as a "black-box" along with chemometrics to develop calibration models to accurately predict these indicators, without knowing the measurement basis. This lack of comprehension makes the models difficult to interpret. To ensure reliable application in practice, it is crucial to demystify this "black-box" and gain a deeper understanding of the NIRS working mechanism. Therefore, we aim to accurately estimate food quality using NIRS and reveal the working mechanism of NIR calibration models through food metabolomics, i.e., to develop science-based NIR spectroscopic methods by food metabolomics for food quality assessments. Two NIR studies on the estimations of tomato sensory quality and broccoli freshness are briefly introduced here.

The first study explored the ability of NIRS to assess the sensory qualities of intact tomatoes by a combination of PLSR analysis and the stepwise selectivity ratio (SWSR) method. Acquired NIR spectra of tomatoes were used to predict the sensory scores evaluated by trained panelists for each sensory attribute such as sweetness, umami, and firmness. PLSR models built based on the informative wavelengths (IWs) selected by SWSR method accurately predicted 8 sensory attributes, particularly sweetness, with a correlation coefficient of validation as high as 0.92. The selected IWs could be associated with the presence of sugars, fruit acids, and amino acids. For further investigation, GC-MS metabolomics analysis was performed on the same tomato samples. High intercorrelations between sensory scores, the amounts of metabolites, and spectral absorbances of IWs were found through PCA. Figure 1 shows the results for sweetness as an example. Metabolites and IWs that are close to the sweetness sensory attribute in the PC loading plot had high absolute correlation coefficients ($|r| > 0.9$). The presence of fructose and glucose explains the perceived sweetness, although the fruit acids levels in tomatoes also affect sweetness. Glutamic acid and aspartic acid are MSG-like amino acids responsible for umami taste, which were found to contribute to sweetness. Taken together, the results confirmed the feasibility and reliability of Vis-NIRS and the IWs selected by SWSR in predicting the sensory qualities of intact tomatoes.

A similar research approach was applied to the study on the assessment of broccoli freshness. Vis-NIR spectra of broccoli were collected as the predictor, and cumulative temperature of broccoli under different storage conditions was calculated as a freshness indicator. An accurate predictive model ($R^2p=0.75$) for broccoli freshness was built using IWs by combining PLSR analysis and SWSR method. Through data fusion of NIR and NMR, strong correlations ($|r| > 0.8$) between the NIR and NMR signals of broccoli revealed that NIR protein absorbance decreased during broccoli storage, while the levels of amino acids such as phenylalanine increased (Figure 2). Protein degradation into amino acids due to vegetable senescence, detected as a change in NIR absorption, can be used to interpret the working mechanism behind the freshness prediction model.

These studies indicate that NIRS combined with chemometrics has great potential in assessing food quality. Food metabolomics performed here helps to interpret the informative spectral WLs and demonstrate the reliability of the predictive model. Combining NIRS with food metabolomics provides a promising approach to opening the "black-box" of NIRS in different studies and rebuilding science-based NIR spectroscopy.

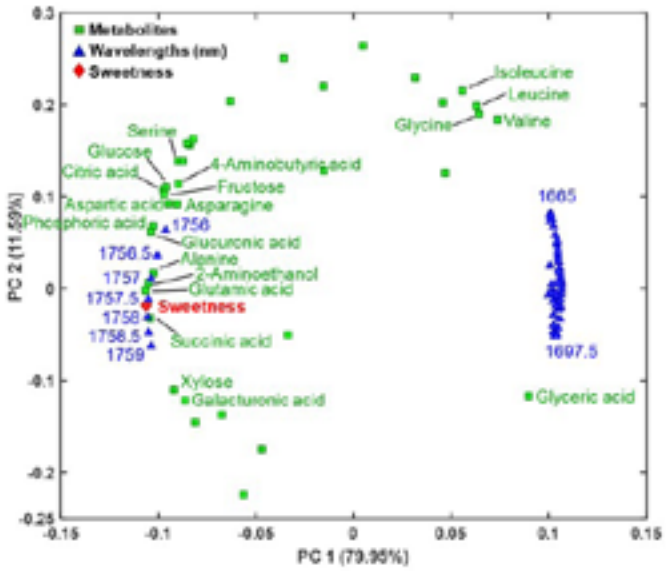


Figure 1. PC loading plots for sweetness attributes. The intercorrelations between the sweetness sensory score, the amounts of metabolites, and the spectral absorbance of the selected informative wavelengths were analyzed by PCA. Variables with high absolute correlation are labeled. (X. Li, et al. *Food Chemistry*, 2021, 343, 128470).

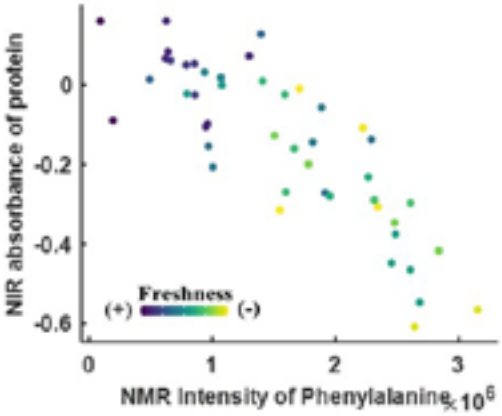


Figure 2. Correlations between the NIR signals of protein, NMR metabolite signals of phenylalanine (as an example), and freshness of broccoli. Samples distributed in the plot were colored with their corresponding freshness. (X. Li, et al. *Postharvest Biology and Technology*, 2024, 211, 112810).

ACKNOLEDGEMENTS: Supported by NEDO project and JSPS KAKENHI (22K20606 & 24K09171).

Keywords
Tomato sensory quality,Vegetable freshness,Measurement basis,Variable selection,Data fusion

O.09.31

INNOVATIVE AQUAPHOTOMICS APPROACH TO NON-INVASIVE DIALYSIS MONITORING

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Introduction

Dialysis is the primary treatment for advanced kidney failure. Recently, UV spectrophotometric sensors and near-infrared (NIR) spectroscopy have been introduced for on-line monitoring of haemodialysis sessions [1,2,3]. While urea is the only uremic solute monitored in spent dialysate, NIR spectra's complexity and water's strong absorption pose challenges [3]. Aquaphotomics [4], a discipline based on NIR spectroscopy, uses water spectra as valuable information sources. Unlike previous studies that removed water signals from UV or NIR spectra, the approach of this research preserves the original signal, including water, in alignment with aquaphotomics principles.

Method and Material:

The study included 18 dialysis treatments across 10 patients (4 females, 6 males) undergoing chronic weekly haemodialysis at the Department of Nephrology, Clinical Centre Zemun, Belgrade, Serbia. Dialysis machines used were HdF100s, F70s (Fresenius Medical Care, Germany), and Polyflux 170H (Gambro Polyflux®, Sweden). Additional dialyzer details are provided in Table 1.

Table 1. Dialyzers data

		HdF100s	F70s	170H
Clearances [ml/min] Qd=500ml/min	Urea	271-354	190-245	196-321
	Creatinine	252-329	177-220	186-281
	Phosphates	240-315	174-216	180-266
	Vitamin B12	190-258	127-145	137-178
Blood flow [ml/min]		300-600	200-500	250-500
Dialysate flow [ml/min]		1000	1000	800
Effective surface [m²]		2.3	1.6	1.7

The time of haemodialysis treatment varied between 180 and 270 minutes, the dialysate flow was between 800 and 1000 mL/min, and the blood flow varied between 200 and 600 mL/min. The Ethics Committee approved the study protocol and written informed consent was obtained from all patients.

The samples of spent dialysate were taken from the drain tube of the dialysis machine (Figure 1) after 5, 45, 90 and 135min of each dialysis session. The samples were sent to the laboratory and the spectral acquisition was performed using double beam spectrophotometer Lambda 950 (Perkin Elmer Italy), in the range 190nm-1700nm with the resolution of 2nm. A quartz liquid sample cell (1 mm path length) was used as a container.

For all 18 dialysis sessions, 4 samples were collected at planned times (50ml) and recorded three times giving in total 216 spectra.

Results and Discussion

The NIRS method has been proposed for measuring urea in spent dialysate, a common marker for accumulated waste solutes in dialysis patients, as urea absorbs in this region [3]. This study focuses on monitoring water cleanliness instead of urea or other waste materials.

Spent dialysate was collected from 18 patients during three haemodialysis sessions at 5, 45, 90, and 135 minutes of treatment. NIR absorbance spectra were analyzed using multivariate analysis and the Aquaphotomics approach. The results (Figure 2) showed that absorbance spectra of spent dialysate could be effectively classified by therapy duration, even by monitoring water absorbance at 1410 nm (free water molecules [4]).

Continuous measurement using online monitoring devices could provide new tools for more efficient dialysis treatment [3].

Acknowledgments: Author is grateful to the patients and the staff of the Department of Nephrology, Clinical Centre Zemun, Belgrade, Serbia for their collaboration, as well as Nanolab, Biomedical Engineering Department., Faculty of Mechanical Engineering, University of Belgrade for providing the equipment and measurements.

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FS.09.04

FROM COMPLEXITY TO SIMPLICITY: THE SHIFT TOWARD SMART MINIATURIZED NIR SYSTEMS

Meltem Yildirim

Hellma GmbH & Co. KG, Müllheim, GERMANY

Smart NIR sensors are evolving rapidly - moving from bulky, fiber-based setups to compact, miniaturized devices with embedded intelligence. This shift is not just about size; it marks a fundamental transformation in how we monitor and control processes in real time. This presentation will discuss the developments in miniaturized NIR technologies, highlighting both their potential and the technical factors that influence their adoption.

FS.09.05

SPEKTRUM: UPDATES ON STANDARDISATION OF SPECTROSCOPIC DATA ACQUISITION SOFTWARE

Brad Swarbrick

KAX Group, Penrith, Australia

This talk provides an update on the concepts presented during NIR 2023 in Innsbruck. A presentation of the software developed will highlight the important features required for a standardised platform for connecting any spectrometer to a simple user interface and access to chemometric modelling and model deployment.

The development and promotion of a single user interface for all instruments translates to easier implementation of technology, particularly in regulated environments and thus provides a means for improved technology adoption and the ability to compare technologies in one all-encompassing software environment.

FS.09.06

HYPERSPECTRAL IMAGING IN PHARMACEUTICALS: ENHANCING PRODUCT ANALYSIS AND OVERCOMING LIMITATIONS FOR INLINE INSPECTION

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The integration of hyperspectral imaging (HSI) in pharmaceutical development heralds a paradigm shift, particularly in the analysis and quality control of tablets, powders, and even crystallization processes. It provides rich and detailed data that goes beyond what is captured by standard imaging techniques thanks to the combination of spatial and spectral dimension (Hypercube). Standard application of HSI offers for example non-destructive, rapid, and comprehensive analysis of tablets (API, Homogeneity). The versatility of HSI extends to detecting physical and chemical deviations, predicting shelf-life, and even in counterfeit detection. Overall, the application of hyperspectral imaging in pharmaceutical development not only enhances product quality and safety but also streamlines manufacturing processes, thereby accelerating the journey of new pharmaceuticals from lab to market

Hyperspectral imaging, however, can have certain limitations when it comes to applying the technology in high-speed online settings or in liquid processes for in-situ measurements. To meet these needs, it is also possible to use fiber optic probes (SAM-Spec technology) capable of remote multipoint measurement at high speed. The presentation will show how HSI can enable laboratories to save time and gather more information on products, but also how it is feasible to ensure high-throughput measurement of tablets (with PLS modelling for hardness, API homogeneity at 100,000 tablets per hour) or the measurement of low concentrations of powder in feedframes.

ACKNOLEDGEMENTS: This work is based on Industrial and university partnership and we would like to thanks more specifically :

Rurgers University

Gent University

University of Montpellier

GSK (USA)

UCB

Pharma Technology

Pr. Rudolf Kessler

Keywords
Hyperspectral Imaging,Spatialy resolved spectroscopy,Tablet,Lyocake,PAT

F.09.25

APPLICATION OF MICRONIR TECHNIQUE FOR RECREATIONAL WATERS MONITORING

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[1] *University of Rome Foro Italico - Department of Movement, Human and Health Sciences, Rome, ITALY,* [2] *University of Rome Sapienza - Department of Chemistry, Rome, ITALY*

Recreational waters intended for use in swimming pools and similar facilities may contain a complex mixture of organic and inorganic compounds, and microorganisms. Pathogens and the associated biological risks can be reduced by appropriate disinfection treatments. Such treatments, causing the formation of disinfection by-products (DBP), increase the chemical risk leading to the need for its surveillance and containment. There are no international Regulations on recreational waters, but just WHO Guidelines [1] and several regulatory guides at national levels [2, 3], which recommend monitoring among the multiple existing contaminants, only for some bioindicators, i.e. E. coli, P. aeruginosa, and chemical parameters, i.e. combined and free chlorine. The guides regulating DBPs in recreational waters are limited and focus only on trihalomethanes among nearly 900 existing DBPs, whose control seems to be crucial also due to their toxicity [4].

Technological leaps have led to the development of high-performance cultural and molecular microbiological techniques and analytical methods for DPBs determination. These methods, i.e. GC-MS, are laborious, expensive equipment and require complex procedures for sample pre-treatment and analysis, expensive instruments and highly qualified personnel. Therefore, they are not suitable for timely assessment of recreational waters safety.

This work aimed to evaluate the feasibility of applying NIR spectroscopy for recreational water safety surveillance, given its advantages, such as rapidity, non-destructiveness, no toxic solvents required for sample handling and given the demand for techniques capable of producing real time reliable results [5, 6].

NIR spectroscopy operating on a miniaturized platform (microNIR) was applied for the monitoring of microbiological indicators and chemical contaminants in recreational waters. The technique applicability was studied through two systematic reviews, conducted according to the PRISMA guidelines and focused on existing methods for DPBs determination in recreational waters and on the NIR application for aqueous matrices. Laboratory tests were performed with a portable microNIR Pro and OnSite-W instruments on mock water samples artificially spiked with microbiological contaminants such as E. coli, Pseudomonas, Enterococci and/or on the water solutions with DBPs such as chloroform.

The feasibility of a microNIR approach applied to recreational waters was pointed out by systematic reviews on the state of the art. Experimental data on mock samples were performed by means of chemometrics, supporting the applicability of microNIR technique for the determination of biological and chemical contaminants in recreational waters.

New perspectives are outlined for the application of microNIR spectroscopy combined with chemometrics as a complementary technique to first-level methods for real time on-site microbiological surveillance and chemical monitoring of recreational waters.

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ACKNOLEDGEMENTS: Authors thank Foro Italico library staff

Keywords
recreational waters,microNIR,DBPs,microbiological parameters,THM

F.09.26

A PHYSICS-BASED DATA SIMULATION AND AUGMENTATION METHOD FOR ENHANCING CALIBRATION MODEL DEVELOPMENT ON A VISIBLE SPECTROMETER

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Spectrometer calibration is a hot topic for PAT industrials and researchers because of the difficulties and challenges it brings [1]. For industrial applications, a low number of calibration samples and/or limited variability often compromise the chemometric calibration model development and hence its performances. Data augmentation is gaining popularity for overcoming this issue on visible or near-infrared spectrometers, for instance by using Deep Learning [2], pure components estimation [3] or interpolation [4].

This work presents a new data augmentation method based on a mechanistic physics-based approach. The method uses an optics model to simulate realistic synthetic spectra. Those simulated spectra are then used to augment measured calibration sets. The study focuses on a data set made out of a small number of samples with a limited variability. Moreover, we study the extreme case where the independent test data is at the edge of the variability range.

The data set consists of 55 phantoms samples displaying varying absorption and scattering properties, inspired by those made by Watté and al. [4]. They were inspected in reflectance and transmittance mode simultaneously, thanks to a double-integration sphere and visible spectrometer setup. Synthetic spectra were computed according to the Kubelka-Munk model [5] with Saunderson correction. Two data augmentation modes were investigated, namely completion and superposition. The augmented data sets are compared with a baseline set, in terms of correlation (R^2) and prediction (SEP) accuracy, by training partial least squares (PLS) regression models. This study investigates the benefits of augmenting the calibration data with synthetic data generated from various calibration sets.

The proposed method shows encouraging results in the extreme case where the test data are at the edge of the variability range (extrapolation). The strengths and weaknesses of the method will be addressed.

This work contributes to the recent trends in development of intelligent instruments for the PAT industry.

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ACKNOLEDGEMENTS: This work was made possible through the support of both Indatech (Chauvin-Arnoux Group) and INRAE Montpellier.

Keywords
Optical Simulation,Data augmentation,Spectroscopy,PAT

F.09.27

COMPARING CHEMOMETRICS AND AI ALGORITHMS FOR TEMPERATURE AND SPECTRA PREDICTION WITH NIR SPECTROSCOPY

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Near-infrared (NIR) spectroscopy has become a fundamental tool for real-time monitoring and control of chemo-physical processes, particularly within industries as Process Analytical Technology (PAT). Changes in process parameters such as temperature, pressure, and concentration induce spectral variations that often require advanced analytical methods for proper interpretation [1,2]. Integrating chemometric techniques is essential to unravel these spectral changes and correlate them with process parameters. Traditional chemometric methods such as PCA and PLS regression have been widely used for this purpose. Simultaneously, the rapid development of artificial intelligence (AI) offers new routes for enhancing predictive accuracy and robustness.

In this study, we investigated the application of classical chemometric methods and novel machine learning algorithms to NIR spectra of water, liquid ammonia, and their mixtures. NIR spectra were acquired in an autoclave with an immersion transmission probe during controlled temperature ramps (up to 200°C).

We first established models for temperature prediction from the spectra of the pure components using PLS regression extending the experimental domain mostly studied in literature [3, 4]. These models were able to capture variations of the absorption bands upon change in temperature. We then extended our analysis by implementing machine learning techniques (such as kNN, SVM and XGBoost) and Neural Networks (NN) to the analysis workflow. The use of machine learning algorithms did not lead to any significant improvement in the predictions. The results of the predictions on the unprocessed data of water can be seen in Figure 1. A linear regression approach was found to be slightly better (RMSE = 3.28) in respect with other methods for predicting temperature from the un-treated data.

Traditional chemometric and ML algorithms can be applied also to predict spectra from process parameters [5]. These models were initially built for predicting spectra of water and ammonia. The use of a Neural Network approach with proper data pre-treatment allows to predict spectra from the temperature values for the different experiments. Figure 2 shows the predicted spectra of water where a NN architecture is used as algorithm (RMSE = 0.089).

The feasibility of the prediction of the spectra of single components suggests how this approach might be used also for the prediction of spectra of the mixture of the two components. Such an approach offers substantial benefits for process simulation and design, allowing the prediction of spectra corresponding to unknown conditions. The methodologies developed provide a framework for addressing complex monitoring challenges, paving the way for more intelligent and responsive process analytical technologies.

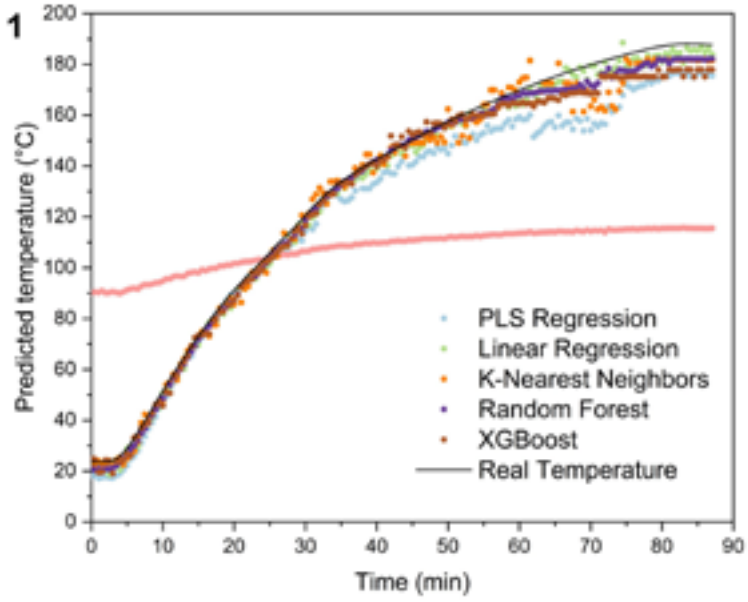
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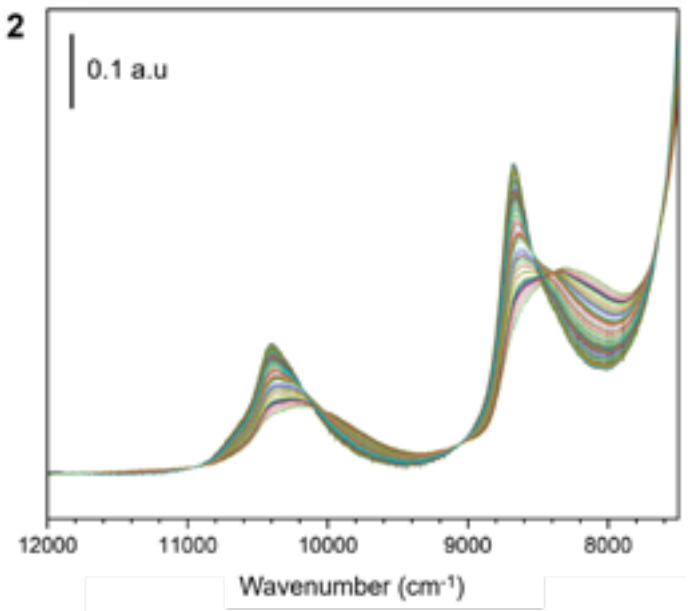
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ACKNOLEDGEMENTS: The authors gratefully acknowledge receiving funding from Innosuisse and Casale SA.

Keywords
NIR Spectroscopy,Machine learning,Spectra prediction,PAT,Online monitoring

ENVIRONMENTAL & SUSTENIABILITY

O.10.32

NEAR INFRA RED CALIBTRATION WOOD DENSITY: WHY IS IT SO HARD?

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Wood density is a key property that underlies the mechanical strength and stiffness of timber used in construction. Tree breeders aim to improve timber density, particularly of softwood species, but to do so requires phenotyping of large numbers, often thousands, of individual trees in replicated breeding trials. Near infrared spectroscopy is an obvious contender to provide rapid throughput of samples. But what is wood density and why is the standard error often large?

INTRODUCTION

Despite many published articles, there is still low accuracy in the predicted values of wood density, often 10-15% relative error. Why then is it so hard to calibrate NIR spectra with wood density? The mathematician Arnold Ross challenged students to "Think deeply of simple things", and this is how wood density should be considered.

Density is defined as the mass per unit volume of a substance. Water has a density of 1.00000 g cm-1 at 4.00 °C (or 0.99802 g cm-1 at 21 °C). That is the simple view of density.

Wood density, however, is determined and defined according to the state of the wood. When the wood is fresh from the tree, i.e. in the "green" state, it still contains water in the cell lumens. Determining the green volume and green mass yields the green density. But wood is never used in the green state and so it is typically dried to somewhere in the range 12-16 % moisture content. As such, the commonly used measure of basic wood density is the ratio of dry mass to green volume.

However, it must be noted that wood is a porous medium, and features such as ray cells, tracheid (cell) lumen, pits, and vessels contribute to the green volume, but do not contribute to the overall mass (Figure 1). Consequently, the mass of the unit volume is lower than it would be if the entire volume was comprised of wood material.

Sample preparation and presentation therefore has significant impact on the determination of wood density.

METHOD

Samples of Pinus radiata and Eucalyptus pellita wood were machined to ca. 20 mm x 20 mm x 20 mm (accurately measured by vernier caliper), then dried to ca. 14% moisture content and weighed. The sample volume was then re-measured in the dry state to allow for shrinkage that occurs during drying. Density was calculated as both green density (green mass / green volume), basic density (dry mass / green volume) and air-dry density (dry mass / dry volume). An adjacent radial strip, 20 mm long and 2 mm wide was also prepared for Silviscan analysis to provide X-ray density and morphological image analysis.

Near infrared spectra were acquired on each of the six faces of the dry cubic sample using a Bruker MPA with fibre optic probe, and then the specimens were ground to pass a 2-mm sieve and the NIR spectra recorded using the integrating sphere of the Bruker MPA.

Existing calibrations for wood chemistry (lignin, cellulose, total hemicellulose, extractives) were used to predict the wood chemical composition. Using literature values for density of the pure chemical components the density of the intrinsic wood material was calculated according to the chemical composition. This "wood matrix" density was used as a regressor in calibration development.

Partial least squares regression was used to develop calibrations for wood basic density using Vektor Direktor (KAX Group, Sydney, Australia) using various combinations of spectral averages of the individual faces, ground wood and the calculated "wood matrix" density.

RESULTS

The statistic of interest used for comparison was the standard error of prediction (SEP). Table 1 shows the differences in SEP with respect to the face used for spectral acquisition. This shows that when measuring density of wood, the face from which the spectra are acquired does have an impact on the error.

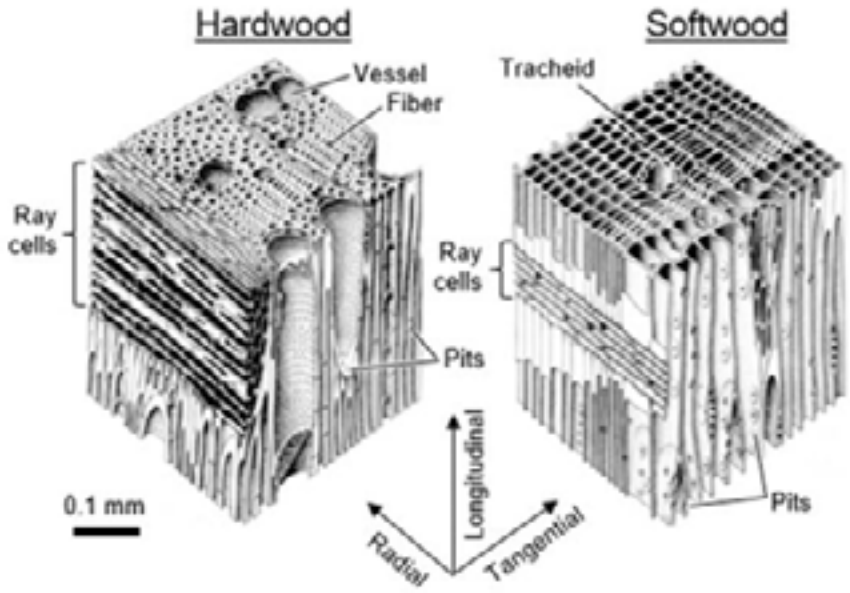


Figure 1. Schematic of hardwood and softwood structures. Source: Jakes et al. 2019[1]

Table 1. SEP for PLS regression calibration of wood basic density according to acquisition surface.

	1x Radial	1x Tangential	1x Transverse	Average of all 6 faces	Ground sample
Eucalypt	27.9	30.0	19.8	18.3	17.8
Pine	25.4	27.6	23.9	20.2	18.9

Keywords
Wood density,Wood chemical composition

O.10.33

INLINE NEAR-INFRARED SPECTROSCOPY FOR POLYMER PROCESSING -FOCUS ON POLYMER COMPOSITES APPLICATIONS

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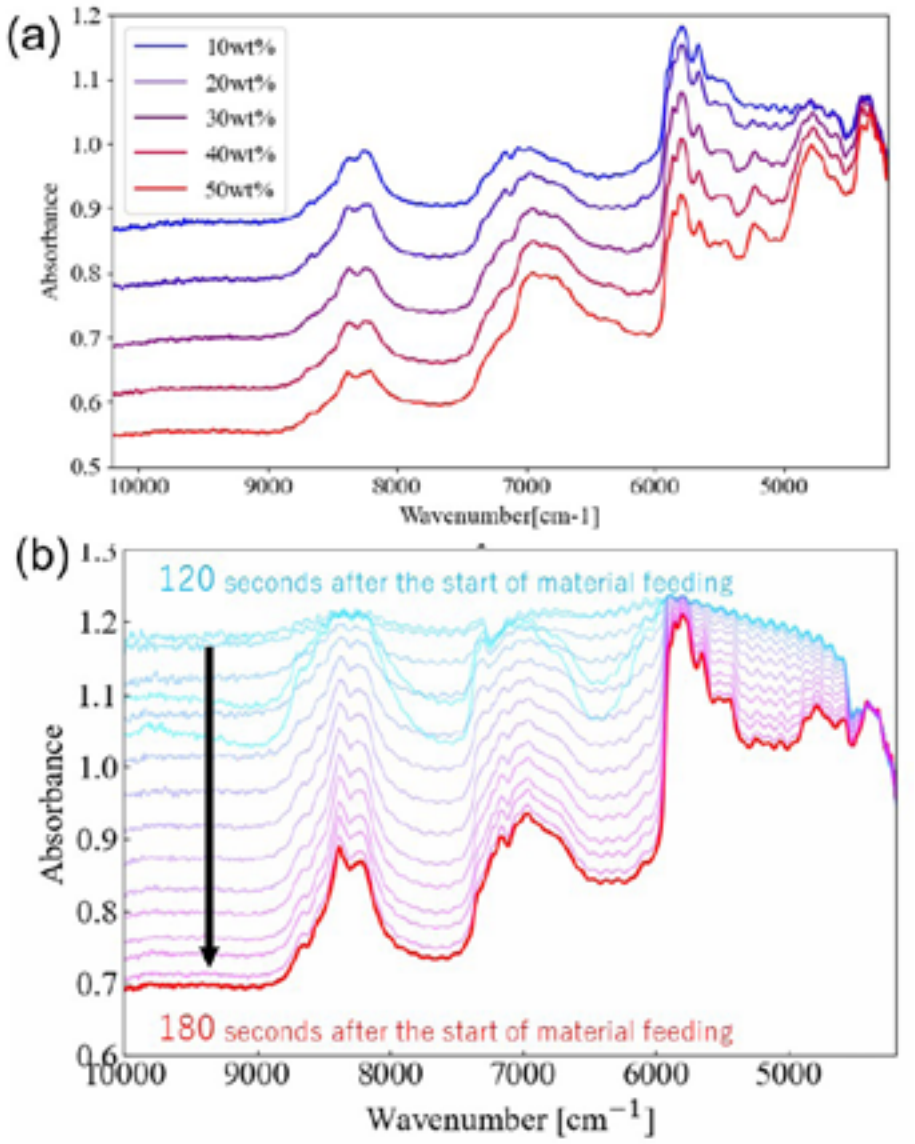
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In recent years, using biodegradable polymers, recycled materials, and biomass-derived materials has expanded as sustainable solutions to reducing plastic waste and dependence on fossil fuels. Despite their environmental benefits, these materials often exhibit considerable variability in composition and molecular weight, resulting in instability in their mechanical and processing properties. To address these issues, the development of technologies for real-time monitoring of material composition and properties during polymer processing is required. This study presents the application of near-infrared spectroscopy for real-time monitoring of compositional variations during injection molding the extrusion processes of polymer composites.

Inline near-infrared spectroscopic measurement was performed during the extrusion process of wood-plastic composites, in which cellulose powder was added to polypropylene. A mixture of polypropylene powder, a compatibilizer, and cellulose powder was fed into an extruder and compounded at a temperature range of 150–190°C, with a rotation speed of 100 rpm. A newly developed heat- and pressure-resistant NIR probe[1] was installed at the extruder head, and connected to a Fourier transform near-infrared spectrometer via optical fibers. The spectra were measured using the diffuse reflectance method in the spectral range of 10000-4000 cm-1, with a spectral resolution of 8 cm-1.

The inline near-infrared spectra obtained for composite materials with cellulose content varying from 10% to 50% are shown in Figure (a). As the cellulose content increased, the baseline slope becomes more pronounced, and the absorbance at spectral band of 4778 cm-1 corresponding to cellulose increased. This is due to the increased scattering intensity and light absorption caused by the cellulose powder. Figure (b) shows the temporal changes in the spectra after feeding the composite material with 40% cellulose content into the extruder. Significant variations in reflectance were observed during the material changeover procedure. These changes in near-infrared spectrum can be used to evaluate the compositional stability of biomass composite materials in real time during melt compounding process.

[1] I. Yoshikawa, S. Hosoe, Y. Hikima. M. Watari, M. Ohshima, Ind. Eng. Chem. Res., 2023, 62 (15), 6338-6346



ACKNOLEDGEMENTS: The authors thank Dr. Masahiro Watari, S.T. Japan and Prof. Masahiro Ohshima, Kyoto university for designing the NIR probe and advising injection molding process. The authors also thank Dr. Yuki Saito and Dr. Yasumasa Ohira, National institute of advanced industrial science and technology (AIST) for supporting experiments.

Keywords
Polymer processing,Inline monitoring, Polymer composite, Cellulose, Biodegradable polymer

O.10.34

RAPID PHENOTYPING OF TREES WITH NIR SPECTROSCOPY AND HYPERSPECTRAL IMAGING

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The goal of this research was to integrate genetic studies with advanced spectroscopic techniques, to enhance the understanding of tree adaptation mechanisms to the local environmental conditions. This new knowledge could significantly impact sustainable forest resource management and effective tree breeding programs in response to environmental changes.

A unique set of wood samples from 50-year-old Scots pine (Pinus sylvestris) trees, representing seventeen diverse national populations from Poland are used as experimental samples. All trees were planted at the same time and grown under identical silviculture, climatic and soil conditions. An additional set of three Slovenian provenances was included to provide a genetic background for the Scots pine populations.

A measurement protocol for these samples was developed using NIR spectrometers for the rapid, accurate, and non-destructive phenotyping and prediction of wood quality descriptors. FT-NIR spectra of samples were acquired using a custom-designed automated measurement station based on the Bruker Optics MPA II FT-NIR multi-purpose analyzer. The system was automated with the ABB GoFa collaborative robot, featuring a custom 3D-printed tool holder and controlled by software developed in ABB RobotStudio.

Chemical analysis was applied to determine a variation of composition within samples. It included Soxhlet extraction for extractive content, the Klason method for determining lignin content, as well as holocellulose and cellulose contents, thermogravimetric analysis (TGA), elemental analysis, and high-performance liquid chromatography (HPLC). These methods provide comprehensive insights into the chemical composition of wood samples, enriching the reference dataset used for tree phenotyping.

Microsatellite DNA markers (SSR) and genetic association analysis, including a large database of single nucleotide polymorphisms (SNP), were determined on each studied tree to express genetic factors. This approach helps uncover neutral genetic variations within and between Scots pine populations, providing insights into the genetic basis for ecotype differences and linking observable phenotypes to genetic variations.

FT-NIR results are complemented by a hyperspectral imaging component, achieved by scanning wooden disks with hyperspectral cameras (VIS: Specim FX10, NIR: Specim FX17, and SWIR) controlled by LUMO software. Data pre-processing and chemometric analysis were performed using EVINCE (Predictera) and the MATLAB PLS toolbox (Eigenvector). This custom-built hyperspectral imaging measurement station ensured elevated precision and innovation in this research.

In summary, the results represent a significant advancement in tree phenotyping. The integration of FT-NIR spectroscopy, robotic automation, hyperspectral imaging, and comprehensive chemical analysis has resulted in a robust and efficient system capable of producing high-quality spectroscopic data. These data are crucial for understanding the genetic and phenotypic factors contributing to the ecological resilience of Pinus sylvestris, ultimately supporting efforts to conserve and manage forest resources more effectively.

ACKNOLEDGEMENTS: Part of the work has been developed within project "Spectroscopic Methods for Rapid Phenotyping of Trees Reflecting their Ecological Resilience" (acronym DENDRO-SPEC), co-financed by the National Science Centre Poland (NCN) No. UMO-2021/43/I/NZ9/02809 and the Slovenian Research Agency (ARRS) N4-0274.

Keywords
wood properties,phenotyping,Pinus sylvestris,trees,genetic plasticity

F.10.28

POTENTIAL SCREENING METHOD FOR COASTAL MANAGEMENT IN THE OCEAN DECADE: DETECTION OF SMALL STAINS IN SAND FROM OIL SPILLAGE USING HYPERSPECTRAL IMAGING

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Introduction: Oil spill accidents have occurred in many parts of the world affecting the marine environment worldwide. Large oil stains are easily detected, however, residual oil trace analysis at grain level is challenging, usually done by sample processing with oil (bio)markers extraction and further analysis using gas chromatography and mass spectrometry. Alternatively, this work describes a direct and non-destructive method to detect traces of oil spillage in sand using near infrared hyperspectral imaging (NIR-HSI).

Objective: The aim is to detect beach sand contamination which would be imperceptible in naked eye, allowing to evaluate the extent of remediation after a spillage event.

Experimental: Oil that appeared in a huge accident in Northeast Brazilian coast in 2019 was collected in two beaches. The oil was mixed with sand in different proportions to simulate stains from heavily contaminated until the contamination was imperceptible in naked eye and considering different moisture content (Figure 1.a). Hyperspectral imaging in the 928-2500 nm range (Sisuchema, Specim) were acquired of 45 cm^2 sample area using a 156 µm pixel size. Through Principal Component Analysis, regions of interest from the synthetic stains were identified (Figure 1.b) and used to select oil/sand training spectra for one class model Soft Independent Model of Class Analogy (SIMCA). Other samples were prepared for validation, carefully adding grains of sand embedded in diluted oil on non-contaminated sand next to dark mineral grains from sand, which could be confounded by naked eye (Figure 1.c). Both sand and these oil-sand spiked particles had the same granulometry range (300 µm – 1 mm).

Results: Different models were built using the whole spectra or selected regions and using different pre-processing. SIMCA model that yielded better external previsions was built in the 2045-2443 nm range using standard normal variate (SNV) as pre-processing, using 4 principal components for class space. The model was sensible to the target contaminant since all grains embedded with oil added to the external validation set were predicted (Figure 1.d), and it was specific as no sand grain (light or dark) was predicted as oil. Moisture plays an important role as good predictions were achieved only when training and validation set were built with dried samples (60 °C overnight).

Conclusion: In conclusion, oil agglomerates in the level of beach sand grain size were detected, and this method has potential be used qualitatively as a sand screening to track the extent and permanence of oil spillage in beaches.

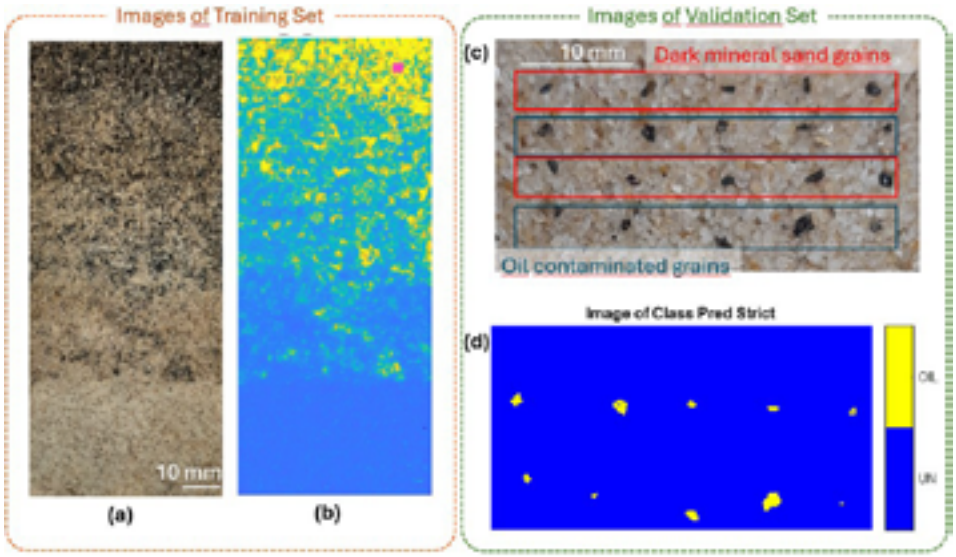


Figure 1. (a) Simulated oil stain sample (b) Image of PCA scores for pixels selection (c) Samples for validation in sand grain level and respective (d) Image of classification model prediction

ACKNOLEDGEMENTS: National Institute of Advanced Analytical Sciences and Technologies - INCTAA (FAPESP 2014/50951-4, CNPq 465768/2014-8)

Keywords
environmental chemistry,hyperspectral imaging,beach contamination

F.10.29

APPLICATION OF SPECTROSCOPY TECHNIQUES TO STUDY THE DEGRADATION OF LIGNOCELLULOSIC RESIDUES USING FUNGAL FERMENTATION

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Lignocellulosic material (LC) has many advantages, such as being inexpensive, renewable, and widely available in most parts of the world, making it environmentally sustainable. Recently, the use of LC has attracted significant research attention due to its potential to be chemically modified to render it a source of biofuels and biomaterials [1]. In fact, LC is a great option for moving from a petroleum-based to a biomass-based economy as it is a biodegradable resource.

LC are mainly composed of cellulose, lignin and hemicellulose and the ratio of these components is varying because of their high heterogeneity. Both structural and chemical factors, such as the content and composition of lignin and the complex interactions between cellulose, hemicellulose, and lignin, are the primary drivers of LC recalcitrance [2]. This makes its biodegradation a difficult process, and the biorefinery platform needs to pretreat the materials to enhance subsequent processing [3]. Luckily, fungi can use their enzymatic machinery to metabolize LC and biotransform it into functional materials [4]. This offers benefits for conversion processes, such as lower energy and economic losses when compared to physical or chemical pretreatments.

This study aims to characterize different types of LC waste materials using near (NIR) and mid infrared (MIR) spectroscopy coupled with multivariate data analysis. In detail, the main categories of LC material considered are: i) virgin wood, i.e. clean wood; ii) treated wood, consisting in wood mixed with glue/resins; iii) residues from agriculture activities/food industry. To this aim LC residues samples belonging to these three different categories have been collected. All the samples have been analyzed both with conventional lab analysis (to determine the raw fiber composition) and with IR spectroscopy. MIR and NIR spectra have been investigated to find out differences among the categories of the LC material analyzed. In addition, a principal component analysis was computed to search for similarities and groupings among the samples based on their chemical-physical characteristics and highlight the correlations among the analyzed parameters.

Successively, one sample for each LC category was selected and used as mainstream in liquid fermentation. The main culture flasks were grown incubating Pleurotus ostreatus in the darkness on a shaking plate (150 rpm) at room temperature (22-28°C) for 7 days. The changes to the lignocellulosic structure caused by fungal deterioration processes were investigated using IR spectroscopy.

The results demonstrated that spectroscopy could be a perfect tool for the rapid screening of the LC composition and the real-time monitoring of the biodegradation of lignin, improving its reuse as a valuable resource for the generation of bioenergy and biomaterials. In fact, it is essential to determine the substrate composition to successfully address its inherent resistance to microbial and enzymatic biodegradation and spectroscopy is a valuable candidate, especially when compared to the slow and costly laboratory procedures.

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Keywords
lignocellulosic waste,lignin,white-rot fungi,green economy,sustainability

F.10.30

HIGH-THROUGHPUT ON-SITE MICROPLASTIC QUANTIFICATION USING NIR SPECTROSCOPY

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High-throughput quantification of microplastics in environmental matrices remains a significant challenge with critical implications for environmental monitoring and remediation efforts. Near-Infrared (NIR) spectroscopy, particularly when deployed on-site using portable devices, holds promise for overcoming these challenges. However, NIR spectroscopy has not been thoroughly examined in terms of its limit of detection (LOD) and sensitivity for microplastics analysis, especially in complex environmental samples.

This study evaluates the performance of the Buchi NIRFlex N-500 (benchtop FT-NIR) and the Si-Ware NeoSpectra Scanner (portable FT-NIR) for bulk quantification of microplastics directly in soil, without sample preparation. Five common polymers were included; PMMA, HDPE, PET, PS, and PA6, as well as two soil types including standard agricultural soil.

The quantitative analysis by means of partial least squares regression (PLSR) was conducted to determine the multivariate limit of detection (mLOD) for each polymer. The achieved detection limits reached levels below 0.1% w/w (1000 ppm) for benchtop spectrometer. While the handheld NIR could not go that low, it also yielded ppm levels low enough to provide rapid screening directly on-site.

This study shows that NIR spectroscopy can suit the needs of a Tier 1 (initial screening) and Tier 2 (bulk quantification) of microplastics directly in solid environmental matrix in a high-throughput manner.

ACKNOLEDGEMENTS: This research was supported by the Austrian Science Fund (FWF) [V1014-NBL].

Keywords
NIR spectroscopy,microplastics,handheld,level of detection

CULTURAL HERITAGE, FORENSICS & PETROCHEMISTRY

0.11.35

A SPATIAL ANALYSIS OF A NORTHERN STONE AGE SITE IN THE LIGHT OF NIR SPECTRAL RESPONSES

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Traces of prehistoric human activities in soils can be studied in many ways. Phosphate analysis is an old and established technique that is used to identify settlement locations. A mix of organic and inorganic materials was used by prehistoric humans in their everyday life and after deposition these materials became incorporated into the soil system and became changed by soil formation and taphonomic processes. Consequently, what we analyse today in soil samples is something different but still possible to elucidate to meaningful information. Pedological and geoarchaeological studies have shown that NIR is a useful technique when used in this kind of research (1,2,3).

Aims and Questions

The aim of this study is to use NIR for spatial analysis in prehistoric contexts in combination with conventional methods and investigate the added values of using NIR spectroscopy.

1) Can NIR spectroscopy analyse a combination of organic and inorganic matter from old residues in soil from prehistoric sites?

2) How does spatial responses in NIR studies relate to corresponding conventional techniques?

3) In what way can interpretations of NIR spectra target the causes for spectral responses?

Material

In this study NIR has been applied to analyse grid samples collected across late Neolithic site in northern Sweden. A soil corer was used to collect 53 B-horizon samples with a varying density of 10 to 20 metres. Samples were dried (30C), homogenised using mortar and pestle and passed through a 1.25 mm mesh sieve.

Instrumentation

The NIR spectrometer used ASD LabSpec4 (350:1:2500 nm), with a contact probe and internal illumination. Phosphate analysis was done according to (1).

Principal Component models have been calculated using software Evince-Prediktera.

Results

In figure 1, a map of the phosphate distribution is presented with elevated levels of phosphate and some parts of the investigated area because of prehistoric habitation activities. This information was used by the archaeologist to target areas for excavations and during the process of excavation several findings were made.

Figure 2 shows a map based on interpolated score values of t2 and category intervals as symbols of t3 superimposed on observations made during the excavation. The very similar spatial variation of phosphate content and the positive t2 score values show that NIR alone can be sufficient for prospection of prehistoric activity areas.

Different sample clusters can in turn be compared using Savitzky-Golay filtering and the group with higher phosphate content consequently display a peak around 1920nm which may be attributet to P-OH bondings.

Conclusion

This study shows that NIR-spectra provides at least as much interpretable information as does conventional wet-chemistry or data provided by XRF. Furthermore, NIR spectra shows a combination of organic and inorganic materials and gives a clear added value to the understanding of the activities of Neolithic people and how space was used.

And as always, the speed and simplicity of NIR analysis is a strong argument for adding this as an obvious tool to archaeological field research.

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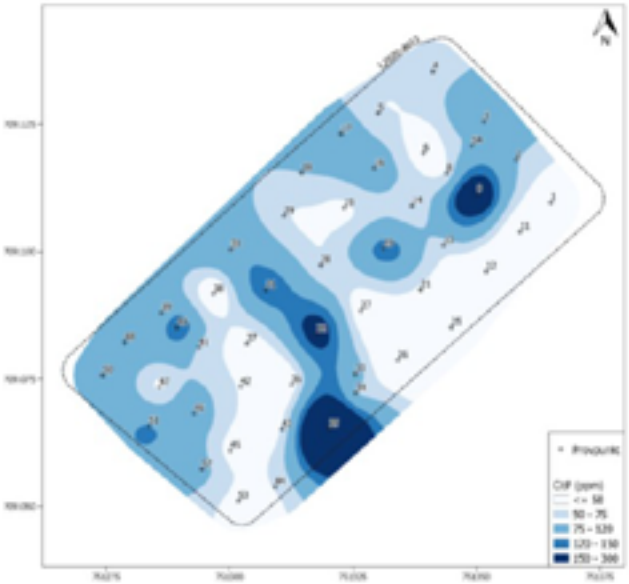


Figure 1. Phosphate distribution across the site prior to the archaeological excavations.

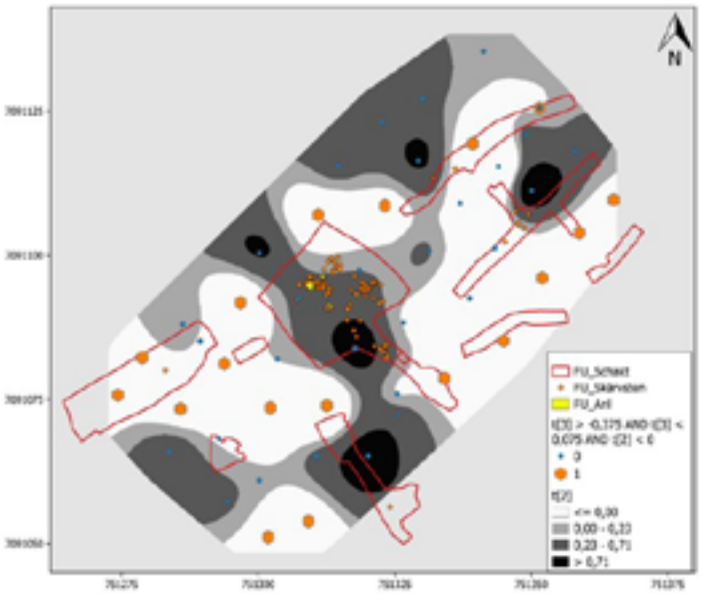


Figure 2. A composite map over the site with sampling points, trenches, features and findings, and interpolated t2 score values and categorised point t3 scores.

ACKNOLEDGEMENTS: The Kempe foundation

Keywords
Archaeology,NIR spectral responses,Prehistoric sites,soil analysis,Spatial analysis

O.11.36

RESEARCH ON APPLICATION OF NEAR INFRARED SPECTROSCOPY IN CHINA OIL REFINING INDUSTRY

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Intelligentization is the inevitable trend of the development of oil refining enterprises. Self-perception of information depth, self-decision of intelligent optimization and self-execution of precise control are three key characteristics of intelligent factories, among which self-perception of information depth is the foundation of intelligent refineries. The data of chemical composition and physical properties analysis of raw materials, intermediate materials and products is an important part of information perception.

In recent ten years, driven by the continuous development of new technologies related to spectral instruments, such as laser, optical fiber, microelectronics, computer and chemometrics, many new types of spectrum and spectrum rapid and online analytical instruments have appeared, such as near infrared, Raman, nuclear magnetic resonance, LIBS, XRF, etc., which make the analytical instruments with complex structure and huge volume that can only be used for material composition analysis in the laboratory can also be used for on-site rapid analysis and real-time online analysis of industrial units. With the increasing demand for digitalization and intelligence in the production process, these modern process analytical technologies are more and more widely used in all production links of the oil refining industry, and the timely and accurate analysis data provided by them play an irreplaceable role in stabilizing production, optimizing operation and saving energy and reducing consumption.

Modern rapid and online analysis technology of oil and petrochemical products with chemometrics as the core provides a very effective means for information perception of chemical components, which will play an increasingly important role in intelligent refineries. Petrochemical Research Institute of SINOPEC has a strong R&D and application foundation in this field, and has developed a database of near infrared spectra of crude oil, gasoline, diesel oil, VGO, residual oil and other oil products, which has been applied to a certain extent in oil refining enterprises. This report mainly introduces the progress of rapid and on-line analysis technology of oil and petrochemical products in recent ten years and the development trend in this field in the future.

ACKNOLEDGEMENTS: The author thanks the late Professor Lu Wanzhen (Academician of China Academy of Sciences) for her contribution to this field.

Keywords
Crude oil,Petroleum,on-line analysis

O.11.37

HEADS UP: UNVEILING PIGMENTS IN A CUTTING-EDGE 3D NEAR INFRARED HYPERSPECTRAL IMAGING (3D-NIR-HSI) INTEGRATED SYSTEM

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Advancements in analytical techniques have been providing benefits to different fields; among them, Cultural Heritage (CH) Science is taking advantage of more effective preservation and studies of artworks [1]. NIR spectroscopies are indispensable approaches providing crucial information related to the material composition, structural details and used techniques, making them unreplaceable tools in conservation and restauration activities. Additionally, hyperspectral imaging (HSI) offers a more comprehensive approach by combining spectroscopies across all the electromagnetic spectrum (including Raman spectroscopy) with spatial information at the macroscopic level; while 3D vision techniques use multiple images with different orientations in the visible range to extract the geometry and surface details of an object, being valuable for documenting sculptures [2-3].

Notwithstanding this, the integration of these techniques is still missing, not only due to alignment issues and inaccuracies in combining spatial and spectral data, but also to difficulties in maintaining focal distances for the different imaging techniques as well as problems in correlating different resolution, scales and depths of analysis. Linking surface information from the 3D vision techniques with spectral data from line mapping HSI it is not evident from an algorithmic point of view.

In this study we explore mapping 2D hyperspectral NIR-HSI images into a unified 3D metric space for combined spectral and geometric analysis. A reproduction of Michelangelo's David head placed on a rotary axis, was painted with white pigments, and scanned from 40 viewpoints using a SWIR line-mapping HSI camera. The captured data was processed with a modified Structure from Motion (SfM) pipeline to generate a point cloud linking image pixels to 3D coordinates. These SfM pipelines, designed for pinhole camera models, produce significant reprojection errors with line-scan images, making the combined estimation of camera positions unachievable. The proposed method addresses this by pre-calibrating the line-scan camera for radial image undistortion and a constrained optimization of intrinsic pinhole parameters, ensuring successful convergence of conventional SfM pipelines. To validate the method, the same sample was scanned with a structured-light stereo sensor, providing geometrical ground-truth for evaluating the hyperspectral point cloud. Spectral unmixing was also successfully applied to isolate and identify chemical components, enabling accurate mapping of the pigments on a 3D surface [4], obtaining both the intricate details of the shapes and textures and the chemical composition.

Through this method we reached a novel pathway: integrating NIR-HSI data with 3D modelling, combining their power to deliver information previously unmatched. Indeed, the integration of advanced spectroscopic tools into a single cohesive system is the very heart of this work, allowing to extract reliable chemical information and structural details from the 3D model. This cutting-edge system enables a comprehensive analysis of the structural geometry and texture of CH, empowering curators and conservators to safeguard the World's CH, and presenting high applicability to other fields, e.g. food and industrial inspection.

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ACKNOLEDGEMENTS: The authors would like to thank Marco Innocenzi from the Painting and Wood Materials Restoration Laboratory of the Vatican Museums for his support.

This work was partially financed by project SIDIRE (PID2023-146230OB-I00) from MCIU /AEI /10.13039/501100011033 / FEDER, UE)

Keywords
NIR Hyperspectral Imaging,Structure from Motion,3D vision,Integration,Cultural Heritage

F.11.31

APPLICATION OF NEAR-INFRARED SPECTROSCOPY FOR ESTIMATION OF LIGNIN CONTENT IN JUTE

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Jute (Corchorus olitorius L.) is an important commercial and eco-friendly fibre crop. India is the second largest producer of jute after Bangladesh [1]. The lignin content in jute plays a crucial role for strength, fineness and durability of a fibre. An increase in lignin content in jute fibers (above 15%) provides several key advantages, particularly in enhancing the strength and durability of jute-based products. These benefits include, resistance to microbial degradation, durability in geotextile applications, enhanced fiber composites and for use in roads, hill slopes, railway track soils, river banks, among others. On the other hand, lower lignin content is desirable for products requiring finer fibres, like textile applications. Estimation of this polymer (lignin) requires very complex, time consuming and costly methods. Even simple chemical analysis cannot be used because of degradation of basic polymeric structures. A non-destructive method for estimating the quantities and proportions required for various applications would be very useful for not only fibre quality determination, but also for selecting the desirable genotype, appropriate for different applications.

Therefore, the objective of this study was to develop a rapid and cost-effective method for predicting lignin content in jute using fiber optics-based Near-Infrared (NIR) spectroscopy combined with chemometric technique. The study also aimed to identify the most effective spectral data preprocessing technique to optimize prediction accuracy. Fifty jute samples were collected from different locations of West Bengal, India. The lignin content was analyzed by acetyl bromide method and observations were taken in three replicates [2]. NIR spectra were collected using a tungsten halogen lamp (12 V/20 W), an NIR spectrophotometer with an InGaAs detector array (900–1700 nm), and a bi-directional fibre-optic probe in a darkroom setup at (25±0.5)°C. Data acquisition, spectral preprocessing, and model development were carried out using SpectraFind software (Ayudyog Pvt Ltd., India). Initially, after acquiring the spectral data from the instrument, the data were subjected to various preprocessing transformations. In the study, three smoothing techniques such as Second Order Derivatives, Standard Normal Variate (SNV) and finally combination of these two were applied and their efficiencies were assessed for model development of partial least square regression (PLSR) [3].

The best calibration model was found when the spectral data were preprocessed jointly with second derivatives and SNV with the coefficient of determination (R2) and RMSEC of 0.87 and 0.5 respectively. The prediction model for jute gave R2, RMSEP, and RPD 0.82, 0.61 and 4.25 respectively. The results establish that PLSR could be an analytical method for prediction of lignin of jute by using second ordered derivatives and SNV treated fiber optics based NIR spectroscopic data. The set up can be used for other marker molecules in plants and their products by suitable modifications in the software.

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ACKNOLEDGEMENTS: Financially supported by Indian Council of Agricultural Research (ICAR) for the program National Agricultural Science Fund vide Sanction Order No. NASF/PA-10009/2023-24 dated 26.02.2024

Keywords
Jute,Lignin,SNV,Second Order Derivatives,PLSR

F.11.32

WASHING AWAY THE FRAUD: CHEMOMETRIC MODELS FOR DETECTING FALSIFIED LAUNDRY DETERGENT

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Laundry detergent is a cleaning agent used to wash soiled fabrics. Detergents in general contain ionic surfactants, bleaching agents, enzymes, sequestrants and chelating agents as well as other additives, such as dyes and fragrances [1]. The global market of laundry detergent was valued at US$ 142 billion in 2024 and is expected to reach US$ 201.88 billion in 2028. In Brazil, during 2023, 1.5 million ton of washing powder were sold, generating approximately R$ 17 billion in revenue. One brand dominates the Brazilian market of washing detergents in terms of quantity sold, being the most remembered name brand. In a billion-dollar industry with brands that inspires quality and trust, washing powder recently became a target for criminals that counterfeit the product. In 2022, during the 'Lavagem Imperfeita' (imperfect washing) operation of the Civil Police, 5 ton of counterfeit laundry detergent were seized in the central-west region of Minas Gerais state in Brazil [2]. This study aims to authenticate original samples of the market leader brand using portable NIRS and chemometrics. For this purpose, 34 seized samples from the 'Lavagem imperfeita' operation as well as 83 samples from the market leader brand and 55 samples from other brands obtained from local supermarket were analyzed utilizing a portable MicroNIR® 1700 spectrophotometer (Viavi Solutions). NIR spectra were obtained in the diffuse reflectance mode, with 20 scans over a wavelength range of 950-1650 nm, at a resolution of 6.25 nm. Data analysis was conducted using MATLAB® software, version 7.13 (MathWorks), jointly with PLS_Toolbox, version 6.5 (Eigenvector Technologies) and DD-SIMCA toolbox version 1.2 provided by the authors of [3]. An unsupervised PCA model and three multivariate classification models were constructed: PLS-DA (Partial Least Square Discriminant Analysis), SIMCA (Soft Independent Modelling of Class Analogies) and DD-SIMCA (Data Driven SIMCA). For supervised methods, the Kennard-Stone algorithm was utilized to separate the training and test samples from each class individually. In the model validation process, various figures of merit (FOM) were estimated. These FOM encompassed metrics such as False Negative, False Positive, Sensitivity, Specificity, and Reliability rates, Accordance, Concordance and Concordance Odds Ratio. No visual differences were noticed among the NIR spectra of the original and counterfeited samples, underscoring the imperative use of chemometric tools to obtain pertinent information for the classification of washing powder samples. The PCA model showed good cluster separation between the laundry detergent samples seized by the Civil Police of Minas Gerais and both the market leader brand and the other brands available in the market. All three supervised classification models presented satisfactory performance for their intended use, with DD-SIMCA showing the best results, with sensitivity of 92.9% and specificity of 97.8% for the independent test set.

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ACKNOLEDGEMENTS: Brazilian funding agencies: FAPEMIG, CAPES and CNPq

Keywords
Handheld NIRS sensor,Portability,One-class modeling,Forensic chemistry

F.11.33

INFRARED SPECTROSCOPY AND HYPERSPECTRAL IMAGING FOR FORENSIC COCAINE SAMPLE ANALYSIS

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The fight against drug trafficking is one of the main challenges for public health and international security. In this context, the scientific analysis of drugs plays a crucial role in the control and monitoring of illicit substances. While traditional analytical techniques are well-established and highly effective, they present significant limitations, including the use of chemical solvents, direct sample handling, and the lengthy time required to obtain results.

This study aims to evaluate the applicability of FT-NIR spectroscopy and hyperspectral imaging (HSI) in the mid- and near-infrared range (3,700-1,900 cm?[1] and 900-1700 nm, respectively) in the forensic analysis of cocaine and its most common cutting agents. The primary objective is to demonstrate how these analytical methods can serve as a valid, fast, and safe alternative to traditional techniques, ensuring high reliability of results while minimizing risks to operators.

Chemometric approaches, including Principal Component Analysis (PCA), Soft Independent Modeling of Class Analogies (SIMCA), and Partial Least Squares Regression (PLS-R), were applied to interpret the spectroscopic and imaging data acquired.

The study examined cocaine samples at varying concentrations from four separate seizures, along with six commonly used cutting agents: creatine, caffeine, levamisole, lidocaine, lactose, and mannitol. All analyses were performed directly on samples sealed in polyethylene sachets, without any manipulation or opening of the packaging.

The results demonstrated the effectiveness of the proposed techniques. Spectroscopy in the NIR region exhibited a high penetration capability through sealed samples, enabling accurate identification of substances even within plastic packaging, and even the possibility of quantifying the amount of drug with an RMSEP of 2.4. Hyperspectral imaging further facilitated spatial mapping of the distribution of cocaine and cutting agents, providing a detailed view of the chemical composition of the samples. Indeed, SIMCA models acquired on the hyperspectral data in the NIR region showed 100% specificity and sensitivity values always higher than 93% on both the training and the test sets.

AGRICULTURE & FEED 1

O.12.38

ASSESMENT ON THE NUTRITIONAL STATUS OF CITRUS LEAVES USING HYPERSPECTRAL VIS-NIR IMAGES AND CONVOLUTIONAL NEURAL NETWORKS

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Applying the correct amount of fertilizers is crucial to reducing production costs, increasing crop yield and ensuring fruit quality. Nutrient imbalances negatively impact plant growth, flowering, and resilience to diseases and abiotic stresses, while excessive fertilizer use poses environmental risks, such as water contamination and soil degradation. Although precise nutrient diagnosis can be achieved via ionomic analysis, it is costly and time-intensive. This study proposes a faster, non-destructive alternative using hyperspectral leaf imaging combined with Convolutional Neural Networks (CNNs) to assess key nutrients, including N, P, K, Ca, Mg, Na, S, Fe, Cu, Mn, Zn, and B.

Spring sprouting leaves of mandarins cv. 'Clementina de Nules' (Citrus clementina Hort. Ex Tan.) were collected in monthly batches from April 2021 to January 2022, for a dataset of 572 leaves. Hyperspectral images of the leaves were captured in the range of 500-980 nm every 10 nm, using 458 for calibration and 114 for test. The actual concentrations of the nutrients were obtained by ionomic analysis. This process included the drying and grinding of the leaves and the determination of the nutrients using Kjeldahl method for N and inductively coupled plasma optical emission spectrometry (ICP-OES) for the other nutrients. CNNs models were used to estimate the nutrient concentration using as input the full image. The large images dimensionality caused a dimensionality problem on the first convolution layer of the CNN which hindered the performance of the models. To improve the convergence of the CNN models, a feature selection was applied to get the most relevant bands for each nutrient using Search Forward Selection (SFS), reducing the original 49 wavelengths to 6. The wrappers used to estimate the relevance of each band were Random Forest, Linear Regression and Partial Least Squares.

CNN models using the selected bands improved the results obtained with traditional machine learning methods used in a previous study [1]. The models to estimate Fe and B obtained the best results (R^2= 0.86 and R^2= 0.80 respectively) with other nutrients obtaining more discrete results, such is the case of Ca and Na (R^2= 0.64 and R^2= 0.62 respectively). CNNs models to estimate Cu concentration obtained the worst results (R^2 = 0.32). Thus, CNNs models improve traditional machine learning models using its ability to extract spatial patterns to model the concentration of nutrients using less spectral information.

CNNs models using the complete hyperspectral image (full set of wavelengths) obtained worst results in most of the nutrients analysed (Fe and Na for example obtained R^2= 0.85 and R^2= 0.48 respectively) than traditional machine learning models without using spatial information [1]. This result shows that complex CNN-based models using the whole image as input are not necessarily better for estimating nutrient concentration than simpler machine learning models using the average leaf spectrum. It is necessary, before choosing one model family or another, to determine whether the CNN-based model has a balanced dataset from a dimensionality point of view (Bands vs. Samples) since increasing the number of bands dramatically increases the number of model parameters and makes it difficult to converge the training process to an optimal solution, making it necessary to increase the size of the dataset to calibrate the model or to apply feature selection methods to reduce the spectral information.

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ACKNOLEDGEMENTS: This work was partially funded through the projects MICIU AEI PID2023-150192OR-C31, C32 and C-33 with the support of FEDER funds

Keywords
Agriculture,Nutritional Diagnosis,Machine Learning,Convolutional Neural Networks,Vis-NIR

O.12.39

TRANSFORMING NIR SPECTRA INTO SOIL INSIGHTS: DEEP LEARNING MODELS FOR ORGANIC CARBON AND CLAY

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The development of agricultural variable rate management systems that require detailed knowledge of soil variability within individual fields has become essential in the context of precision agriculture. The implementation of rapid methods, such as near-infrared (NIR) spectroscopy combined with machine learning models for soil property prediction, represents a promising solution for expeditious and cost-effective soil mapping techniques. This study developed and evaluated two deep learning artificial neural network (ANNDL) models to investigate the prediction of soil organic carbon and clay content from raw soil spectra.

The calibration dataset comprises 460 soil samples obtained from northern Italy, while the validation dataset consists of 55 samples from two fields located in the upper Po Valley. Soil samples were collected according to a regular 50-meter grid at a depth of 30 centimeters. To map SOC concentration and clay content, these samples were analyzed by an external laboratory employing standard wet reference methods. The NIR analysis was conducted using the NIRFlex N500 (Buchi) in diffuse reflectance mode over the 1000-2500 nm range.

Two distinct calibration ANNDL models were developed. In both cases, the TensorFlow framework and the Rmsprop optimizer were utilized, and they are designed as a multilayer densely connected architecture, with dropout layers set to 0.5 to enhance robustness, though their number of layers differed. In the case of soil organic carbon, the spectral data was first compressed using PLS (8 latent variables) to enhance both the training performance and the focus on chemical information that is most correlated to the SOC target. After an optimization process, the number of epochs was set to 90 to minimize loss, while a batch size of 23 was chosen to circumvent overfitting issues. For the clay content, the spectral data was directly utilized for the training process, without the application of PLS compression. In this case, scattering is influenced by the dimension of the clay particles and the PLS compression could eliminate informative signals.

The NIR-based estimation for organic carbon content was evaluated using Root Mean Square Error (RMSE) and BIAS metrics. The machine learning calibration models showed the following results: the first model for SOC evaluation has RMSECV= 3.42 g/kg and BIAS (cal.) =-0.07 g/kg; the second model for clay has RMSECV=41.71 g/kg and BIAS (cal.) =-5.57 g/kg. The prediction on the two fields demonstrated varied performance, attributable to the variability inherent in the soil.

In the first field, characterized by high variability, the SOC model achieved error metrics suitable for effectively mapping soil characteristics, with RMSEP=2.05 g/kg and BIAS (pred.) =0.67 g/kg. Similarly, the clay model shows RMSEP=18.69 g/kg and BIAS (pred.) =12.27 g/kg, which are acceptable for this context. For the second field, the SOC model has RMSEP=2.58 g/kg and BIAS (pred)= -1.46 g/kg; for clay model RMSEP=22.27 g/kg and BIAS (pred.) =-13.88 g/kg. Due to the low field variability in this case, the model's error metrics make it unsuitable for identifying distinct management zones.

This study demonstrates the potential of deep learning models in leveraging NIR spectroscopy for rapid and cost-effective soil property mapping, provided that field variability is sufficiently high. The findings highlight the importance of tailoring model architecture and data preprocessing to the target property's characteristics, paving the way for improved precision agriculture through advanced soil mapping techniques.

Keywords
NIR spectroscopy,Precision agricolture,Deep Learning ANN

OS.12.04

APPLICATION OF PORTABLE NIR SPECTROMETER IN QUALITY CONTROL DURING OLIVE PROCESSING

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The evaluation of moisture and oil content for whole olives, olive paste and olive pomace is crucial for olive oil processors. Reference methods, such as Soxhlet extraction, used for oil content determination in olive fruits are time- and solvent- consuming and labor intensive. The bench-top NIR instrument, which offers a more comprehensive analysis, remains financially out of reach for most operators. Thus, there's a growing demand for faster, portable, accessible and low-cost solutions throughout the processing. In the present work, we collected hundreds of samples of whole olives, olive paste, and olive pomace from various origins or processors spanning the Mediterranean region, and the reference values were determined using the oven-drying moisture method and the Soxhlet oil extraction method. Subsequently, the spectra of the samples were scanned using the IAS-Olive portable NIR analyzer. Finally, the calibration models were established based on the PLS method. Results indicate a satisfactory correlation between the reference method and NIR methods, with all R2 greater than 0.90. The SECV of moisture in whole olives, olive paste, and olive pomace is 1.95, 0.68, and 1.31, respectively. The SECV of oil content in whole olives, olive paste, and olive pomace is 1.51, 0.62, and 0.25, respectively. A cheap, portable instrument has been shown to be a viable alternative to large bench-top instruments.

Keywords
portable,whole olives,olive paste,olive pomace,quality control

F.12.34

EXPLORATION OF NIR AND HYPERSPECTRAL IMAGING TECNOLOGIES IN THE DETERMINATION OF BELL PEPPER SEED QUALITY

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Near infrared spectroscopy (NIR) has become a very interesting tool for analysis and quality control in the food industry. NIR technology is based on irradiating the sample or matrix to be analyzed with radiation in the late visible and near infrared region. Part of this light is absorbed by the sample depending on its composition, i.e. the type of molecular bonds, and part is reflected. The relationship between the reflected part and its composition, using chemometric techniques, is what provides key information about the sample. Hyperspectral imaging technology combines imaging with spectroscopy. It captures an image and on each pixel of the image, the NIR spectrum of the image is obtained. In this case, both spectral and spatial information is obtained.

The main activity of the company Ramiro Arnedo, S.A. is the research, development and innovation, production and sale of seeds. It is one of the leading companies in the production of new varieties highly demanded by the horticulture sector. The company Ramiro Arnedo is interested in knowing the potential of portable NIR technology and hyperspectral imaging (IHE) for the measurement of maturity in bell pepper seeds and germination rate, which is often related to the level of chlorophyll and other physiological aspects of the plant.

Several proof-of-concept tests were carried out with NIR and hyperspectral imaging equipments in order to determine which technologies are the most suitable to perform quality control of bell pepper seeds. The images were processed pixel by pixel, identifying the variety of spectra that make up the matrix of the seeds. Various spectral preprocessing methods were applied, such as Standard Normal Variate, derivatives, along with linear and non-linear models, neural networks, and combinations of spectral data.

In view of the results obtained, it can be concluded that both technologies (NIR and IHE) behave similarly in predicting the four degrees of germination. The germination parameter, for which the company shows more interest, has the potential to be well modeled. In the case of hyperspectral imaging the correlation coefficient is higher than with the portable NIR device but this device does not have a high error of ± 2.31% seeds within a wide range of 88 to 99 germinated seeds.

ACKNOLEDGEMENTS: This project has received funding from the European Unions's Digital Programme under the grant agreement N° 101083411 Views and opinions expressed are however those of the authors only and do not necessary reflect those of the European Union (DGCNECT). Neither the European Union nor the granting authority can be held responsible for them. IRIS EDIH provides services through PADIH thankd to funding from the European Union through the funds of the Recovery, Transformation and Resilience Plan – Next GenerationEU.

Keywords
NIR,hyperspectral imaging,seed quality,germination

F.12.35

HYPERSPECTRAL IMAGING FOR MAPPING OF LIPID PEROXIDATION IN SWEET PEPPER LEAVES GROWN UNDER VARYING NITROGENOUS FERTILIZER DOSES

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Nitrogen (N) is vital for plant growth, being a key component of proteins, nucleic acids, and chlorophyll [1]. N fertilizers are crucial for maximizing crop yields amid rising food demand. However, excessive N use causes groundwater nitrate pollution, eutrophication, and oxidative stress in plants, reducing yield and quality [2]. Optimizing N levels is essential to balance growth and minimize damage. Optical sensors offer rapid monitoring of crop N status, aiding sustainable agriculture.

The objective of this study was to monitor lipid peroxidation in sweet pepper leaves under different N treatments by near-infrared hyperspectral imaging (NIR-HSI) and chemometrics.

For that purpose, an experiment was conducted in a polycarbonate greenhouse where sweet pepper plants (Capsicum annuum L.) cv. Infante (Ramiro Arnedo, S.A.) Lamuyo type were grown under conditions simulating a typical Mediterranean climate. Plants received a Hoagland-type nutrient solution. Twenty-two days after planting, 3 nitrogen treatments were applied: (1) Control with 12.0 mM of Nitrate; (2) 2N with 24 mM of Nitrate; and (3) 3N with 36 mM of Nitrate. Treatments were administered for either 3 or 10 days to evaluate the effects of short- and longer-term N excess. Then plants were irrigated with the control solution for 3 days. Thereafter, 12 plants per treatment were randomly selected for further analyses.

Lipid peroxidation was measured as the amount of thiobarbituric acid reactive substances (TBARS) in µMg-1 of fresh weight (FW) using the method described in [3]. Then, samples at 3 different dates, 36/day (n=108), were scanned by a HIS camera (HSI Xeva 1.7-320-100 Hz) with an InGaAs detector, sensitive to the 900-1700 nm range, with a spectral resolution of 3.14 nm (256 wavelengths).

The image processing encompassed multiple stages to calculate the mean spectra of each leaf. Samples were randomly divided into calibration and validation sets, accounting for the 70% (n=76) and 30% (n=32), respectively. A range of pre-processing techniques were applied to the dataset, including mean centering (MC), standard normal variate (SNV) and derivatives (1D and 2D), either in isolation or in combination. Subsequently, partial least squares (PLS) regression combined with variable selection algorithms was employed to predict TBARS content and to build chemical maps to visualise its distribution in the surface of the leaves. All data processing was conducted using the PLS_Toolbox 9.5 under MATLAB R2024a.

The results shown that the amount of lipid peroxidation, assessed as TBARS, ranged from 1.29 to 4.5 µMg-1 FW with a mean and a standard deviation of 3.34 and 1.56 µMg-1 FW, respectively. In general, satisfactory prediction rates were obtained in all PLS models. The application of SNV followed by MC resulted in optimal outcomes, with an R2 value of 0.85 for the prediction set and a Root Mean Square Error of Prediction (RMSEP) of 0.709 µMg-1 FW. This result was obtained by employing 48 wavelengths and 5 latent variables, accounting for 99.49% of the variance. A pseudo-color map of TBARS was drawn using the best PLS model to provide a visual screening method for lipid peroxidation, showcasing the potential of NIR-HSI for quantitative monitoring of TBARS in pepper leaves

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ACKNOLEDGEMENTS: This work was financed by the Ministerio de Ciencia, Innovación y Universidades (PID2021-125281OR-C21).

Keywords
Nitrogen over-fertilization,Near-infrared Spectroscopy,TBARS,Chemometrics,PLS

F.12.36

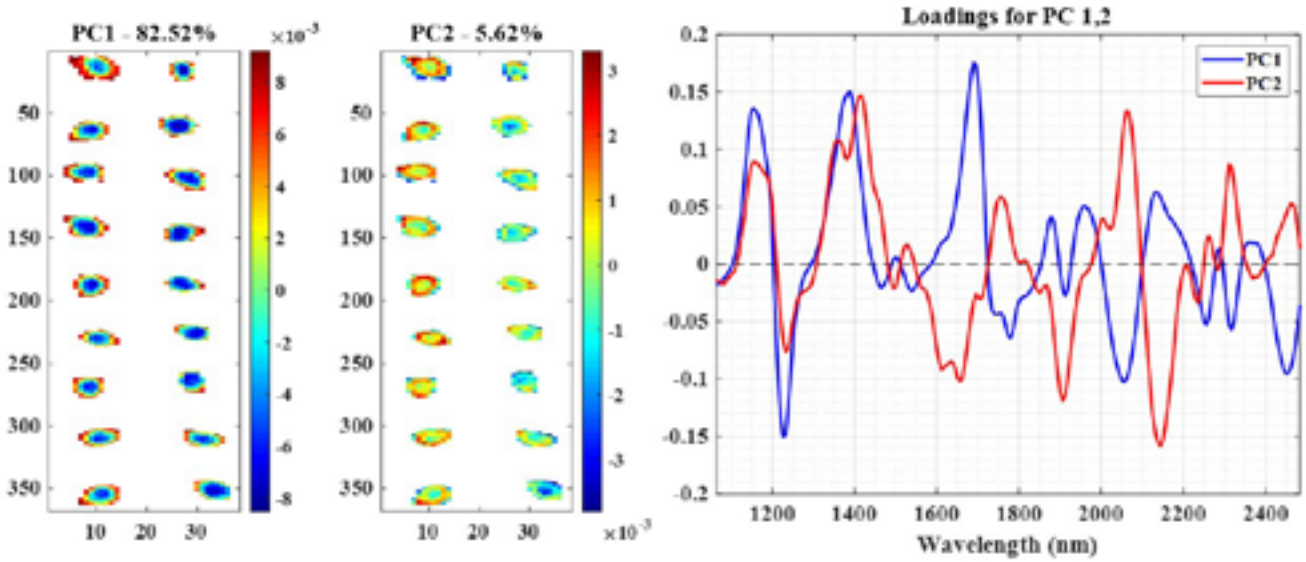
CLASSIFICATION OF TOMATO SEED VARIETIES USING HYPERSPECTRAL IMAGES

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Tomato is one of the most economically important crops, representing 15% of global production [1]. The increase in global productivity has driven the development of new varieties; therefore, it is essential to classify them from the seed stage for the continuous improvement of cultivation, commercialization, and value addition. Variety identification based on morphological characteristics faces limitations due to low genetic diversity and techniques such as molecular markers and biochemical analyses are costly and destructive [2]. Thus, the aim of this work is to assess hyperspectral images in the Near Infrared range (HSI-NIR) and chemometrics for the classification of tomato seed varieties. Seeds of the San Pedro variety and Marmande were studied. The images were acquired using an HSI-NIR from the SWIR -Specim® (Spectral Imaging Ltd., Oulu, Finland), with a spectral range from 1000 nm to 2500 nm. The HYPER-Tools software was used for preprocessing and classification steps. The spectra were preprocessed using 1st derivative with 9-point window and 2nd polynomial order. After that, an exploratory analysis (PCA) was performed. The preliminary results indicate that depending on the seed varieties, there are distinct pixels, particularly in the embryo region (Fig. 01a). The loadings (Fig. 01b) indicate that absorptions between 1600 and 1750 nm and 1450 to 1650 nm, related to the N-H bonds of proteins and lipids (fatty acids), are important for the observed trends [3]. A pixel-based approach was employed to build PLS-DA model, evaluated by sensitivity, specificity, and accuracy. The performances of the PLS-DA model for sensitivity and specificity were 0.92 and 0.94 (Training), 0.90 and 0.93 (Cross-validation), respectively. The HSI-NIR technique proves to be promising for the classification of tomato seed varieties and may represent a significant improvement in the management and quality control processes within the seed production sector. To explore its potential, a comparison will be made between pixel-based and object-based PLS-DA models.

Fig.01. PCA results a) PC1 and PC2 score surfaces; and b) loading profiles



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ACKNOLEDGEMENTS: INCTAA, CNPq, CAPES, FACEPE, UFPE, UPV-EHU, EMBRAPA

Keywords
Seeds,Cultivars,Hyperspectral Image,Chemometrics,Classification

AGRICULTURE & FEED 2

O.13.40

UNEXPECTED EFFECT OF AGRICULTURAL PRODUCTS/FOODS IN NIR MEASUREMENT -LIGHT SCATTERING IN SAMPLE AND CURVATURE AND VARIABILITY IN SAMPLE SHAPES-

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Near-infrared spectroscopy (NIRS) is widely applied in the quality inspection of agricultural products and food, as well as in non-destructive measurement techniques. However, estimation accuracy can be significantly impacted by sample-derived factors such as light scattering within the sample and variations in sample shape and curvature.

This presentation introduces a novel method developed by the authors for precise determination of absorption and scattering coefficients using time-of-flight near-infrared spectroscopy (TOF-NIRS). We also present case studies demonstrating the reduction of light scattering effects to enhance foreign object detection in food. In particular, we propose a visible-NIR single-pixel imaging (Vis-NIR-SPI) method for detecting small insects within food samples. This approach leverages the non-destructive, rapid, and multi-component analysis capabilities of Vis-NIR light while addressing challenges such as dark noise, high scattering, and cost-effectiveness through SPI technology.

Finally, we address corrections for curvature and shape variability in near-infrared hyperspectral imaging (NIR-HSI), highlighting its potential for non-invasive quality assessment of diverse agricultural products.

Keywords
Light scattering,TOF-NIRS,Vis-NIR-SPI,NIR-HSI

O.13.41

NEAR-INFRARED REFLECTANCE SPECTROSCOPY FOR PREDICTION OF NUTRITIONAL COMPOSITIONS IN FERTILISER MATERIALS

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Near-Infrared Reflectance Spectroscopy (NIRS) is an innovative technique that require the absorption or reflection of near-infrared light to facilitate qualitative and quantitative analyses of chemical and physical properties of the test sample [1]. The 'no chemical' concept applied by the NIRS technology is a positive effort in reducing waste and preserving the environment [2]. Near-infrared reflectance spectroscopy has been used to quantify nutrient contents in food, plant and soil materials [3,4]. However, limited information is available on the application of NIRS technique for fertiliser analyses, especially in the tropic regions of Malaysia. The objectives of this study were to develop a robust and reliable non-destructive method for the determination of total nitrogen, total phosphorus, total potassium and total magnesium contents in fertiliser materials, and to support the quality monitoring programme of fertilisers for a sustainable agriculture sector in Sarawak. A total of 1433 fertiliser samples of different formulations were collected as parts of the fertiliser quality monitoring programme for various agriculture, and research and development projects implemented by the Department of Agriculture Sarawak from the year 2020 to 2024. Spectral measurements were performed by scanning the ground samples using a FOSS DS2500 NIRS across the full wavelength range between 400 nm and 2500 nm. Calibration equations were derived using the modified partial least square regression and cross-validation techniques. Several pre-process treatments including scattering, mathematical pre-treatments and aggression methods were optimised to increase the accuracy of the calibration models according to the coefficient of determination for calibration (Rc2), standard error of calibration and cross-validation (one minus the variance ratio, 1-VR). The equation model then validated using an independent set of 20 samples selected randomly. As a reference analysis for total nitrogen, each sample was analysed using Dumas combustion method. For total phosphorus, potassium and magnesium, the samples were digested using a concentrated nitric acid, and the analytes were determined using an inductively coupled plasma-optical emission spectroscopy. The calibration data showed that NIRS has a poor to excellent predictive capacity for total nitrogen, phosphorus, potassium and magnesium for different fertiliser materials. The Rc2 values were ranging from 0.374 to 0.990 for total nitrogen, 0.190 to 0.896 for total phosphorus, 0.058 to 0.970 for total potassium and 0.483 to 0.990 for total magnesium. The wide variation in the element concentrations may be attributed by the differences in the correlation coefficients. The considerable variation within the samples could also be due to the environment and factors affected it during sampling and handling. The accuracy of predictions could be improved by increasing the calibration data set. The quick and chemical-less NIRS technique has the potential to simplify the time-consuming and tedious process of chemical analysis in the laboratory.

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ACKNOLEDGEMENTS: The authors thank the Sarawak Government for financial support and staff of Analytical Chemistry Laboratory for their technical assistance.

Keywords
non-destructive,cost effective,sustainable

O.13.42

POTENTIAL FOR MONITORING DIURNAL CHANGES IN CRASSULACEAN ACID METABOLISM PHOTOSYNTHESIS USING NEAR-INFRARED SPECTROSCOPY AND 1H NUCLEAR MAGNETIC RESONANCE

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Introduction

Photosynthesis in Crassulacean Acid Metabolism (CAM) has rhythmic patterns of a carbon fixation process [1]. These circadian rhythms respond to environmental conditions, making understanding the diurnal variation in photosynthetic metabolism crucial for physiological research. Diurnal changes in CAM photosynthesis have traditionally been assessed by CO2 gas exchange and metabolite content in leaves. Newer methods, such as chlorophyll fluorescence, provide rapid assessment of photosynthetic activity [2], but have not yet been able to capture diurnal changes in metabolism. This study focuses on the use of near-infrared spectroscopy (NIRS) to measure diurnal changes in photosynthetic metabolites. While NIRS has been used to study metabolic processes in agricultural products over weeks to months [3,4], attempts to capture the diurnal variation of carbon cycling photosynthetic metabolism are lacking. We explore the potential of NIR and nuclear magnetic resonance (NMR) spectral analysis of pineapple leaf extracts for this purpose.

Material and Methods

Six 12-month-old pineapple plants were acclimatized to a 12-hour light (9 AM to 9 PM) / dark (9 PM to 9 AM) cycle for three days. Leaf samples (0.3 g FW) were collected from the longest leaves at 9 AM, 3 PM, and 5 PM on the third day and immediately frozen in liquid nitrogen.

Sample Preparation

Each leaf sample was homogenized with 3 mL of TES-NaOH buffer (pH 7.0), followed by centrifugation and filtration. The extracted solution was prepared for analysis (n=54).

NIR Spectral Measurement

The wavelength range was 400–2500 nm at 0.5 nm intervals using an NIRS (DS2500, Foss) with a gold reflector.

NMR Spectral Measurement

For NMR analysis, 0.6 mL of the extract was mixed with 0.1 mL of DSS-D2O as an internal standard. ^{1}H-NMR spectra were measured using a Bruker 500 MHz NMR spectrometer.

Results and Discussion

Diurnal Changes in NIR and NMR Spectra

The second derivative NIR spectra showed minimal changes across sampling times in all wavelength ranges. Principal component analysis (PCA) revealed characteristic scores for sampling times in the 1130–1700 nm range (Figure 1). PCA of the NMR spectra showed characteristic scores for each sampling time in the 3.4–2.5 ppm range, corresponding to glucose, malic acid, and citric acid.

NIR-NMR Correlation Analysis Revealing CAM Metabolic Characteristics

The NMR signals of glucose, fructose, and malic acid were significantly correlated with the NIR signals. Partial least squares regression analysis (PLSR) of the NIR spectra with metabolite concentrations showed that the diurnal changes in estimated concentrations followed trends like those predicted by NMR (Figure 2). In CAM, malic acid is stored during the dark period and consumed in the light period as substrates for sugar production [5]. The diurnal changes in the NIR spectra likely reflect the decarboxylation of malic acid and sugar generation.

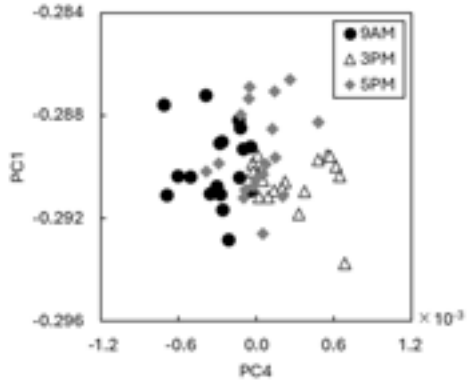


Figure 1. Score plot of PC1 vs. PC4 from PCA of NIR spectra in the 1030–1700 nm range.

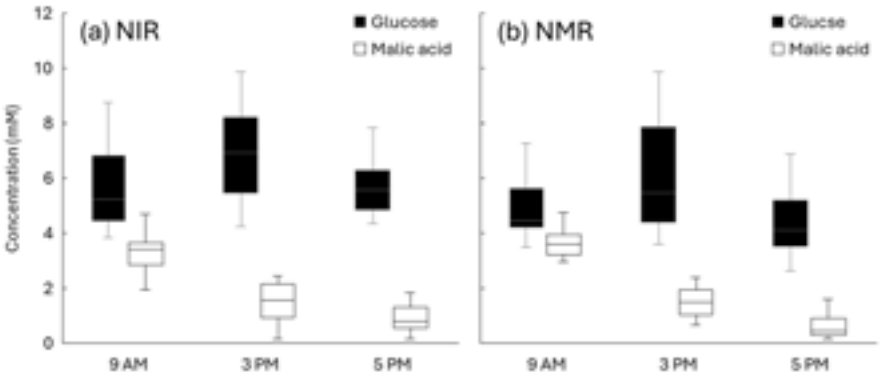


Figure 2. Diurnal changes in glucose, and malic acid concentrations at 9 AM, 3 PM, and 5 PM. The data were estimated using (a)NIR and (b)NMR spectra. The box plots display the median and interquartile range (IQR), while the whiskers extend to the smallest and largest values within 1.5 times the IQR.

Conclusion

This study examined the potential of NIR spectroscopy to measure diurnal changes in photosynthetic metabolites. Individual metabolites quantifying by NIR has several limitations, but the correlation analysis between NIR and NMR spectra suggests that NIR could capture metabolic dynamics. Our future research will focus on continuous monitoring of NIR spectra in interactance from pineapple leaves to develop a non-destructive method for measuring the circadian rhythm of CAM photosynthesis.

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Keywords
crassulacean acid metabolism,photosynthetic metabolites,diurnal changes,nuclear magnetic resonance,principal component analysis

F.13.37

TOWARDS A HANDHELD NIR SPECTROMETER TO EVALUATE THE FRUIT ORGANOLEPTIC AND NUTRITIONAL QUALITY IN THE CONTEXT OF ORGANIC FRUIT BREEDING

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During the last few decades, the use of near infrared spectroscopy (NIRS) as a rapid and non-destructive tool for determining quality and nutritional parameters of fruits has been largely investigated.

InnOBreed for 'Innovative Organic fruit Breeding and uses' is a European project (2022-2026) which aims to promote and unite existing 'Innovative Solutions" focused on the participative selection in organic production of fruit varieties from a broad range of species (apple, pear, peach, apricot, plum, almond, cherry and citrus). As fruit quality evaluation is time consuming, NIRS could help, as it enables non-destructive and rapid phenotyping of fruit and could contribute to improve the practices in breeding and selection programs of pome and stone fruit trees. Therefore, one of the objectives is to develop and validate the application of NIRS for the analysis of fruit organoleptic and nutritional quality, and especially to deliver a high-throughput, efficient and robust tool for a more efficient selection.

Experiments were carried out in 2023 and 2024 by several European partners. According to the partners' expertise, a panel of representative varieties of apple, pear, apricot and peach was characterized at harvest and after a period of post-harvest, and different NIR instruments were used, including both benchtop and portable tools. Among quality traits defined as reference data, the firmness, soluble solid content (°Brix), titratable acidity, color (L*a*b), and dry matter content were evaluated for organoleptic traits and the content of fibres and polyphenols for nutritional quality.

In this presentation a focus is made on our experiment done on apple and pear. Spectra from benchtop spectrometer (XDS from FOSS) and handheld instruments (F-750 from Felix instrument and MicroNIR from Viavi) were used to develop our own calibration models to predict different quality parameters. Validation of these models will be performed during the 2025-2026 seasons with new experiments to be carried out directly in field conditions by fruit growers, considered as 'case studies'.

At the project scale, the challenge will be to pool the data from the different partners in a common database in order to build robust predictive models based on a large European fruit variability using in priority spectra acquired with the Felix instrument and various chemometrics approaches applied to both spectral and reference data.

ACKNOLEDGEMENTS: Horizon Europe-CL6-2021-BIODIV-01 programme, convention no. 101061028

Keywords
near infrared spectroscopy,handheld instruments,fruit quality,organic fruit breeding

F.13.38

RAPID DETERMINATION OF TOTAL RECOVERABLE SUGARS IN SUGARCANE JUICE USING NEAR-INFRARED SPECTROSCOPY AND MULTIVARIATE MODELING

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Sugarcane has become Brazil's primary bioenergy source due to its high efficiency and robust ethanol industry. The 2023/24 harvest is projected to reach a record 713.2 million tons, a 16.8% increase compared to the previous season, with an average yield of 85,580 kg/ha. Sugarcane quality is assessed by sucrose concentration, estimated indirectly in mills through technological analysis (TA). TA evaluates wet bagasse weight (WBW), soluble solids (SS), and sucrose content via saccharimetric analysis (LS), enabling the calculation of key parameters like sucrose content, purity, reducing sugars, and total recoverable sugars (TRS). TRS, a vital economic indicator, is used to determine sugar yield per ton of cane. While TA is relatively simple, LS determination requires Octapol® reagent for juice clarification, making it costly and time-consuming.

Furthermore, TA results need to be more accurate. High-performance liquid chromatography (HPLC) offers exact quantification of sucrose, glucose, and fructose but is expensive, labor-intensive, and requires skilled analysts. This study proposed a rapid and accurate method for directly determining total recoverable sugars (TRS) in sugarcane juice using near-infrared spectroscopy (NIR). This method correlates NIR spectra with TRS values obtained via technological analysis (TRSTA) and high-performance liquid chromatography (TRSLC) through a multivariate model employing partial least squares regression (PLS). TRSTA values were indirectly determined using CONSECANA equations, while HPLC analysis was used to quantify sugars to calculate TRSLC. NIR spectra of the juice were used as independent variables, while TRSTA and TRSLC were used as dependent variables. A high correlation (R = 0.90) was observed between TRSTA and TRSLC values. The results presented in Table 1 show the statistical parameters of the models for TRS quantified through technological analysis and HPLC. For each analysis, the selection of variables using the Ordered Predictors Selection (PLS-OPS) improved the parameters of the models compared to the models using all variables (PLS). This improvement is evident when one notices the decrease in the root mean square error of prediction (RMSEP), from 9.05 to 7.8 kg/ton in the case of TRSTA and from 11.22 to 9.8 kg/ton in TRSLC. The reduction in RMSEP indicates a better ability of the PLS-OPS models to predict the real TRS values. The RMSEP values for TRSLC are higher compared to TRSTA due to the higher numerical values of TRSLC. Additionally, HPLC is a more accurate separation and quantification technique for sugars, resulting in generally higher values for TRSLC, which is naturally reflected in a larger dataset and RMSEP values.

Table 1. Parameters of the PLS models for the TRS property

Properties (kg ton^{-1})	Method	RMSEC	Rc	RMSEP	Rp	LV
TRS^{TA}	PLS	7.03	0.91	9.05	0.88	10
	PLS-OPS	5.98	0.94	7.8	0.91	10
TRS^{LC}	PLS	8.39	0.88	11.2	0.81	10
	PLS-OPS	8.82	0.86	9.8	0.86	10

ACKNOLEDGEMENTS: RIDESA, CAPES (Project 001), FAPEMIG.

Keywords
Sugarcane juice,Technological Analysis,HPLC,Total recoverable sugars

F.13.39

FEATURE EXTRACTION FROM AN ENTIRE TWO-DIMENSIONAL CORRELATION MAP USING CONVOLUTIONAL AUTOENCODER TO ENHANCE SPECTROSCOPIC DISCRIMINATION OF GEOGRAPHICAL ORIGINS OF AGRICULTURAL PRODUCTS

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Hanyang University, Seoul, SOUTH KOREA

A convolutional autoencoder (CAE) has been demonstrated as an effective tool for extracting features from entire two-trace two-dimensional (2T2D) correlation maps while avoiding spatial information loss. An autoencoder (AE) is a neural network-based manifold learning model of non-linear features and an unsupervised method that does not require excessive training samples. CAE incorporates an AE structure composed of convolutional layers that recognize spatially distributed features in multi-dimensional data related to target analysis. Due to this ability, CAE can extract descriptive features from 2T2D maps while considering the spatial distribution of correlation information through successive convolutional layers, so the extracted features are potentially representative of the whole map. Particularly, pointwise convolution was used as the first layer in the CAE structure to combine all information from both synchronous and asynchronous maps.

To evaluate the proposed scheme, two spectroscopic datasets, near-infrared (NIR) and Raman spectra of chili pepper samples and NIR spectra of perilla seed samples, were employed for hetero- and homo-spectral 2T2D correlation analysis, respectively. Then, CAE-extracted features from the hetero- and homo-spectral 2T2D correlation maps were used for the discrimination of the geographical origins of chili pepper and perilla seed samples, respectively, using support vector machine (SVM). Also, principal component (PC) scores obtained from the raw spectra and slice spectra chosen from the 2T2D correlation maps were employed as inputs for SVM. Accuracy improved by employing CAE-extracted variables in both cases compared with those using slice spectra chosen from the 2T2D maps. Moreover, to provide clearer insight into the models, gradient-weighted class activation mapping (Grad-CAM) informing the variables significantly contributed to the discrimination was employed in parallel.

ACKNOLEDGEMENTS: This work was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (2020R1A6A1A06046728, RS-2023-00274557).

Keywords
Two-dimensional correlation,Convolutional Autoencoder,Feature Extraction,Geographical Origin

POSTER SESSION A

MONDAY JUNE 9, 2025 **13:15-14:00**

AGRICULTURE & FEED

P01.01.01

LOQUAT INTERNAL QUALITY PREDICTION AND VARIETY CLASSIFICATION WITH HYPERSPECTRAL IMAGING

Salvador Castillo-Girones [1], Marina Lopez-Chulia [1], Sergio Cubero [1], Sandra Munera [2], Alejandro Rodríguez [2], Juan Gómez-Sanchís [3], José Blasco [1]

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Food fraud and quality pose significant concerns for farmers, the food industry and consumers. One form of fraud involves the mixing of similar cultivars of fruits despite notable disparities in quality and sensory attributes, thereby risking consumer dissatisfaction. Moreover, given consumers' preference for flavorful fruits and the necessity for farmers to find optimal harvest times, parameters like Total Soluble Solids (TSS) and Titratable Acidity (TA) assume paramount importance. Consequently, the ability to non-destructively predict cultivar types, TSS, and TA content holds immense value. This study focuses on two loquat cultivars, Algerie and Xirlero, cultivated in Callosa d'en Sarrià (Alicante-Spain), which exhibit similar external appearances but distinct sensory profiles. Utilising 600 samples equally split between both cultivars (300 from Algerie and 300 from Xirlero), the study employed hyperspectral imaging (900-1700 nm) to capture images of the fruits. The mean spectra of each fruit were extracted, and a Standard Normal Variate pre-treatment was applied for scatter correction. The spectra were then divided into a training set (70%) and an independent test set (30%). Two classification models, Partial Least Squares Discriminant Analysis and Support Vector Machines, were used to differentiate between the two cultivars. Additionally, Partial Least Squares Regression and Support Vector Machines were employed to create predictive models for Total Soluble Solids (TSS) and Titratable Acidity (TA). All models demonstrated accuracy rates exceeding 85% in cultivar identification. Regarding qualitative parameter predictions, R^2 values surpass 90% for TSS prediction and 80% for TA prediction. These results underscore the potential of hyperspectral imaging as a promising technology for controlling food fraud and predicting the internal quality of loquats.

ACKNOLEDGEMENTS: This work is part of the MICIU AEI PID2023-150192OR-C31, C32 and C-33 with the support of FEDER funds and GVA-PROMETEO CIPROM/2021/014. Salvador Castillo Gironés thanks INIA for the FPI-INIA grant number PRE2020-094491, co-funded by EU EFS funds. Sandra Munera thanks the postdoctoral contract Juan de la Cierva-Formación (FJC2021-047786-I) co-funded by MCIN/AEI/10.13039/501100011033 and European Union NextGenerationEU/PRTR. This work is co-funded by the AEI through the project PID2023-150192OR-C31

Keywords
Loquat,Variety,Quality,Hyperspectral,Machine learning

P01.01.02

PROXIMAL SENSING IN FIELD TRIALS TO EXPLORE THE FEASIBILITY AND BUILD THE FOUNDATION FOR EFFECTIVE BIOTIC STRESS MONITORING FROM HYPERSPECTRAL SATELLITES

François Stevens [1], Louise Leclère [2], Yannick Curnel [2], Viviane Planchon [2], Maxime Troiani [3], Pierre Defourny [3], Vincent Baeten [1], Philippe Vermeulen [1]

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Agriculture is both a significant contributor to the climate and biodiversity crises and one of their primary casualties. However, agriculture has also the potential to address these crises and even mitigate them. The use of adapted crop varieties and the early detection of biotic and abiotic stresses can help to rationalize the use of chemical inputs, protect soils, optimize yields and ensure food security. Currently, digital agriculture uses satellites to monitor crop properties, detect issues, support decision-making and optimize agricultural practices. Today, most remote sensing applications are based on multispectral data in the visible (VIS) and near infrared (NIR) range. The potential contribution of hyperspectral technology needs to be assessed.

The SPAGHYTI project ('Space for Agriculture with Hyperspectral Teledetection & Innovation') proposes to develop solutions for agriculture based on hyperspectral imaging. These will provide the farmers, authorities or researchers with relevant information and actionable insights directly at parcel level through user-friendly applications. The project also aims to support the development of a constellation of small satellites carrying hyperspectral cameras, enabling access to high-value information at a marginal cost with frequent revisits. Two applications are currently defined in the user segment: assessment of nitrogen status and detection of diseases in soft winter wheat. This presentation focuses on disease detection.

To assess the feasibility of an approach based on hyperspectral satellite imagery, which were not available in sufficient quantities, ground spectral data (not imaged) were collected. Two intensive field measurement campaigns were organised on field trials and on farmers' fields in Wallonia (BE). Yellow rust, brown rust, take-all, septoria leaf blotch, fusarium head blight and common bunt have developed to different degrees depending on natural infection pressure, inoculation infection and fungicide treatment. At different growth stages, VIS-NIR reflectance spectra (350-2500 nm) were collected using the ASD FieldSpec4 spectrometer equipped with the pistol grip. Each spectrum was considered to represent the whole trial plot or field landscape unit. Disease-specific and global health status scores were recorded visually. A drone campaign and two PRISMA images covering some of the fields were also exploited to upscale the models.

Diverse modelling approaches were used to assess the effect of biotic stress on spectral properties and the potential to detect it. Firstly, multiple spectral indices mentioned in the literature for their ability to characterise vegetation or identify disease symptoms were calculated. Secondly, additional indices were developed using wavelength combinations selected with genetic algorithm. Finally, classical multivariate methods (PLS-DA, SVM-DA) were used to discriminate between different levels of infection. The different indices and models obtained were validated on trials and farmer's fields, with different varieties and phenological stages to determine their robustness. The performances of the predictive models validated on ground-based data will be assessed on satellite imagery data.

ACKNOLEDGEMENTS: The Spaghyti project is funded by the Public Service of Wallonia (SPW) Economy, Employment, Research - Skywin-Wagralim Clusters – Call 33, under grant agreement no. 8614. The consortium also includes the industrial partners Constellr, Amos, and Deltatec, involved in various aspects of the project. The authors acknowledge the staff of the Crops and forest health Unit, the Crop production Unit and the Quality and authentication of agricultural products Unit of the Walloon Agricultural Research Centre for setting up the trials and collecting the ground data.

Keywords
biotic stress,winter wheat,proximal sensing,hyperspectral,field trial

P01.01.03

LUPINS - FROM WILD PLANT TO EDIBLE, PROTEIN-RICH CROP, ASSISTED BY CHEMOMETRIC STUDY AND MACHINE LEARNING

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The use of lupin seeds in food and stock feed has garnered much interest in recent years due to their dietary value and potential as an alternative gluten-free protein source [1]. A major challenge in transforming this protein-rich legume into a food crop is its bitterness and potential toxicity, generally attributed to quinolizidine alkaloids (QAs) produced by lupin seeds of all species [2]. To allow safe consumption, Australia, New Zealand, the UK, and France have determined a minimal threshold for QA levels (<200 mg/g) [3-4], which wild lupin seeds of different species often exceed [5]. As QAs also have beneficial therapeutic qualities, not all QAs are hazardous or require full removal. Thus, more selective approaches are sought and might be achieved through prior consumption treatments or by agricultural modes of cultivation.

To mitigate this challenge, we developed an indicative toolbox to evaluate the quality of the agricultural produce. Reliable, fast, and affordable evaluation tools for agro-product quality are needed to provide growers and developers with direct feedback on the results of their practices. Using Artificial Intelligence (AI) to develop machine learning abilities to assess qualities in the field of agricultural developments towards sustainability will greatly save costs and enable prompt control over the crop products [6] (Drinkwater et al., 2007).

Motivated by the potential of lupin seed to serve as food or feed crops for their beneficial qualities and high protein values, together with the need to monitor QAs levels in the seed, we explored the potential of NIRS calibrations to enable these goals. QA compositions and the total amount of lupin seeds were assessed using Liquid Chromatography (LC) and gas chromatography (GC) coupled with mass spectroscopy (LC-MS-MS and GC-MS) [5]. Protein amounts and phenol total amount were determined. This database was used to create NIRS calibrations.

FT-NIR multi spectra [7] coupled with the related chemical properties are statistically manipulated for decades as an innovative technique to construct classification and calibration models to predict targeted chemical properties [8]. This further enables the characterization of an unknown sample using its NIRS spectrum.

To establish such a chemometric toolbox we sampled 31 local wild lupin populations of 2 species, L. pilosus and L. palaestinus, and 7 other non-local lupin seeds (with five replicas sampled for each population). We used Near-Infrared Spectroscopy and FT-NIR tool sets to construct prediction equations for phytochemical evaluation. Prediction equations based on coupling between NIRS readings of the ground seeds and its measured chemical qualities, calibrated with a broadened database suggested a strong, reliable, and affordable tool [9]. Quantifying secondary metabolites as alkaloids and phenols and other attributes such as protein content was achieved. Developing such an inexpensive tool allowed us to push further the possibility of lupin development into an agricultural food crop.

ACKNOLEDGEMENTS: The authors wish to thank Ms. Kineeret Rozenblat for her significant contribution to the development of the NIRS calibrations.

Keywords
lupins,alkaloids,proteins,chemometrics,NIRS

P01.01.04

EXPLORING REGIONAL AND ANNUAL EFFECTS ON AUSTRIAN WHEAT USING ASCA AND NIRS

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Wheat is one of humanity's most important agricultural crops. Near-infrared spectroscopy (NIRS) has been employed since the 1970s for wheat quality assessment due to its non-destructive, rapid, and cost-efficient nature. To fully leverage these advantages of NIRS, complex calibration development often involving resource-intensive reference methods is required (1).

This study explored the combination of analysis of variance simultaneous component analysis (ASCA) with NIRS to investigate regional and annual effects on Austrian wheat quality and examined the influence of spectral preprocessing methods. Our findings demonstrate significant ($p < 0.001$) influences of year, sampling site, and their interaction on wheat composition. Spectral preprocessing notably impacted ASCA outcomes, with extended multiplicative scatter correction yielding the least residual variance and most effectively highlighting annual and regional effects. ASCA revealed that annual differences account for most of the variance observed in the NIR spectra, likely due to annual weather variations, while regional differences were less pronounced. Using ANOVA on the results obtained from an on-demand calibration led to the same conclusions as those obtained by ASCA (2).

There are two major outcomes of this study. Firstly, ASCA can be used to assess the significance of regional or annual effects on wheat quality without the need to establish calibrations for parameters such as protein, moisture, fat, and starch content. Secondly, ASCA holds potential as a tool to guide spectral preprocessing in chemometric workflows. Ultimately, this study underscores ASCA's potential as a simple, green, and efficient alternative for studying effects in NIR datasets, offering insights beyond traditional calibration-dependent approaches.

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ACKNOLEDGEMENTS: This research is part of the PHOTONFOOD project, which received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 101016444 and is part of the PHOTONICS PUBLIC PRIVATE PARTNERSHIP.

Keywords
Wheat,ASCA,spectra preprocessing

P01.01.05

AN OPEN-SOURCE CHEMOMETRIC PIPELINE AS A RESEARCHERS' TOOL

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Technological advancements have greatly improved accessibility and affordability of NIR spectroscopy technology. A range of spectrometers is now available, including desktop and portable handheld types. Much less has been accomplished in terms of releasing "agnostic" software that can easily integrate with the variety of spectrometers, hence limiting the users to specific NIR solutions and to the range of pre-loaded NIR models. An open-source pipeline was developed by our group to enable researchers to use NIR spectroscopy as a researcher's tool, including development of NIR models. In this presentation we will provide an overview of the software pipeline, followed by specific examples on how researchers can use the software pipeline to: 1) develop accurate experiment-wise NIR models, and 2) use the software pipeline to reduce the cost of laboratory analyses by approximately 70%. The data and NIR models from forages[1] and tobacco[2] presented in this talk have already been published in peer-reviewed journals; however, this is the first time that we will present the software pipeline and the NIR analysis approach to use the pipeline as a researcher's tools for a variety of spectrometers ranging from benchtop to handheld portable devices.

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Keywords
open-source,pipeline,forages,tobacco,NIR

P01.01.06

THE APPLICATION OF NIR HANDHELD DEVICES FOR SOIL MONITORING

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Monitoring Soil health will be a requirement of all Member States under the new EU Soil Monitoring and Resilience Law [1], and soil spectroscopy provides a rapid and cost-effective alternative to traditional wet chemistry methods for national scale monitoring campaigns [2]. MIR models were derive using a Bruker mid-infrared (MIR) benchtop spectrometer to for soil particle size (sand, silt, and clay), organic matter (OM), pH (SMP buffer), total carbon (TC), total organic carbon (TOC), total nitrogen (TN), bulk density (BD) and other parameters. These models were validated using external soil archives to verify the applicability across diverse soil types and this study.

This study aims to investigate the potential to transition to portable near-infrared (NIR) devices, specifically the NeoSpectra (1350–2500 nm) for the same suite of parameters. Soil samples (n ~ 800) representing the northern half of Ireland were processed under standardised conditions (drying at 40°C for 48 hours and sieving to <2 mm). Samples were scanned using the handheld device, and regression models based on support vector regression (SVR) were built to predict OM, TC, TOC, TN, pH (SMP buffer), clay, sand, silt, and BD. Initial results demonstrated promising predictive accuracy for several soil parameters, with accuracy metrics (R2val, RMSEP, RPIQval) indicating good model performance: OM (0.62, 2.33, 2.23), TC (0.68, 1.04, 2.10), TOC (0.71, 0.91, 2.25), TN (0.67, 0.09, 2.30), pH (SMP buffer) (0.72, 0.24, 2.79), clay (0.78, 3.09, 2.91), sand (0.62, 7.09, 2.12), silt (0.35, 5.99, 1.50), and BD (0.73, 0.07, 2.59).

The results indicate that NIR handheld devices can predict soil properties with moderate to good accuracy, except for silt, with applications varying from nutrient management to monitoring networks, enabling reduced costs and time required for large scale monitoring. This research contributes to improving soil resilience, carbon stock monitoring, and soil health monitoring [1]. Future work will focus on refining these models, combining the spectra with other handheld devices to complete the full NIR range (900 – 2500 nm), increasing the number of soil archives in our dataset, and investigating their applicability for field analysis to support the sustainable soil management.

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ACKNOLEDGEMENTS: Teagasc staff.

Keywords
Handheld NIR,Soil Monitoring Law,Soil health,soil analysis,chemometrics

P01.01.07

IN-FIELD HYPERSPECTRAL IMAGING FOR GRAPE QUALITY ASSESSMENT USING COMPUTED TOMOGRAPHY IMAGING SPECTROSCOPY

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Grape quality assessment is crucial for optimizing vineyard management and ensuring product excellence in the wine industry [1][2]. Beyond grape quality, hyperspectral imaging holds promise for evaluating a wide range of crops and fruits, making it a versatile tool for precision agriculture. This study, a follow up study from [3], focuses on in-field measurements of grape quality using a snapshot hyperspectral imaging (HSI) system based on Computed Tomography Imaging Spectroscopy (CTIS). This study was conducted as a collaborative effort between the University of Southern Denmark, Cabra Robotics, and Newtec Engineering. The system was mounted on a field robot and deployed across five excursions during 2023 and 2024 in a vineyard in southern Denmark, capturing data on Rondo wine grapes at different ripeness stages. °Brix and pH values were measured using a refractometer and a pH meter as reference methods. Partial Least Squares Regression (PLSR) models were used to correlate CTIS spectra with reference measurements. Compared to a state-of-the-art line-scan HSI system, the CTIS system demonstrated strong performance despite its narrower spectral range. Its affordability, portability, and reduced motion error susceptibility further highlight its potential for in-field crop quality monitoring.

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ACKNOLEDGEMENTS: We acknowledge partial funding from the Innovation Fund Denmark (IFD) under File No. 1044-00053B. We acknowledge partial funding from Food & Bio Cluster Denmark and the SDU Climate Cluster.

Keywords
Agricultur,Snapshot Hyperspectral Imaging,Partial Least Squares Regressi

P01.01.08

RAPID IDENTIFICATION OF BLACK RICE VARIETIES FROM NORTHEAST INDIA BY DETERMINING TOTAL FLAVONOID CONTENT AND ANTIOXIDANT ACTIVITIES USING NEAR-INFRARED SPECTROSCOPY

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This study aimed to perform identification and discriminant analysis between the three principal Black Rice (Oryza sativa) varieties collected from Northeast India based on the simultaneous rapid determination of the Total Flavonoid Content (TFC) and Antioxidant activities using a Near Infrared Spectrometer (NIRS). The purple-pigmented Black rice is rich in flavonoids, offering high nutritional benefits and antioxidant properties. It has been reported to prevent various ailments, including cancer, diabetes, cardiovascular diseases, and metabolic disorders. As a significant contributor to the economy of its cultivating states, the three black rice varieties require rapid qualitative differentiation. To ensure the authenticity of a specific variety, it is crucial to standardize the varieties based on their metabolite profile and therapeutic efficacy. Furthermore, due to the increasing popularity of Black rice, this rapid qualitative gradation is becoming essential for gaining confidence from both cultivators and consumers [1].

The Black rice samples were collected from different parts of Northeast India, mainly the districts of Manipur and Meghalaya. The Black rice samples were powdered and extracted with a hydroalcoholic solution (70:30, v/v). Both the TFC and antioxidant activities were determined by validated methodologies based on the literature. Besides, to acquire spectroscopic data, finely powdered samples (mesh size of 125 µm) were taken to obtain the spectral data. A developed portable NIRS (Ayudyog Pvt. Ltd., India) recorded the diffused reflectance between a wavelength region of 900 -1700 nm with a spectral resolution of 2.92 nm [2]. The raw spectral data was pre-processed using multiple techniques such as SNV, MSC, 1D, 2D, and Savitzky-Golay. Successive Projections Algorithm (SPA) was employed to extract the most informative wavelengths. The preprocessed spectra were analyzed over the selective wavelength regions extracted by SPA, and a predictive model was developed using partial least square regression (PLSR). Partial Least Squares Discriminant Analysis (PLS-DA) was studied to classify the varieties.

The most efficient method was the predictive model utilizing the pre-processed data, SNV + 1D, and selected wavelengths. SPA indicated that the 1080 to 1550 nm wavelength range was the most informative and significant area. The spectral data within this wavelength range demonstrated the optimal calibration model, effectively predicting TFC values and antioxidant activities with a lower RMSEP (0.34 mass fraction) and higher R^2p (0.896) and RPD (3.98). The three-dimensional plot generated by the Partial Least Squares Discriminant Analysis (PLS-DA) provided a simple and interpretable visualization of the three black rice varieties. This analysis produced three distinguishable clusters among the varieties.

It can be concluded that the developed NIR spectroscopic model, combined with SPA and PLS-DA, may enable the rapid and non-destructive identification of black rice varieties based on the determination of TFC and antioxidant activity. This approach could effectively identify and offer informative insights into gradation based on therapeutic effectiveness.

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ACKNOLEDGEMENTS: The authors express gratitude for the financial support from the Department of Biotechnology, Ministry of Science and Technology, India, Sanction No.: BT/PR45213/NER/95/1933/2022.

Keywords
Black rice,TFC,Antioxidant activity,SPA,PLS-DA

P01.01.09

IMPROVING LOQUAT HARVEST MATURITY ASSESSMENT: A COMPARISON OF NIRS AND COLOUR INDEX METHODS

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Loquat fruit is ready to harvest when it achieves a soluble solid content (SSC) level above 10°Brix [1], [2]. Traditional SSC determination involves analysing the juice, which is a destructive method. SSC in loquat has been correlated with skin colour, leading to the development of harvest maturity indices based on external colour. Colour charts are available for maturity evaluation that match the surface colour for specific cultivars. To ensure an optimum stage of maturity that meets consumer expectations, 'Algerí' cultivar should be harvested after reaching a colour index (CI) of +6, while 'Xirlero' fruit achieve commercial maturity at CI = +10 [3].

However, estimation of colour as a maturity indicator presents challenges due to fruit colour heterogeneity and varietal differences. Moreover, relationship between skin colour and palatability changes along the season [4]. Therefore, it is interesting to look for other non-destructive alternatives to estimate internal fruit maturity accurately and objectively. This study evaluates the potential of non-invasive technologies based on near-infrared spectroscopy (NIRS) to determine the optimal harvest time for loquat fruit, rather than relying on colorimetry.

Loquats from two cultivars ('Algerí' and 'Xirlero') were harvested over four consecutive weeks during the 2023 commercial harvest period and five weeks in the 2024 campaign. A total of 1,080 fruits (540 per cultivar) were analysed across both years. The dataset was split into 80% for training and testing and 20% for validation. Spectral data was acquired in reflectance mode for each fruit with a spectrometer, and SSC was measured by refractometry. Models were developed using partial least squares regression (PLSR) to correlate visible and near-infrared spectra with SSC for each cultivar. Three spectral ranges were studied (450-750 nm, 600-960 nm, and 980-1695 nm). The best SSC predictions achieved an R^2 of 0.84 for 'Algerí' and 0.82 for 'Xirlero'.

Fruits used for model validation (108 per cultivar) were evaluated to compare SSC predictions from NIRS with colorimetry. Based on SSC and CI thresholds, the percentage of misclassified fruits was calculated for each method. For 'Algerí', colorimetry resulted in 11% of fruit being misclassified, whereas SSC prediction with NIRS reduced this to 4%. For 'Xirlero', the misclassification rates were 24% for colorimetry and 13% for SSC prediction with NIRS.

These results demonstrate the potential of NIRS technology for evaluating the internal quality of loquats, enabling more accurate determination of harvest maturity compared to colour-based indices.

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ACKNOLEDGEMENTS: This work was partially funded by ERDF funds of the GVA 2021-2027 and projects GVA-IVIA 52204B and GVA-PROMETEO CIPROM/2021/014.

Keywords
Chemometrics,Near-infrared spectroscopy,Harvesting,Fruit maturity,Internal properties

P01.01.10

APPLICABILITY OF NEAR INFRARED SPECTROSCOPY FOR PREDICTING RUMEN METHANE PRODUCTION FROM ALFALFA SILAGE USING IN VITRO METHODS

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BACKGROUND

The determination of gas production is crucial in ruminant nutrition and feed science research. However, common evaluation methods are often labor-intensive, costly, or raise ethical concerns regarding animal welfare [1,2]. Previous studies have demonstrated that near infrared spectroscopy (NIRS) can effectively predict rumen methane production, offering a rapid, cost efficient, and less invasive alternative. [3,4]. As a key roughage for ruminants, alfalfa silage also contributes to methane emissions. This study aims to develop predictive models for gas and methane production from alfalfa silage digestion using NIRS.

METHOD

In vitro method was used to measure 24- and 48-hours gas production and methane emissions from 88 alfalfa silage samples. Total digestible nutrients (TDN) and relative feed value (RFV) were calculated based on acid detergent fiber (ADF) and neutral detergent fiber (NDF) contents. Fresh and dry samples were scanned separately using an NIR analyzer to generate two spectral datasets. The spectral data were preprocessed using detrend and standard normal variate (SNV) techniques. Partial least squares (PLS) regression was then applied to correlate the NIRS predicted values with laboratory measurements using UCal™ version 4.0 software (Unity Scientific, Brookfield, CT, USA).

RESULT

In Table 2 and 3, the model demonstrated stronger performance for RFV (R2CrV=0.821; SECrV=7.651; RPD =2.36) compared to TDN (R2CrV=0.763; SECrV =0.729; RPD =2.05). Unfortunately, the predictions for gas production were suboptimal in both dry and fresh samples, with the best results observed for 24h Gas (R2CrV=0.254; SECrV =2.586; RPD =1.06), 48h Gas (R2CrV=0.264; SECrV =2.941; RPD =1.15), 24h methane (R2CrV=0.374; SECrV =0.273; RPD =1.26), and 48h methane (R2CrV=0.479; SECrV =0.325; RPD =1.39). Overall, the models exhibited better predictive ability for methane production than for gas production, with 48-hour predictions outperforming those at 24 hours. The physical properties of the samples did not significantly affect model performance.

CONCLUSION

The models for alfalfa silage feeding value indicators (TDN, RFV) showed moderate success, whereas all gas production models performed poorly. One possible explanation is the presence of numerous uncontrollable factors during the in vitro experiment. Additionally, longer incubation times appeared to improve model performance, suggesting that NIRS may be less suitable for predicting short-term gas production. Further research should focus on enhancing model accuracy by extending in vitro incubation times or adopting more precise gas collection methods.

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Table 2. Calibration and cross-validation statistics for NIRS analysis of chemical composition of alfalfa silage (n = 88) based on dry matter basis.

Statistics	TDN	RFV	24h Gas	48h Gas	24h CH_4	48h CH_4
N[1]	84	85	86	87	84	81
Range	6.25	73.27	10.73	14.63	1.533	2.145
Mean	72.283	144.92	19.289	25.823	1.737	2.408
SD	1.498	18.04	2.757	3.377	0.346	0.451
Mathematical treatment[2]	4,16,16	2,16,16	3,8,8	3,4,4	3,4,4	1,8,8
PLS factors[3]	10	11	6	4	2	1
Calibration						
R^2_C[4]	0.881	0.905	0.506	0.559	0.443	0.491
SE_C[5]	0.517	5.552	1.938	2.242	0.258	0.322
Cross-validation						
R^2_{CV}[6]	0.763	0.821	0.230	0.264	0.327	0.479
SE_{CV}[7]	0.729	7.651	2.623	2.941	0.282	0.325
RPD[8]	2.05	2.36	1.05	1.15	1.23	1.39
RER[9]	8.57	9.58	4.09	4.97	5.44	6.60

[1] Number of samples, exclusive of outliers, used in building the calibration dataset. [2] In mathematical treatments, the first, second, third, and fourth numbers indicate derivative order, gap (nm) over which the derivatives are computed, and derivative smooth, respectively. [3] Number of factors in partial least square equation, which was selected according to the minimum SE_{CV}. [4] R^2_C = coefficient of determination of calibration. [5] SE_C = standard error of calibration. [6] R^2_{CV} = coefficient of determination of cross-validation. [7] SE_{CV} = standard error of cross-validation. [8] RPD = ratio percentage deviation (standard deviation/SE_{CV}). [9] RER = range error ratio (range/SECrV).

Table 3. Calibration and cross-validation statistics for NIRS analysis of chemical composition of alfalfa silage (n = 88) based on fresh matter basis

Statistics	TDN	RFV	24h Gas	48h Gas	24h CH_4	48h CH_4
N[1]	83	85	83	79	85	83
Range	6.25	73.27	10.72	14.44	1.533	2.102
Mean	72.199	144.455	19.253	25.908	1.731	2.450
SD	1.421	17.536	2.754	3.351	0.344	0.434
Mathematical treatment[2]	2,16,16	2,16,6	3,16,16	2,4,4	1,16,16	2,16,16
PLS factors[3]	9	11	12	1	6	3
Calibration						
R^2_C[4]	0.836	5.337	0.612	0.073	0.521	0.503
SE_C[5]	0.576	0.907	1.715	3.226	0.238	0.306
Cross-validation						
R^2_{CV}[6]	0.712	0.761	0.254	0.042	0.374	0.428
SE_{CV}[7]	0.797	8.952	2.586	3.259	0.273	0.329
RPD[8]	1.78	1.96	1.06	1.03	1.26	1.32
RER[9]	7.84	8.18	4.15	4.43	5.62	6.39

ACKNOLEDGEMENTS: This study was financially supported by Cooperative Research Program for Agriculture Science and Technology Development (Project No. RS-2024-00398233) Rural Development Administration, Public of Korea.

Keywords
Alfalfa Silage,In vitro incubation,Methane,Near Infrared Spectroscopy

P01.01.11

BASIC STUDY OF NEAR-INFRARED SPECTROSCOPY FOR EVALUATING GLUCOSE AND MALIC ACID DYNAMICS IN CRASSULACEAN ACID METABOLISM USING STANDARD SOLUTIONS

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Introduction

In Crassulacean Acid Metabolism (CAM) plants, glucose synthesized during the light period serves as a substrate for malic acid synthesis in the dark period. Therefore, during the light period, glucose increases while malic acid decreases, resulting in an increase in the glucose-to-malic acid ratio. In contrast, during the dark period, this ratio decreases as malic acid accumulates and glucose declines. These diurnal variations in the glucose-to-malic acid ratio reflect the dynamics of photosynthetic metabolism [1] and are influenced by enzymatic activities such as malate dehydrogenase. Since these metabolic fluctuations are influenced by environmental factors such as water status, nutrient levels, light intensity, and temperature, they are considered indicators of plant stress. However, a rapid and non-destructive method for measuring these metabolite dynamics has not yet been established. This study aims to evaluate the accuracy and applicability of near-infrared spectroscopy (NIRS) for detecting photosynthetic metabolites by analyzing the behavior of glucose and malic acid under controlled conditions. We investigated the feasibility of using NIRS to detect metabolite variations with standard solutions.

Materials and Methods

Standard solutions (SS) of glucose and malic acid were prepared by dissolving each reagent in ultrapure water at concentrations of 1, 2, 4, 6, and 8% (n=5). Additionally, mixed solutions (MS) with glucose-to-malic acid ratios ranging from 0.2 to 1.6 were prepared (n=12).

NIR Spectral Measurement

The wavelength range was 400–2500 nm at 0.5 nm intervals using an NIRS (DS2500, Foss) with a gold reflector.

Results and Discussion

NIR Spectra

The second derivative spectra of the SS were similar, with water absorption peaks observed at 1400–1600 nm and 1900–2100 nm. To identify characteristic absorbance changes, difference spectra were compared across concentrations. Spectral peaks in the 1300–1500 nm and 2200–2400 nm regions showed distinct patterns between glucose and malic acid.

Partial Least Squares (PLS) Regression Analysis for Identifying Relevant Wavelengths

Higher prediction accuracy was achieved in specific wavelength ranges compared to the full-wavelength PLS model. For glucose, the 1800–2400 nm range achieved an RMSECV of 0.28%, while for malic acid, the 1200–1800 nm range showed an RMSECV of 0.49% (Table 1). These wavelength ranges align with those found in previous studies [2], confirming their relevance for glucose and malic acid absorption. However, the relative RMSE was 15.1% for glucose and 36.3% for malic acid, highlighting some limitations in quantification.

Principal Component Analysis (PCA) of NIR Spectra

Based on the PLS results, PCA was performed on the second derivative spectra in the 1200–2400 nm range, which contains absorption features of both glucose and malic acid. The third principal component score showed a nonlinear relationship with the glucose-to-malic acid ratio, exhibiting an exponential decrease as the ratio increased (Fig. 1).

Table 1. Results of PLS regression analysis of the mixed standard solutions.

Wavelength range (nm)	Glucose (%)		Malic acid (%)	
	R^2cv	RMSECV	R^2cv	RMSECV
400-2400	0.85	0.77	0.79	0.69
400-1000	0.02	1.49	0.20	1.42
600-1200	0.18	1.22	0.36	1.22
800-1400	0.43	0.99	0.47	1.14
1000-1600	0.86	0.76	0.62	0.91
1200-1800	0.70	0.72	0.90	0.49
1400-2000	0.70	0.71	0.78	0.69
1600-2200	0.74	0.66	0.78	0.69
1800-2400	0.97	0.28	0.83	0.61

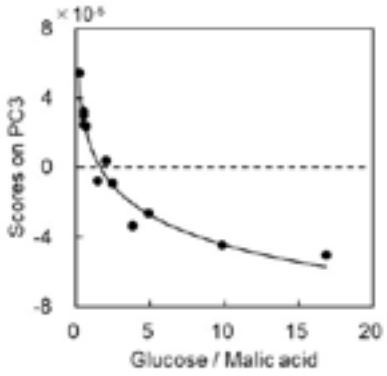


Figure 1. Scatter plot of scores on PC3 vs. the ratio of glucose to malic acid.

Conclusion

The PLS regression results suggest that direct quantification of malic acid in the tested mixtures using NIRS is challenging. However, in CAM plants, glucose and malic acid are interconverted as metabolic substrates, and their ratio fluctuates diurnally. The findings indicate that NIR spectra may capture these dynamic changes in the glucose-to-malic acid ratio. This study used standard solutions as a preliminary investigation, and future research should focus on evaluating the practicality of NIRS with actual plant samples.

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Keywords
crassulacean acid metabolism,photosynthetic metabolites,PLS regression analysis,principal component analysis,standard solutions

P01.01.12

HYPERSPECTRAL PHENOTYPING AS A TOOL FOR ASYMPTOMATIC FLAX DISEASE DETECTION

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Early detection of plant diseases is a key issue in agriculture and plant breeding. In the actual context of climate change and global warming, plant susceptibility to some pathogens may increase [1] leading to economic losses. Verticillium wilt of flax is a fungal disease caused by Verticillium dahliae fungus, whose occurrence in flax growing areas has notably increased over the last decade. Most of the time, flax Verticillium infection remains silent until the retting stage [2], when fibres are damaged by the pathogen and their quality is severely reduced.

This work presents the monitoring of disease progress using an automated high-throughput phenotyping system including VNIR (350 to 900 nm) and SWIR (900 to 1700 nm) hyperspectral cameras. Vegetation indices calculated by VNIR could help to distinguish Verticillium infected plants from non-infected plants at late infection stages, not earlier than visual phenotyping. Meanwhile, spectral scores obtained by Principal Component Analysis (PCA) allowed to clearly separate infected from healthy plants early in the infection. This data treatment could also help to discriminate between different levels of sensitivity of flax to Verticillium wilt disease. This study lays the foundation for the development of a screening tool to help breeders identify flax varieties according to their susceptibility to Verticillium dahliae.

Keywords
Disease detection,Flax,Hyperspectral Phenotyping,VNIR,SWIR

P01.01.13

USING OF NIR SPECTROSCOPY IN AGRICULTURAL LABORATORY

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The aim of this work was to study a possibility of using near infrared (NIR) spectroscopy in agricultural laboratory. The NIR spectroscopy is an excellent tool for screening of parameters that are usually performed by more demanding laboratory methods of wet chemistry. Overview of the methods used and qualitative parameters reached are described.

Method of NIR spectroscopy in NRL UKZUZ laboratories is preferably used for two main fields of interest in analysis of agricultural samples. The method is widely used for determination of selected nutrient parameters in main field crops (cereals, legumes and oilseed) and also parameters describing soil organic matter (SOM).

The NIR spectra were recorded on a Nicolet Antaris II (Thermo Fisher Scientific, U.S.A.). The reflectance spectra were measured from 10 000 to 4 000 cm-1, with resolution 2 cm-1. The ground samples were measured in cells with 3 cm diameter, the unground samples were measured in cells approx. 12 cm diameter. The spectra of the samples were scanned in 120 scans under continuous sample rotation. The spectra were processed using TQ Analyst 8 (Thermo Electron Corp., USA) instrument software.

The first part of the study was aimed for developing of calibration models and analysis of parameters describing soil organic matter (SOM) and determination of parameters such as organic carbon (Cox), total Carbon (TOC), total nitrogen (Ntot), glomalin and Q4/Q6 quotient in soils. The samples originated from the Czech Soil Monitoring System and System of Agricultural Soil Testing. [1,2]

The next part of experiments were focused on the development of calibration models for samples covering the field of variety testing. The ability of prediction for determination of nutrient composition (protein, fat, fibre, starch, ash etc) was tested. The samples covered mainly cereals and legumes (wheat, barley,rye, triticale, oats, corn, peas, bean etc). The attention was also paid to verification of calibration models for some rheological properties determination in wheat flour. The calibration models for parameters including oil of oilseed (rape, sunflower, soya, poppy seeds, flax, mustard) were also successfully developed. NRL UKZUZ laboratories provide analysis of variety testing samples originated from division of operating and testing representing different parts of the Czech Republic.

The calibration models for each parameter were developed succesfully by using Partial Least Squares (PLS) algorithm. The calibration models were verified by cross-validation (CV) and also using external validation (prediction ability was verified by measurement of group of unknown samples which were not part of the calibration data set). The capability of the calibration models to predict component concentrations based on spectral data was expressed using Student test t by reaching an agreement between reference laboratory values and values predicted from proposed NIR calibration equations. [3]

The accuracy and precision of the calibration was expressed by correlation coefficient (R) and root mean square error of cross validation (RMSECV). Good correlation results were achieved for most parameters. The correlation coefficient (R) for the most parameters of interest was in the range between 0,82-0,98.

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Keywords: NIR, variety testing, soils, analysis, calibration model

ACKNOLEDGEMENTS: National Reference Laboratory UKZUZ Brno

Keywords
NIR,variety testing,soil,calibration

P01.01.14

MULTIPLE INSTRUMENT CALIBRATIONS FOR CHLOROPHYLL CONTENT PREDICTION OF CANOLA SEEDS USING A 3 FILTER NIR SPECTROMETER

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Eight Instalab 700 (Dickey-john) instruments, used for moisture prediction, were equipped with two filters in the visible spectrum (674 and 696 nm) and one in the infrared (2100 nm) to assess chlorophyll content in canola seeds. For both wet-chemistry and Near-Infrared (NIR) analyses, 30 g of canola seeds were freshly ground daily. All samples underwent triplicate analysis using both methods. Wet-chemistry results for each sample were considered acceptable when the coefficient of variation was below 10%. Two sample sets collected during the 2023-24 shipping season, comprising both canola exports and individual harvest survey samples, were used for this project. The calibration set consisted of 134 samples with chlorophyll content ranging from 1.5 to 66.4 mg/kg (average 15.9 mg/kg, median 12.1 mg/kg), whereas the independent verification set included 66 samples, with chlorophyll levels between 2.2 and 43.6 mg/kg (average 15.6 mg/kg, median 12.1 mg/kg). Spectrometer absorbance data of the ground canola seeds were recorded at three different times during the day for each instrument and readings were averaged to provide one set of results per sample and instrument. The averaged spectrometer readings from the calibration set were used to develop multiple instrument models. A multiple linear regression (MLR) was used to determine the equation for the model using the calibration set. Statistical analyses were performed, comparing the reference data with the NIR predictions for each instrument using the independent verification set. The results for the eight instruments showed promising consistency with a standard error of prediction ranged from 1.67 to 1.71 mg/kg, a bias ranging from -0.95 to 0.53 mg/kg and R-square values ranging from 0.97405 to 0.97539. These results suggested that developing a common calibration for multiple instruments was successful, indicating that these modified Dickey-John instruments could effectively predict chlorophyll content in ground canola seeds.

ACKNOLEDGEMENTS: Nicole Pogorzelec, Ruth Towes

Keywords
chlorophyll,visible-NIR spectrometry,multiple instruments

P01.01.15

AUTHENTICATION OF LAMB CARCASSES PRODUCED ON SALINE AREAS BY NEAR-INFRARED SPECTROMETRY

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Introduction

The spontaneous halophyte flora represents a potential source of fodder for sheep, and may exert a beneficial influence on the meat quality of lambs produced in saline environments within the Mediterranean basin [1]. The authentication of carcasses and meat produced on saline area would be of interest, in case in labelling. This requires the development of tools that can be used quickly and cheaply in practice. Near-infrared (NIR) spectrometry could be used for this purpose. Consequently, the aim of this study was to evaluate whether NIR spectrometry on muscle and fat could be employed to differentiate the carcasses produced on areas rich in halophytes from those produced in sheepfolds.

Materials and Methods

Two trials were carried out in 2023. The first study was conducted in the Kerkennah archipelago, (Tunisia). 20 lambs were reared on saline rangeland until weaning. They were then allotted in two groups. Ten lambs were kept on the saline area during the finishing periods (3 months). The remaining 10 lambs were raised in sheep pens during the finishing period, on a wheat straw diet. During this period, all the animals were supplemented with 500 grams of a mixture of barley and wheat bran (50/50).

In the second experiment, conducted in Burdur Lake, (Turkiye), 20 lambs belonging to two flocks (10 lambs from each flock) were used. During the finishing period (3 months), the lambs of the first flock, grazed a pasture devoid of halophytic vegetation. In contrast, the animals of the second flock were permitted to graze a pasture comprising non-halophytic plants in this period.

At the end of trials, lambs were slaughtered. After a chilling period of 24 hours, samples of Longissimus Lumborum muscle and perirenal fat were obtained and stored in aluminium foil at a temperature of -20°C until the analysis of spectra. After thawing, the reflectance spectra of the fat and muscle samples were measured at 6 nm intervals between 935 and 1665 nm using a Flame NIR instrument. Spectral data underwent a second derivative treatment according to the methodology of [2]. The pre-treated spectres were then classified according to their fattening method (Halophyte pasture vs control) using the RMS X-residuals methodology proposed by [3].

Results and discussion

Table 1 shows the discrimination results obtained in this study. In both experiments the discrimination success rate was 100%.

Tissue	N	Well classified samples	
		N	%
Tunisia			
Longissimus Lumborum	20	20	100
Perirenal fat	20	20	100
Turkey			
Longissimus Lumborum	20	20	100
Perirenal fat	20	20	100
N=Number of samples			

Table 1: Discrimination of muscle and perirenal fat samples from rangeland halophyte systems vs. other systems by NIR spectrometry

Conclusion

These findings indicate that it is feasible to differentiate between the rangeland-halophyte system and other systems through the utilisation of portable infrared spectra in the segment of 900-2700 nm. These preliminary results deserve to be confirmed by increasing the number of samples used.

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ACKNOLEDGEMENTS: This study was carried out as part of the HaloSheep project, with the support of the PRIMA programme of the European Commission and with the financial support of the Agence Nationale de la Recherche (ANR) of France (ANR-22-PRIM-0008), the Ministère de l'Enseignement Supérieur et de la Recherche Scientifique (MESRS) of Tunisia and the Scientific & Technological Research Council of Turkey (TUBITAK), (Project No. 122N057).

Keywords
NIR,sheep,meat,halophytes,traceability

P01.01.15b

APPLICATION OF NIR SPECTROSCOPY FOR DETECTING ADULTERATION IN INSECT FLOURS

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Insect flours represent an alternative to traditional protein sources due to their high content of proteins, essential amino acids, and lipids, combined with a reduced environmental impact in terms of water consumption, greenhouse gas emissions, and land use [1]. However, the growing commercial interest in these food products, which are characterized by a high market value, increases the risk of adulteration with cheaper raw materials, such as cereal flours. Therefore, it is essential to develop rapid, reliable, and non-destructive analytical methods for quality control and authentication of insect flours intended for human consumption. In this study, the applicability of near-infrared (NIR) spectroscopy was evaluated as an analytical technique for the identification and quantification of adulteration in insect flours [2]. Samples of cricket and buffalo worm flours were mixed with different concentrations of wholegrain cereal flours, simulating increasing levels of adulteration. NIR spectra were collected in reflectance mode using a Nicolet 6700 FT-NIR spectrometer (Thermo Scientific). Spectral data analysis was conducted using chemometric approaches to develop predictive models for adulteration detection. Partial Least Squares (PLS) regression [3] was implemented to construct models capable of estimating the percentage of adulteration in the analyzed samples. The results indicate that NIR spectroscopy, combined with chemometric methods, is a promising technique for detecting adulterated samples. The application of this methodology could serve as an effective screening tool for quality control in the food industry, contributing to consumer protection and the prevention of food fraud in the emerging insect flour market.

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Keywords
Insect flour,adulteration,food authenticity,chemometric analysis,NIR spectroscopy

AQUAPHOTOMICS

P01.02.16

NIR-AQUAPHOTOMICS ANALYSIS OF SKIN USING INDICATORS OF BARRIER FUNCTION

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Objective

The skin is a cosmetically important organ and serves as an important barrier function, a boundary between the inside and outside of the body [1]. Trans-epidermal water transpiration (TEWL) and skin hydration are used as indicators of this barrier function [1]. This study used near infrared (NIR) spectroscopy and Aquaphotomics [2,3] (NIR-Aquaphotomics) to compare the skin that have different barrier function strength.

Methods

Forty-one female subjects in their 20s to 50s who gave consent were asked to wash their face using a jell cleanser first, and a foaming cleanse. NIR spectra of both cheeks at 5, 10, and 20 minutes after the washing have been measured using a MicroNIR ES 1700 (VIAVI). At each elapsed time, five consecutive NIR spectra were measured without moving the device from the measurement point on the cheek. Just before the NIR spectra measurement at 20 minutes, TEWL and skin hydration were measured at the same locations as the spectra measurement. Thermo Recorder TR-73U (T and D) was used to measure the experimental environment. The measured NIR spectra were analyzed using Pirouette 4.5 (Infometrix).

Results

Subjects were divided into four groups compared to the respective median of TEWL and skin hydration (Table 1). The Soft Independent Modeling of Class Analogy (SIMCA) discriminated the four groups at 5, 10, and 20 minutes after washing, with 92.41%, 97.37%, and 97.37% accuracy, respectively.

Comparison of the four groups at 5, 10, and 20 minutes after washing with Aquagram (Figure 1), which shows the absorbance spectral pattern of water [3], revealed that water solvation shells, trapped water, and proton hydrates (1366, 1373, 1385, 1397, and 1397, 1565 nm) [2,4] are abundant in Group 3 and 4, while proton hydrates, free molecules, hydration water, water solvation shell, and water molecules with two to four hydrogen bonds (1342, 1410, 1422, 1435, 1441, 1453, 1466, 1472, 1497 nm)[2] were abundant in Group 2. The spectral pattern of Group 1 was similar to that of Group 2 after 20 minutes of washing, but showed a different pattern from Group 2 after 5 minutes of washing.

Conclusion

SIMCA results and Aquagrams showed that when subjects were divided into 4 groups according to TEWL and skin hydration, the spectral patterns of the four groups were already different after 5 minutes of face wash. This indicates that while conventional equipment requires to wait 20 minutes or more after cleansing to measure TEWL and skin hydration, Aquaphotomics analysis of NIR spectra after 5 minutes of cleansing offers the possibility of fast barrier function evaluation.

In the future, we plan to discern the relationship between water structures and water functionalities of the skin, and further explore the possibility of skin evaluation by Aquaphotomics analysis of NIR spectra using other indicators that can be used to evaluate skin, such as elasticity.

Table 1. The Groups 1 to 4, classified based on the median values of the subjects' TEWL and stratum corneum moisture content (hydration).

	hydration	TEWL
Group 1	high	low
Group 2	high	high
Group 3	low	low
Group 4	low	high

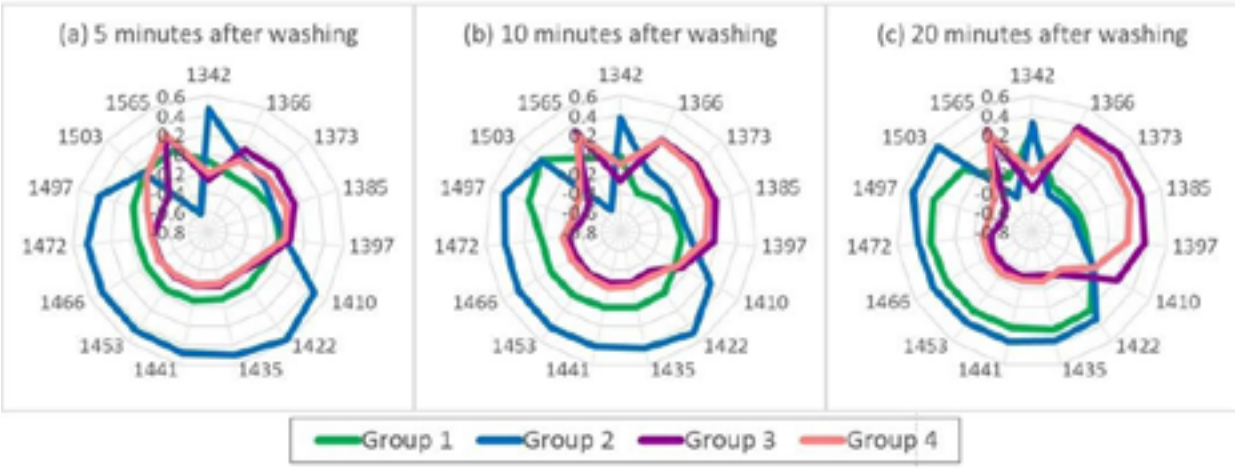


Fig 1. Aquagram showing the differences between groups classified by TEWL and skin hydration. (a), (b), and (c) at 5, 10, and 20 minutes after face washing, respectively.

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ACKNOLEDGEMENTS: We would like to express our gratitude to everyone who contributed to this research.

Keywords

Aquaphotomics,skin,Aquagram,near-infrared spectroscopy

P01.02.17

NEAR-INFRARED SPECTROSCOPY AND AQUAPHOTOMICS FOR INVESTIGATION OF WATER AND PROTEIN STRESS IN TOMATO PLANTS

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Tomato (Solanum lycopersicum) is one of the most widely cultivated vegetable crops globally. The productivity of plants is considerably affected by various abiotic stresses. Plant stress detection is critical to increase crop yield. Some methods for quantitative estimation of stress effects, such as metabolomics methods, provide very accurate data, but they are sampling destructively and very slowly, which prevents dynamical studies. Recently, spectroscopy in the visible and near-infrared region was used to investigate the effect of abiotic stress. Aquaphotomics is a novel scientific approach based on the near-infrared spectra of water in different materials, mainly biological samples, and analysis of changes in water spectra caused by the influence of various factors.

The main objective of this study was to evaluate the possibilities of near-infrared spectroscopy (NIRS) and aquaphotomics for investigating water and protein stress in tomato plants.

The two sorts of tomato plants - Red Bounty and Mirsini, were growing up in a polyethylene greenhouse on training experimental fields of Trakia University. Plants were divided into three groups. The plants from the first group were grown under optimal agrotechnical conditions, the second group had one-third reduced nitrogen fertilization, and the third group had one-third reduced watering. Spectral measurements of leaves from each group were made on-site at the flowering stage of tomato plants. Spectral acquisition in the 900–1650 nm was made by a portable, handheld AlbaNIR spectrometer in the diffuse reflectance mode. A Pirouette 4.5 (Infometrix, Inc., Bothell, WA, USA) was used for spectral data processing. Soft Independent Modeling of Class Analogy (SIMCA) was used for classification. The F1 score and total accuracy were used as the classification model performance indicators. Additionally, so-called aquagrams were calculated.

Results show a difference between the spectra of leaves from control plants and those grown with reduced nitrogen fertilization or watering for both investigated tomato sorts. The differences were bigger for Red Bounty plants, compared to Mirsini plants. Aquagrams clearly show the differences in water structures in the three groups of plants. The performance of developed SIMCA models for discriminating plants according to growing conditions was very high. The F1 score of the model for Red Bounty tomato plants varied from 96.2 to 98.8%, and the total accuracy was 97.09%. The respective values for Mirsini plants were: F1 from 71.17 to 90.06% and a total accuracy of 79.03%.

The results show successful differentiation of the control and stressed plants. The aquagrams and spectra of the investigated two tomato varieties also show the differences related to the tolerance of tomato plants to nitrogen and water stress.

ACKNOLEDGEMENTS: ACKNOWLEDGEMENTS: The authors gratefully acknowledge receiving funding from the National Science Fund of Bulgaria, project "Aquaphotomics approach for investigation of stress-induced changes of water molecular structure in green species" (Contract No. KP-06-N51/3).

Keywords
Tomato (Solanum lycopersicum),water stress,nitrogen stress,aquagrams,SIMCA

P01.02.18

CHEMOMETRIC METHODS FOR UNDERSTANDING WATER STRUCTURES BY NEAR-INFRARED SPECTRUM

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Water structure takes an important role in chemical and biological systems, because most of the molecules may interact with water in the system and the interaction may bring a change in structure and function of the molecules. Due to the flexibility and the complexity of the hydrogen bonding, the structure of water changes adaptively under perturbations such as the variation of composition, temperature, pH and concentration. Therefore, the change of water structures can be a reflection of the composition and properties of the analyzing system. Near-infrared (NIR) spectroscopy has been proven to be a powerful technique in analyzing the structure of water due to its sensitive response to OH and extensively employed in the studies of water structures [1,2]. The spectral features of water structures can be distinguished by analyzing the NIR spectra of water, and the spectral changes of water under perturbations can be used to reveal the structural changes and the interactions in aqueous systems. In these works, chemometric methods were developed to enhance the resolution or to extract the spectral features from the NIR spectra of water. Besides the commonly-used continuous wavelet transform (CWT), wavelet packet transform (WPT) was also employed for extracting high resolution spectral component from the NIR spectrum of water [3]. With the help of these method, the spectral information of different hydrogen-bonded water can be obtained and used as a probe for chemical and bio-processes. The inhibition mechanism of polyproline (PPro) and poly(vinyl alcohol) (PVA) to the ice growth was analyzed [4,5]. The stable existence of interfacial water is critical for the antifreeze activity of PPro and PVA inhibits ice growth by directly binding to ice. Temperature-induced self-assembly of copolymer poly(N,N-dimethylacrylamide)-poly(diacetone acrylamide) (PDMAA30-PDAAM60)2 in aqueous solution was also analyzed [6]. The spectral information of hydrated water surrounding the hydrophilic PDMAA and hydrophobic PDAAM blocks was obtained, revealing the roles of water in morphological transition of the copolymer from spherical to worm-like micelles.

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ACKNOLEDGEMENTS: Acknowledgement: This work was supported by the National Natural Science Foundation of China (Nos. 22174075 & 22374082).

Keywords
chemometrics,aquaphotomics,wavelet transform,water structure

P01.02.19

MOLECULAR DYNAMICS SIMULATION-AIDED SPECTRAL ANALYSIS OF WATER STRUCTURE DURING ICING

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Water freezing occurs widely in nature and plays pivotal roles in life activities, such as climate, biological survival, and diverse industrial applications. The structure of water (hydrogen bonding) during icing changes significantly in phase transition, often involving multiple metastable intermediate states. However, the dynamic change in water structure and the intermediate states during ice nucleation and crystal growth remain unclear [1,2]. In this work, we utilized near-infrared (NIR) spectroscopy, which is highly sensitive to water, and advanced chemometric methods, to analyze the dynamic behavior of hydrogen-bonded structures throughout the freezing process [3]. Encouragingly, we have successfully resolved the spectral features of water structures and uncovered the subtle structural changes that are crucial to the freezing process but are difficult to capture. With the help of molecular dynamics (MD) simulations, the spectral features corresponding to the short-lived ordered structures preceding ice nucleation and the intermediate states during crystal growth were further identified. Analysis of the MD trajectories indicates that the intermediate states during ice growth are a class of interfacial molecules located between the ice and liquid phases, involving cubic-interfacial and hexagonal-interfacial molecules, with different types of hydrogen-bonding connections [1,4]. More importantly, simulation evidence for conversion between hexagonal and cubic interfacial molecules at the ice-water interface was found, which helped identify the two peaks of the water structures observed in the NIR spectrum. These interfacial intermediate structures bridge the ice and liquid phases and drive the growth of ice crystals. These findings may help in understanding the mystical properties of water and also illustrate the important role of MD simulations in structural analysis using NIR spectroscopy.

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ACKNOLEDGEMENTS: Acknowledgement: This work was supported by the National Natural Science Foundation of China (Nos. 22174075, 22374082 and 22373051).

Keywords
Molecular dynamics simulation,spectral analysis,chemometrics,water structure

P01.02.19b

NIR AQUAPHOTOMICS FOR MONITORING SOIL AND PLANT RESPONSES TO ORGANIC FERTILIZERS

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Soil acts as a fundamental medium for plant growth, serving as a reservoir of water and habitat of microorganisms. Its characteristics are shaped by various factors such as organic fertilizers which change the soil structure, the nutrients present, microbial activity, and soil permeability. Organic fertilizers, unlike industrial fertilizers, are more environment-friendly, as they are sourced from the plants and animals that have decomposed. Examining the impact of organic fertilizers by conventional methods often require extensive time, expertise and resources. Due to these limitations, a fast and cheap method is required for real-time in-vivo monitoring of agricultural systems. For this purpose, this research used NIRS aquaphotomics to investigate the influence of organic fertilizers on soil and plant dynamics. Spectral measurements by MicroNIR OnSite-W (Viavi Solutions, USA) across various time points were taken on several beds with varying treatments (with pig manure (PM), with pig manure and liquid fertilizer (PM+LF), and without fertilizers (Control)), and their major macronutrients, such as Nitrogen (NO??-N), Phosphorous (PO?^{3}?) and Potassium (K), were assessed. Reports indicated that the application of organic fertilizers, especially PM+LF, led to increase water retention, nutrient concentration, soil microbial activity, and overall soil health. Aquagrams illustrated fertilized soil with higher absorption of strongly-bounded water (1490-1583 nm) right after organic fertilizers application, while at a later stage, PM+LF treatment was best able to improve microbial activity (1300-1400 nm) [1]. Spinach and mustard were cultivated on the treated soil, and their spectral data was acquired by the same spectrometer. Leaf aquagrams of the control group were characterized with water absorbance bands in the range of 1364-1422 nm, related to dehydration and senescence [2]. The absence of 1440 nm within the control group corresponds to the decrease in bound water due to cellulose and cytoplasmic shrinkage, indicating structural degradation. On the other hand, organic fertilizers were able to improve the water retention properties and structural stability of the plants at early vegetative stage, while reducing plant stress, visible at 1515-1583 nm. At later stages, fertilized plants exhibited a decrease in less hydrogen-bonding (1360-1391 nm), which implied reduced dehydration. In addition, regression analysis results showed sufficient accuracy (Rval = 0.59 - 0.84) for prediction of moisture content, electrical conductivity, nitrogen, phosphorus, and potassium, demonstrating the potential of this approach for real-time nutrient monitoring and soil quality assessment. This research underscores the promising ability of NIR aquaphotomics as a fast, cheap and non-invasive approach to assess soil and plant health in agroecosystems.

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Keywords
Non-invasive,Precision agriculture,Soil fertility,Soil-plant interactions,Spectral analysis

CHEMOMETRICS

P01.03.20

MACHINE LEARNING INTERPRETABILITY METHODS APPLIED TO CALIBRATION MODELS DEVELOPED ON NEAR INFRARED SPECTROSCOPIC DATA

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In the past decades, Machine Learning (ML) models have become more and more complex, leading to improvements in their predictive performance. However, these models can often be described as "black boxes", in the sense that it is very difficult to explain how results are obtained by a model from the input data. As complex Machine Learning models are increasingly used to make decisions, for instance in industrial or medical applications, there is a growing need to improve their interpretability in order to have greater confidence in their results [1], providing the so-called Explainable AI (Artificial Intelligence).

This study is focused on the interpretability of Machine Learning models after model calibration on near-infrared spectroscopic data, a.k.a. the "post-hoc explanation" of ML models.

Several regression models such as Support Vector Machines (SVM), Artificial Neural Networks (ANN) and Extreme Gradient Boosting (XGBoost), have been trained and compared to the classical PLS calibration models.

The study is applied to the freely available Benchmark dataset consisting of Near Infrared spectra of minced meat samples recorded with the FOSS Tecator Infratec Food and Feed Analyzer in the 850-1050nm wavelength range [2].

Various ML model interpretability algorithms currently applied to non-spectroscopic data have been tested and compared: Local Interpretable Model-agnostic Explanations [3], Shapley Additive Explanations [4] and pseudo-samples prediction [5]. They have also been compared to the regression coefficients of an intrinsically interpretable Partial Least Squares model. While the applicability to spectroscopic data of pseudo-samples prediction has been demonstrated [5], Local Interpretable Model-agnostic Explanations and Shapley Additive Explanations are theoretically better suited to uncorrelated variables [6].

The results indicate that the outputs of the interpretability methods tested appear to be in good agreement with the Partial Least Squares regression coefficients. Thus, they allow the identification of wavelengths ranges used by "black box" Machine Learning models. Moreover, they could be used as indicators of the risk of overfitting for complex models developed on spectroscopic data.

In conclusion, this study shows the potential for applying different Machine Learning model interpretability methods to Near Infrared spectroscopic data. Further work on different NIR datasets will be conducted to confirm these results.

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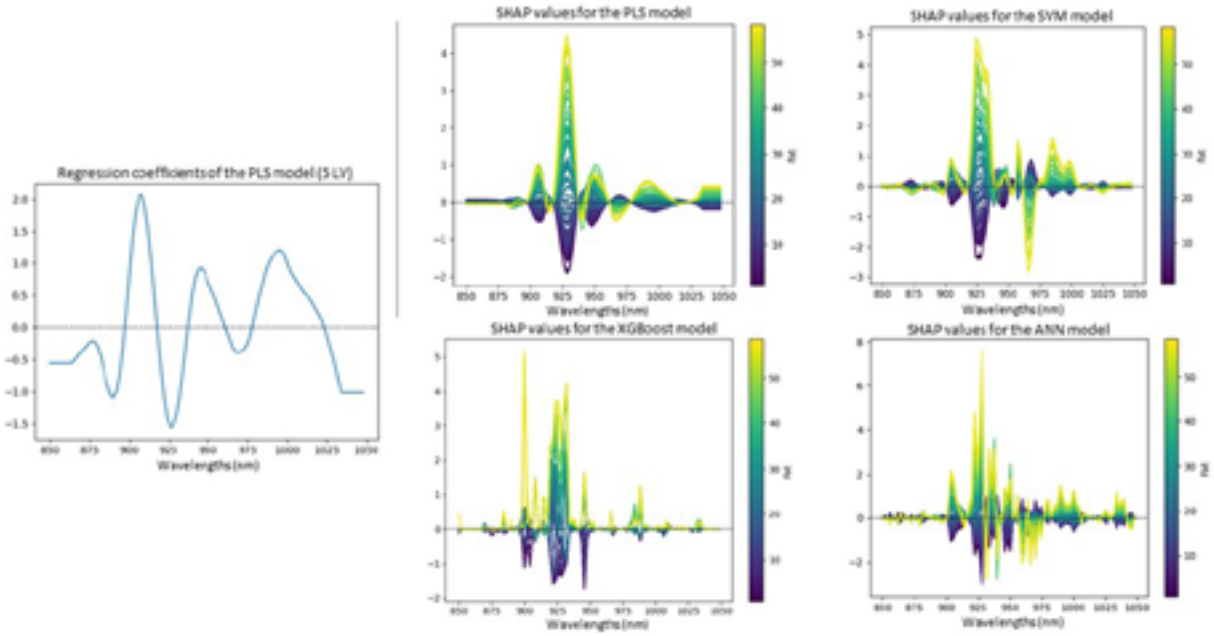
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Figure 1: Example of results obtained on the calibration spectra with the Shapley Additive Explanations method for different Machine Learning models



ACKNOLEDGEMENTS: Ondalys wishes to acknowledge the Occitanie Region for the funding of the R&D projects on Explainable AI/ML.

Keywords
Machine Learning,Explainable ML,Interpretability,SVM,ANN

P01.03.21

POTENTIAL OF PORTABLE AND BENCHTOP NIR SPECTROSCOPY FOR THE QUANTIFICATION OF CANNABINOIDS IN MEDICINAL CANNABIS OILS

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Medicinal cannabis products, such as oils, represent a promising therapeutic strategy for managing various diseases, including refractory epilepsy, which predominantly affects children. Research has demonstrated that cannabidiol (CBD), one of the main cannabinoids in these products, can significantly reduce seizure episodes, thereby improving the quality of life for patients [1]. However, the high cost of legalized cannabis products in Brazil has led families and non-profit associations to produce artisanal cannabis oils, which are not regulated by the national health authority. Ensuring the quality and canabinoid content of these oils is essential to guarantee their therapeutic efficacy.

This study explores the use of portable (microNIRTM 1700, Viavi Solution, USA) and benchtop (Spectrum Frontier, Perkin Elmer) near-infrared spectroscopy for the quantification of cannabinoids in medicinal cannabis oils from diverse matrices. As a reference method, cannabinoid quantification was performed using HPLC-DAD with a chromatographic method adapted from Carvalho et al. [2]. The modified chromatographic method was also validated to ensure its reliability for this application. Samples analyzed included donations from non-profit associations in Brazil, as well as laboratory-prepared oils with varying extract percentages in different matrices. NIR spectroscopy (using both microNIR and benchtop instruments) was employed to determine the content of major cannabinoids, such as cannabidiol (CBD) and tetrahydrocannabinol (THC). Calibration models were constructed using laboratory-prepared samples, with 48 samples for calibration and 21 for prediction.

The NIR spectral data of medicinal cannabis oils were preprocessed using the first derivative with a 5-point window preceded by second-order polynomial smoothing and subsequently analyzed using Principal Component Analysis (PCA). The first principal component differentiated the samples based on the type of vehicle or oil employed in their preparation. Considering that artisanal cannabis oils are often prepared using a variety of vehicles, such as olive oil, coconut oil, and sunflower oil, this distinction could potentially be used to identify the vehicle employed in the preparation of cannabis oil. Additionally, further principal components allowed for the discrimination of samples based on the type of extract and the percentage of extract used. Building on these findings, multivariate calibration models were constructed, testing various spectral preprocessing techniques to establish a robust method for the quantification of cannabinoids in oils from different matrices. The calibration models yielded an RMSEP of 0.12% for THC in the calibration range of 0% to 3.23%, and an RMSEP of 0.02% for CBD in the calibration range of 0% to 0.88%. These results highlight the potential of NIR spectroscopy to support regulatory frameworks and improve the quality control of medicinal cannabis products.

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ACKNOLEDGEMENTS: LAC, LTM, CNPq, PPGQ-UFPE, GPETCAM, INCTAA, UNIMORE

Keywords
Medicinal cannabis,NIR,canabinoids,quantification,chemometrics

P01.03.22

HOW IMPORTANT IS THE NUMBER OF CLASSES? IDENTIFICATION OF COFFEE PLANTS USING HYPERSPECTRAL IMAGES

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Last years artificial intelligent, IA, has become a trendy topic, however, machine learning has been widely used. These models allow us to classify our sample. As different models (e.g. PCA, SIMCA, PLS-DA) can be used to do that task, parameters as class number (binary classes or multiclass) could be an important parameter to take into account. The class definition can be a time-consuming task when hyperspectral images are analysed, so reducing it without altering the accuracy can be an effective way to work. We also want to know how the use of a different number of classes affects the performance of the different models. For these reasons the aim of this work is to verify if the number of classes in different types of machine learning models can alter their accuracy. This work can provide a first insight into a sector such as precision agriculture, where the use of these and other sorting models is of major importance. [1] [2]. Evaluation of the influence of the number of classes was carried out in five different classification model: Soft independent modelling by class analogy (SIMCA), Data-Driven SIMCA (DD-SIMCA), Partial Least-Square Discriminant Analysis (PLS-DA), Random Forest (RF) and Support Vector Machine (SVM). We used two different hyperspectral images taken by drone in two different farms of Colombia and in different days. These hyperspectral images provide us a dataset that goes from 400nm to 2500nm (UV-VIS to MIR). With the intention of obtaining the best results, the preprocessing of the data set was optimized for every model, trying different options (e.g. 1st derivative, SNV) and comparing the robustness related to CV. The results obtained from each model was compared using sensitivity, specificity and accuracy in two different situations: two classes and 8 classes. While one of the images was used for the modelling the other was use for the validations of the result obtained from the models. We can therefore see a difference in the results obtained in the different models using different classes. This is why this paper has assessed the extent to which this may compromise the different models.

ACKNOLEDGEMENTS: Thanks to all who have made this report possible and to BlackSquareSAS for providing the hyperspectral imagery.

Keywords
Machine Learning,Hyperspectral Images,Drone imagery,Classification models,Multiclass

P01.03.25

ENHANCING FOOD AUTHENTICITY: CHEMOMETRICS AND NIR SPECTROSCOPY TO DIFFERENTIATE FRESH AND THAWED SEAFOOD

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Food fraud is a significant concern in today society, particularly in the context of global supply chains, with significant implications for food safety, product quality, and consumer trust. This issue affects many food products: fish, meat, vegetable oils, biological and dairy products, bakery goods, alcoholic and non-alcoholic beverages, etc. In the seafood sector [1], a common fraudulent practice involves the sale of previously frozen fish as fresh, with economic and health-related repercussions [2]. Transparency and truthfulness of the products offered to the consumers must be ensured throughout the food supply chain, to avoid practices of adulteration that not only damage the credibility of food brands but also weaken the customer trust towards the food industry and the effectiveness of the governmental programs of food control.

In this context, the present study proposes the use of NIR spectroscopy to develop an advanced and non-destructive analytical method able to identify structural and biochemical changes induced by freezing and subsequent thawing, allowing the identification and differentiation between fresh and thawed seafood products. The analysed species belong to the family of cephalopods, and are octopus, cuttlefish and musky octopuses. Three different instruments were used: a benchtop full range NIR spectrometer – MPA (Bruker), a pocket-size spectrometer – SCiO (Consumer Physics), and a portable one – MicroNIR (VIAVI).

The spectroscopic data were analysed through Chemometrics to firstly extract relevant information, using unsupervised exploratory tools such as Principal Component Analysis (PCA). This first step allowed to investigate the structure of the datasets, identifying interesting grouping trends among the samples linked to their conservation, and their physical state (fresh or thawed). Further, classification models were built to enable reliable and reproducible differentiation between fresh and thawed samples, with the aid of Partial Least Square-Discriminant Analysis (PLS-DA). This approach aims at highlighting the potential of NIR spectroscopy combined with Chemometric analysis as a powerful, non-destructive, and rapid tool for detecting food fraud, ensuring authenticity, and safeguarding the quality of seafood products.

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ACKNOLEDGEMENTS: Esselunga is aknowledged for providing the samples. Istituto zooprofilattico is aknowledged for the support in the analyses.

Keywords
Food authenticity,NIR spectroscopy,Chemometrics

P01.03.26

DEEP LEARNING FOR NIRS DATA, APPLICATION TO AGRONOMICAL AND ENVIRONMENTAL DATA: SOIL AND POST-HARVEST

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With the increasing deployment of NIRS sensors in the laboratory, in the field or greenhouses, spectral databases have increased in size and tend to be more complex and non-linear. To deal with these challenges of chemometrics, deep learning (DL) opens a new research path. However, despite its increasing popularity and massive adoption in other fields such as computer vision and natural language processing, it is still in its infancy for spectral data. The adoption of DL in spectroscopy faces numerous limitations, starting with the availability of open-source codes and architectures. One of the strengths of DL, is its ability to provide generic models, applicable to a diversity of databases thanks to transfer learning mechanisms. In the case of spectral data, it remains to determine how to benefit from the generic features learned with diverse data to tune models for specific applications. In this work, we propose to present standard neural networks architectures, adapted from computer vision for spectroscopic data. The purpose is to highlight the current limitations of DL regarding the state of the art in chemometrics and to determine perspectives to unlock the scientific barriers. From this standpoint, an open-source solution to experiment, deep chemometrics models, is presented. We present a case study on open spectral databases: OSSL [1] and Wheat Kernel [2]. We compared regression and classification results obtained with our basic exploratory DL models, including a transfer learning strategy, with standard approaches in chemometrics, PLS (Partial Least Squares) and LWPLS (Locally weighted PLS). Among the architectures, we tested some developed specifically for chemometrics. In addition to some emblematic approaches of computer vision that we adapted for spectral data, including: CuiNet [3], DarioNet [4], ResNet[5], DeepSpectra [6], ViT [7].

The results obtained show that even large models, such as transformers, can achieve high performances on databases of intermediate size. However, it was observed that these approaches, although considerably more expensive in terms of training time and more complex to optimise due to the large number of parameters, do not currently outperform traditional methods such as PLSR (Regression) or PLSDA (Discriminant Analysis). Transfer learning has not improved performance, probably due to the ultra-specialisation of spectral databases. Unlike computer vision, where large generic databases are widely available, the spectral databases accessible are generally highly specialised. As a result, transferring a model trained on one specialised database to another specialised database does not currently offer satisfactory performance.

This work highlights DL's limitation, namely the lack of integration of specific chemometrics knowledge into DL contributions. To achieve this, the chemometrics community must provide its codes, models, and data to accelerate the development of DL-chemometrics and open up new research trails for physicochemical properties and spectral simulation.

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ACKNOLEDGEMENTS: This work was performed using HPC resources from GENCI–IDRIS (Grant 2025-AD010114820R1)

and grant ANR-23-LCV2-0015

Keywords
Deep Learning,Chemomectrics,calibration transfer,Model optimisation

P01.03.28

TRANSFORMER BASED MODEL FOR NEAR INFRARED SPECTROSCOPY DATA FOR ESTIMATION OF QUIINE CONTENT IN CINCHONA

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Quinine, the most important alkaloid found in cinchona bark, is still relevant in malaria treatment today. Beyond its medicinal use, it is added to beverages as a bittering agent, serves as a catalyst in asymmetric organic synthesis, and functions as a chiral selector in stationary phases [1]. India hosts one of the largest cinchona plantations, covering approximately 2,800 hectares in the Darjeeling hills. However, extensive cross-pollination and seed propagation over the past century have led to a decline in quinine content, reducing market value and export potential. To address this, a large-scale effort is underway to rejuvenate the plantation through vegetative propagation of high-alkaloid trees. This requires assessing alkaloid content in millions of plants, a task impractical through time-consuming and skill requiring conventional methods like UV Vis spectrometry, fluorimetry, polarography, atomic absorption spectroscopy, membrane electrodes and chromatography [2]. Therefore, the development of rapid estimation techniques is crucial for initial selection.

The objective of this study was to develop a cost-effective and field deployable method for predicting quinine content in cinchona using Near-Infrared spectroscopy combined with an advanced technique called Transformers. For the present study, two hundred twenty bark samples from cinchona tree were collected from about 26,000 acres of plantations area varying between an altitude of 1,200 to 6,000 ft above mean sea level of Munpoo, Labdah, Munsang, Sittong, Rungbee, Rongo, Burmeik, Dalgawn division in Darjeeling, West Bengal, India. The NIR spectra were collected using a tungsten halogen lamp , an NIR spectrometer with an InGaAs detector array of 900 nm to1700 nm (Ayudyog Pvt Ltd., India). The model adopts an encoder-decoder architecture, which first utilizes the encoder's self-attention mechanism to capture the spectrum's critical information and then integrates the extracted feature information through the decoder to achieve quantitative prediction [3]. This architecture can handle complex nonlinear relationships and better capture the spectrum's global features. We predicted quinine content in cinchona and obtained RMSE, R^2, and RPD of 0.92, 0.87, and 5.43, respectively.

These metrics confirm that the Transformer-based model provides predictive power for quinine content estimation of cinchona. The multi-head self-attention mechanism present in the Transformer based model allows it to simultaneously focus on multiple parts of the input spectral data, effectively identifying and weighing relevant features from different spectral regions, leading to more accurate predictions in bio-marker profiling. The results of the present study demonstrate the efficacy of using a Transformer-based model for bio-marker profiling with NIRS data. In the future, this technique can be effectively extended for predicting bio-marker quality traits in other plants as well.

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ACKNOLEDGEMENTS: Indian Council of Agricultural Research (ICAR) for the program National Agricultural Science Fund vide Sanction Order No. NASF/PA-10009/2023-24 dated 26.02.2024

Keywords
Transformer model,Encoder-decoder,Cinchona,quinine,NIRS

P01.03.29

APPLICATION OF NEAR-INFRARED SPECTROSCOPY TECHNOLOGY FOR RIPENESS ASSESSMENT IN RASPBERRIES

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In recent years, there has been a remarkable increase in the consumption of raspberries in countries belonging to the European Union, such as Portugal. In particular, the sale of raspberries grew 12.5 times between 2010 and 2023. Therefore, it is necessary to use tools that help to monitor the quality control and traceability of this type of product.

For this study, 8 raspberry varieties were used, and their spectra were taken individually during the period from September to January in order to collect sufficient variability in the parameters to be predicted. The spectra were taken with the Luminar 5030 NIR analyzer (Brimrose Corporation, Baltimore, USA), equipped with an InGaAs detector. The parameters of greatest interest for determining the optimal ripening stage of raspberries were determined analytically: total soluble solids (TSS) (ATAGO PR-101 (Atago Co., Ltd., Tokyo, Japan), acidity (Crinson Compact Tritrador - version S (Crison Instruments, S.A., Barcelona, Spain)) and the ratio between TSS and acidity. The development of the quantitative models was carried out using the partial least squares (PLSR) algorithm (CAMO® Unscrambler X vs 10.5 software), using the absorbance spectrum (Log 1/R, where R is the reflectance). The quantitative models were evaluated based on the cross-validation coefficient of determination (1-VR) and the root mean square error after cross-validation (RMSECV).

The PLSR models developed for the TSS showed values above 0.50 for 1-VR and an error below 0.93. However, the acidity and ratio models both exceeded 1-VR values of 0.80. Similarly, both models expressed low results for the RMSECV (0.44 and 0.90, respectively). Thus, the data indicate that NIR technology in combination with PLSR could be a tool capable of quantifying physicochemical parameters of interest in raspberries.

ACKNOLEDGEMENTS: This study was carried out within the framework of the project "Advanced, innovative and digital technologies for the EUROACE agri-food sector (0100_TID4AGRO_4_E)", co-financed by the European Regional Development Fund (FEDER) through the European Commission's INTERREG VI-A Spain-Portugal Program (POCTEP) 2021-2027. Lucía León thanks the Ministry of Science and Innovation for the grant PTA2021-021189-I

Keywords
PLSR Modeling,Intact fruit,Non-destructive

P01.03.30

A COMPARISON OF TWO MACHINE LEARNING METHODS FOR THE EARLY DETECTION OF ESCA

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Esca is a destructive disease that occurs on the trunk of the vine and is considered to be one of the most destructive grapevine trunk diseases worldwide [1]. The pathogens responsible for this disease end up damaging vital organs of the plant, leading to the death of the vine. In recent decades, optical sensor technologies, particularly hyperspectral imaging (HSI), have emerged as powerful tools for plant disease detection. HSI not only identifies and quantifies diseases but also provides insights into plant-pathogen interactions [2].

The objective of this study was to evaluate the potential of HSI in combination with two machine learning methods, namely Partial Least Squares Discriminant Analysis (PLS-DA) and Random Forest, for the early detection of esca disease symptoms at the leaf level.

To achieve this, 12 vines of the Tempranillo variety were selected. Hyperspectral images were captured in the field using the Specim IQ camera (Oulu, Finland), a line-scanning device that operates within the 400–1000 nm spectral range. The camera produces images with a resolution of 512 × 512 pixels and captures a total of 204 spectral bands within this range.

The chemometric analysis was conducted using the Classification Toolbox v.7 [3] within MATLAB R2021b environment (The MathWorks, Natick, MA, USA). This analysis involved applying two machine learning techniques: PLS-DA and Random Forest. The study included data from the day symptoms became visible (day 4) as well as from the three preceding dates (day 1 to day 3). The selected dates do not necessarily align, as esca symptoms typically appear in a staggered manner across the vineyard rather than on a single uniform date.

The results revealed differences in the mean leaf spectra, particularly in spectral regions associated with chlorophyll. The peaks around 550 nm and beyond 700 nm correspond to areas where chlorophyll exhibits low absorption capacity [4]. Over time, this absorption capacity declines, suggesting a potential early indicator of esca onset, which can be detected using hyperspectral imaging.

Regarding classification models, Random Forest achieved superior classification accuracy across all days, with sensitivity values ranging from 0.94 to 0.99 (indicating the model's ability to correctly classify samples within their respective classes) and specificity values between 0.97 and 0.99 (reflecting its ability to exclude samples from incorrect classes). External validations confirmed the model's strong performance, though some confusion occurred between days 2 and 4. This suggests that differences in leaf development stages may be influencing the classification, warranting further investigation in future studies. Nonetheless, the model achieved 96% accuracy in cross-validation and 89% in external validation.

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ACKNOLEDGEMENTS: This work was financed by the Spanish Ministry of Science and Innovation and from the European Union Next-GenerationEU/PRTR (TED2021-130364B-I00).

Keywords
PLS-DA,Random Forest,Machine Learning,Sensitivity,Specificity

P01.03.31

USE OF NEAR-INFRARED SPECTROSCOPY FOR DISCRIMINATION OF FRESH VS. FROZEN/THAWED TENCH (TINCA TINCA L.) MEAT

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The tench (Tinca tinca, L.) is a freshwater fish traditionally farmed in rural areas of Spain, such as Extremadura, Castilla y León, and Andalucía. The increasing consumer demand for higher-quality products that are convenient, fresh-looking, and capable of preserving their natural flavor and nutrients has led to the need for filleted tench sales formats. Furthermore, the high content of unsaturated fatty acids susceptible to lipid oxidation in filleted tench has encouraged research into preservation in frozen format. The difficult identification of a product that has been frozen and thawed and may have lost quality makes it necessary to use rapid methods to ensure traceability. Therefore, the objective of the present study was to evaluate the capability of near-infrared reflectance spectroscopy (NIRS) technology for the discrimination of fresh and frozen/thawed tench fillets.

A total of 90 tench fillets (longissimus dorsi muscle) were used, of which 30 were sampled 2 h after slaughter (fresh product), while the spectra of the remaining pieces were acquired after frozen storage (in a freezing chamber at -20°C ± 2) for 45 (n = 30) or 90 (n = 30) days and subsequent thawing. Spectra were taken along the fillet zigzagging in the range between 908.10 and 1676.20 nm with the portable equipment: MicroNIRTM 1700 OnSite-W (VIAVI) by direct contact. Instrument manipulation and data recording were carried out using MicroNir Pro v2.2 software (VIAVI Solutions, Inc., San Jose, California, USA). Once the possible existence of spectral anomalies had been studied, a selection was carried out to constitute the calibration and validation sets (70% and 30% of the total samples, respectively). The classification models were obtained using the partial least squares algorithm (PLS-DA) (CAMO® Unscrambler X vs 10.5 software), using the absorbance spectrum (Log 1/R, where R is the reflectance). For the evaluation of the models the cross-validation determination coefficient (1-VR) was used. After validation the sensitivity (SE), specificity (SP) and precision statistics were used to evaluate the model [1].

The PLS-DA model showed a high predictive capacity for the classification of tench fillet meat in the different freezing times (0, 45 and 90 days) with 1-VR values higher than 0.75. After external validation, the classification accuracy of the samples exceeded 92%. The results indicate the capability of NIRS technology as a tool to support traceability control in this product.

Keywords
Tinca Tinca, Near infrared spectroscopy, Freezing storage

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ACKNOLEDGEMENTS: This study has been funded by the European Agricultural Fund for Rural Development (FEADER) (TENCAEX) and the European Regional Development Fund (FEDER). Lucía León thanks the Ministry of Science and Innovation for the grant PTA2021-021189-I.

Keywords
Tinca Tinca,Near infrared spectroscopy,Freezing storage

P01.03.32

MODEL TRANSFER BETWEEN INSTRUMENTS USING THE SCORES DOMAIN

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Büchi (Switzerland)

NIR databases are valuable assets for any company that works with spectroscopy instruments. They form an integral part of a company's intellectual property having been developed over the years. Model developers always aim to leverage the existing databases when adopting a new NIR system. As a result, spectral transfer is crucial when adopting new systems. Spectral transfer techniques convert the database from one domain (primary instrument) to another (secondary instrument). The limitation of spectral transfer techniques like piecewise direct standardization (PDS) and direct standardization (DS) is that they require a significant overlapping region between the primary and secondary instruments. Without this overlap, the primary database becomes unusable.

NeoSpectra TFX (Score transfer algorithm) focuses on transferring chemical information from the PLS scores space instead of the whole spectrum. The main assumption is that the chemical information can be extracted from the spectra, regardless of the instrument's spectral range. This enables leveraging the database of the primary instrument even if there is no overlapping spectral region between the instruments. The algorithm relies on the main PLSR model from the benchtop instrument to compute the PLS scores of the common samples (measured on both the benchtop and the NeoSpectra Scanner). Then, a PCA is performed on the spectra of these common samples from the NeoSpectra Scanner. A linear regression is established between the PLS scores and the PCA scores to create a transformation model that links the two instruments.

For any new sample measured by the NeoSpectra Scanner, the following steps are applied: the PCA transformation is computed, followed by the linear regression model. The resulting projected PLS scores are then used with the benchtop PLSR model to predict the parameter of interest.

The objectives of this study are: i) to analyze the performance of the models using transfer scores from two different benchtop databases; ii) to evaluate the impact of non-overlapping spectral regions on the predictive models.

In this study, two wheat databases with different scanning modes and spectral ranges were used, as shown in Table 1. The study is focused on the protein content.

	Database 1	Database 2
Scanning mode	Reflection	Transmission
Spectral range	570 – 1842 nm	400 – 1090 nm
Number of samples	584	3208
Overlapping spectral range with NeoSpectra scanner (1350 – 2550 nm)	Partial overlapping (1350 – 1842 nm)	No overlapping

Table 1. Benchtop database descriptions

65 samples were measured on both, the benchtop instrument and the NeoSpectra Scanner, serving as the transfer sample set. The protein models generated by NeoSpectra TFX were validated on 30 whole wheat samples using the NeoSpectra scanner, achieving a standard error of prediction (SEP) of 0.51% for the model generated using Database 1 and 0.45% for the model generated using Database 2. The initial PLS regression errors were 0.35% and 0.21% for databases 1 and 2, respectively.

The results show that the limiting factor for transferring models is the initial model error, not the scanning mode or spectral region. Finally, the algorithm enables model transfer between instruments, even when they do not share a common range within the electromagnetic spectrum.

The proposed algorithm offers a method to maintain a single model that can be used across multiple instruments by leveraging a set of common samples. Analytical references are not required, making the process cost-effective and quick to implement. Additionally, there is no need to maintain two separate model lifecycles for the same materials, reducing the operational costs of NIR models compared to traditional spectral transfer methods.

ACKNOLEDGEMENTS: We would like to express our heartfelt gratitude to the Sensor Team–Bassem, Ali, and Ashar–for their invaluable contributions, expertise, and dedication throughout this project. Your relentless pursuit of innovation and excellence has been instrumental in driving success.

Keywords
Model transfer,NIR,Calibration

P01.03.33

COMPARISON OF DIFFERENT CLASSIFICATION STRATEGIES FOR OREGANO AUTHENTICATION THROUGH NIR HYPERSPECTRAL IMAGING

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Dried oregano is frequently adulterated with dried leaves of other plants of lower commercial value, including olive, myrtle, strawberry tree or sumac. In this scenario, Near Infrared Hyperspectral Imaging (NIR-HSI) can be used as a rapid and non-destructive analytical method able to authenticate dried oregano samples, allowing to obtain both spatial and spectral information from spatially heterogeneous samples [1].

The authentication issue under investigation is a typical one-class classification problem with authentic oregano as the target class of interest, and class modelling (CM) classification methods are generally recommended to face these situations. In principle, CM requires only a proper sampling of the target class as they are based on modelling the similarities between samples belonging to it. Then, a new sample is tested against the class model of the target class and accepted or rejected [2]. However, non-target samples are required for a proper validation of the model and to have an idea of its applicability domain. In addition, CM provide poor results when target and not-target classes severely overlap.

As an alternative approach, soft discriminant analysis methods combine the advantages of both CM and discriminant analysis (DA), as they allow to maximise the differences between target and non-target classes even when they overlap, and to potentially detect foreign samples belonging to categories not considered during model calibration. Furthermore, a soft discriminant model can be updated toward the identification of new samples belonging classes not previously considered, making this approach more flexible.

In this study, we considered a total of 49 samples including samples of authentic oregano, pure adulterants (i.e., myrtle leaves, sumac leaves, strawberry tree leaves and olive leaves) and oregano adulterated with different percentages of the considered pure adulterants. The samples were acquired using a line scanning NIR-HSI system working in the 980 – 1660 nm spectral range.

In order to test the effectiveness of both CM and soft DA approaches in differentiating authentic oregano and pure adulterants, as well as evaluating their ability in recognising foreign samples (i.e., adulterants not used for model calibration), different classification models were calculated by iteratively keeping aside one adulterant at a time. Soft Independent Modelling of Class Analogies (SIMCA) algorithm was considered for CM, while Soft Partial Least Squares Discriminant Analysis (Soft PLS-DA) [3] was used as a soft DA method.

Generally higher specificity values were obtained with Soft PLS-DA models, suggesting that soft discriminant approaches represent a valid alternative to CM to face authentication issues.

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Keywords
classification,food authentication,class modeling,soft discriminant analysis,oregano

P01.03.34

NIR SPECTROSCOPY AND GC-IMS COUPLED WITH CHEMOMETRIC DATA FUSION FOR ASSESSING THE AUTHENTICITY OF ITALIAN HONEY

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Ensuring the quality and authenticity of food with advanced analytical methodologies is crucial for consumer confidence, safety and fraud prevention. At the same time, scientific research must minimize environmental impact while maintaining high accuracy and reliability in authenticity methods. Honey is considered a natural high-quality product much appreciated for its nutritional and medicinal properties [1]. Since the price of honey depends mainly on its floral origin, mislabeling of the floral and geographical origin, as well as the addition of cheaper sweeteners or water are the most common economic frauds suffered by this product. Therefore, purity of honey is obviously a priority for producers and regulatory authorities, which need to guarantee the quality of their products. In this study, the attention will be focused on the floral and geographical origin of the investigated samples. In particular, the potential of Near-Infrared (NIR) spectroscopy and Gas Chromatography-Ion Mobility Spectrometry (GC-IMS) combined with chemometric data fusion [2] to discriminate between authentic and non-authentic honey samples will be explored. A dataset of 100 honey samples, including both genuine and adulterated varieties, was analyzed by NIR spectroscopy to assess compositional attributes and by GC-IMS to characterize volatile organic compounds. Advanced chemometric techniques were applied to single and fused datasets to enhance classification accuracy.

This research was supported by University of Modena and Reggio Emilia - Modena Foundation through FAR Mission Oriented 2024 funds.

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Keywords
Honey,Authenticity,Chemometrics,NIR spectroscopy,GC-IMS

P01.03.34b

CALIBRATION TRANSFER TO PORTABLE NIR FOR GREEN COFFEE BREEDING PHENOTYPING

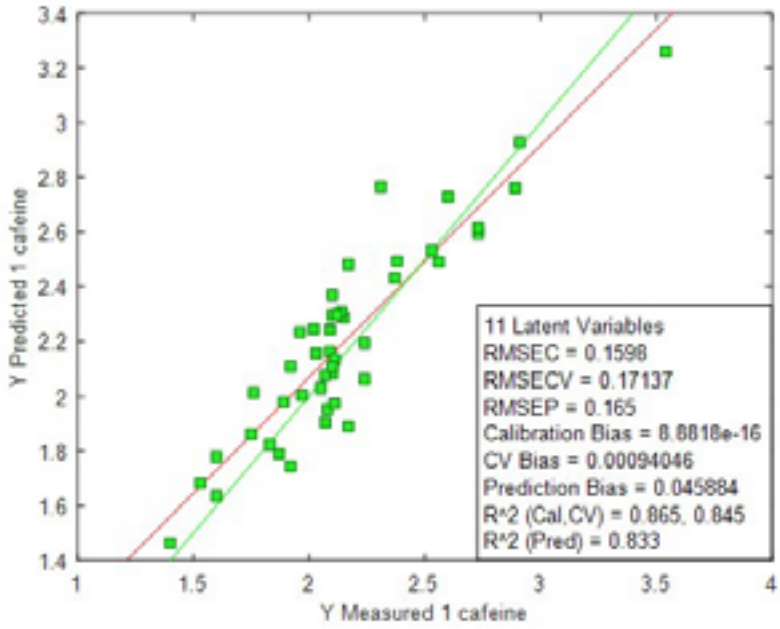
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Coffee breeding aims to develop new varieties focusing on important quality parameters related to coffee function (e.g. caffeine), healthy (e.g. fat & diterpenes), and sensory (e.g. sugars). Currently Near Infrared Spectroscopy (NIR) benchtop models were developed allowing a rapid phenotyping of coffee beans composition over the large population in the field. However, this process still requires time consuming shipping of hundreds of samples from remote breeding field (such as America) to Nestlé France.

The use of a portable NIR device on farms is therefore desired, allowing for in-situ analysis and cloud sharing of data. The main challenge up to date is the inferior performance of portable device compared to benchtop and the need of calibration transfer.

In current study, various machine learning (ML) algorithms were tested on calibration transfer such as Piecewise Direct Standardization (PDS) and direct calibration transfer to Principal Components via Canonical Correlation Analysis (PC-CCA). Based on hundreds of 2024 field samples, some Robusta parameters, like caffeine were transferred from benchtop to portable NIR with acceptable performance. Portable NIR models were deployed to external cloud for on farm testing in future harvest seasons.



CULTURAL HERITAGE & ARCHAEOLOGY

P01.04.35

NON-DESTRUCTIVE DISCRIMINATION OF ANCIENT GLUE FOR HISTORICAL DOCUMENTS AND RICE POWDER IN KOZO PAPERS BY NEAR-INFRARED SPECTROSCOPY

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1. Purpose

Sutras and historical paper-based materials produced in East Asia from the Ancient to Middle Ages often feature a unique adhesive made from soybeans for binding paper seams. In contrast, papers incorporating rice powder into Kozo (paper mulberry) fibers were selectively used in the 16th- and 17th-century Japan for Hosho (letters issued by a noble's steward upon their order). To contribute to historiography, we examined rice powder in Kozo paper and soybean-based adhesives to discriminate these materials using a small near-infrared spectrophotometer (NIRs).

2. Methods

(1) 29 samples of Kozo paper and Kozo paper with rice powder were prepared. Of these, 19 were used for modeling, and 10 for test data. As reflectance varies based on the sample's thickness, reflectivity (R infinity (Ri)), measured by backing many layers of the sample, is used to evaluate paper characteristics per the Kubelka-Munk theory [1]. However, when measuring cultural property, Ri can be calculated from 2 reflectance spectra, R1 and R2, measured with two backing types, Rg1 and Rg2 [2].

Absorption spectra Abs1 and Abs2 were obtained in the measurement range 2000-2450 nm by the diffuse reflection method using small NIRs (NIRONE S2.5, Spectral Engines, Inc.). Five measurements were performed for each sample converted to reflection spectra R1 and R2; Ri was calculated and converted to the Kubelka-Munk function (K/S). The absorption coefficient (K) was obtained from the scattering coefficient (S) with Ri and the basis weight of paper (W). Standard Normal Variate (SNV) was applied to each spectrum for partial least squares discriminant analysis (PLS-DA).

(2) Two types of soy-based or starch-based adhesives were applied to Kozo paper, which was used as a sample for creating models. Three ancient soy-based adhesives used at the seams of historical documents from the 8th to 12th century were used for the tests. Subtraction of the support paper's spectrum was applied to each sample as these spectra were a mixture of the support paper and adhesive absorption. The support paper spectra were multiplied by a constant factor (0.1-0.7, 0.2 intervals) and subtracted from each adhesive's spectrum (Raw) to create data for the models. Similarly, for test data, each support paper's absorption spectrum was multiplied by the factor (0.1-0.5) and subtracted from the seam's spectrum (Raw-0.1Paper, Raw-0.3Paper, Raw-0.5Paper). Two types of sample backing were used to measure Abs1 and Abs2. K/S was reconverted after subtraction of Abs converted from Ri. SNV conversion and PLS-DA were performed after the second derivative using the Savitzky-Golay method.

3. Results

Table 1 shows PLS-DA results of rice powder discrimination at 99% confidence intervals using Abs1, Abs2, K/S, and K. Using K/S and K improved accuracy based on the rice powder discrimination model.

Table 2 shows the PLS-DA results of soy-based adhesive discrimination. The highest accuracy was obtained for Raw-0.5Paper at 93.3%.

Table.1 PLS-DA results for kozo papers with rice powder (Factors: 5) *

Models	Kozo paper		Kozo paper with rice powder		Accuracy (%)
	Correct	Incorrect	Correct	Incorrect	
Abs_1	1	9	25	15	52.0
Abs_2	5	5	26	14	62.0
K/S	8	2	35	5	86.0
K	3	7	32	8	70.0

* Rsults with 99% confidence Level

Table.2 PLS-DA results for ancient soybean glues in historical documents (Factors: 2)

Models	Accuracy (%)*			
	Raw data	Raw -0.1 paper	Raw -0.3 paper	Raw -0.5 paper
Abs_1	76.7	73.3	73.3	62.1
Abs_2	90.0	90.0	90.0	93.3
K/S	86.7	86.7	90.0	93.3

* Rsults with 95% confidence Level

4. Conclusion

Using K/S or K calculated from Ri was proven to reduce the influence of the sample's thickness. For discriminating adhesives, using spectrum subtraction was effective. Obtaining quantitative values of rice powder in Kozo paper and increasing the samples' numbers for the model could improve accuracy and be applied to historical materials.

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ACKNOLEDGEMENTS: This work was supported by JSPS KAKENHI Grant Numbers JP23H00011 and 21K00825. We express our sincere gratitude to the conservation laboratory at the Historiographical Institute, The University of Tokyo, for providing us with rice-powder paper.

Keywords
Historical documents,Kozo paper,Rice powder,Soybean glue,near-infrared spectroscopy

ENVIRONMENT & FORESTRY

P01.05.36

PROXIMAL SENSING FOR NEAR REAL-TIME MONITORING OF ORGANIC CARBON POOLS FOR CLIMART SMART MANAGEMENT

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Soil organic carbon (SOC) is often cited as the single best indicator of an ecosystem's status, and dissolved organic carbon (DOC) in soils is often referred to as the most sensitive indicator to changes in ecosystem function. DOC and SOC data for carbon sequestration potential at depths is essential for emissions trading and carbon budgeting, however, the collection of data is costly and time consuming. Recent advances in technology using infra-red spectroscopy for proximal soil and water sensing offers a more affordable solution, delivering rapid results, but needs the development of mathematical models and procedures for monitoring, reporting and verifying soil carbon data.

PRISM is a co-funded project by the New Zealand and Irish governments which will develop proximal sensing tools for monitoring and assessment of SOC pools and DOC loss in farm landscapes in Ireland and New Zealand. The project will assess technologies such as MIR and NIR spectroscopy for measuring SOC sequestration in soils, so that these technologies can be easily deployed in the field for any user, to (1) identify soils vulnerable to C loss, (2) to determine the soil C sequestration potential of soils (3) to measure on-farm baseline values of soil carbon stocks (4) will allow users to monitor changes in SOC.

Phase 2 of the project has begun and the objective is to develop a national spectral library of Irish soils using SOC data. Soil samples collected from farms in Ireland as part of the Signpost Programme were analysed for their carbon content. Samples comprise of 580 core and 589 profile samples; sampled at the depth of 15 cm, 30 cm, 45 cm and 60 cm. A total of 1160 samples were scanned with 2 near-infrared handheld scanners Neospectra NIR and Innospectra NIR. Afterwhich, 56 samples were selected from the library of 1160 making up 14 profiles i.e. 15-60 cm for carbon fractionation. Carbon fractionation was done using the physical fractionation method. Three fractions were obtained from each sample namely particulate organic matter (POM), mineral-associated organic matter (MAOM) and dissolved organic matter (DOC). All fractions were tested for their carbon concentrations.

Spectra models using NIR data to predict carbon fractions and sequestration potential of soil without the need for physical fractionation and these data will be used in developing a national scale soil spectral libraries for Ireland and New Zealand

ACKNOLEDGEMENTS: Department of Agriculture Food & Marine, Ireland

New Zealand's Ministry for Primary Industries

Teagasc Staff

Keywords
carbon sequestration,soil,spectroscopy,proximal sensing,NIR

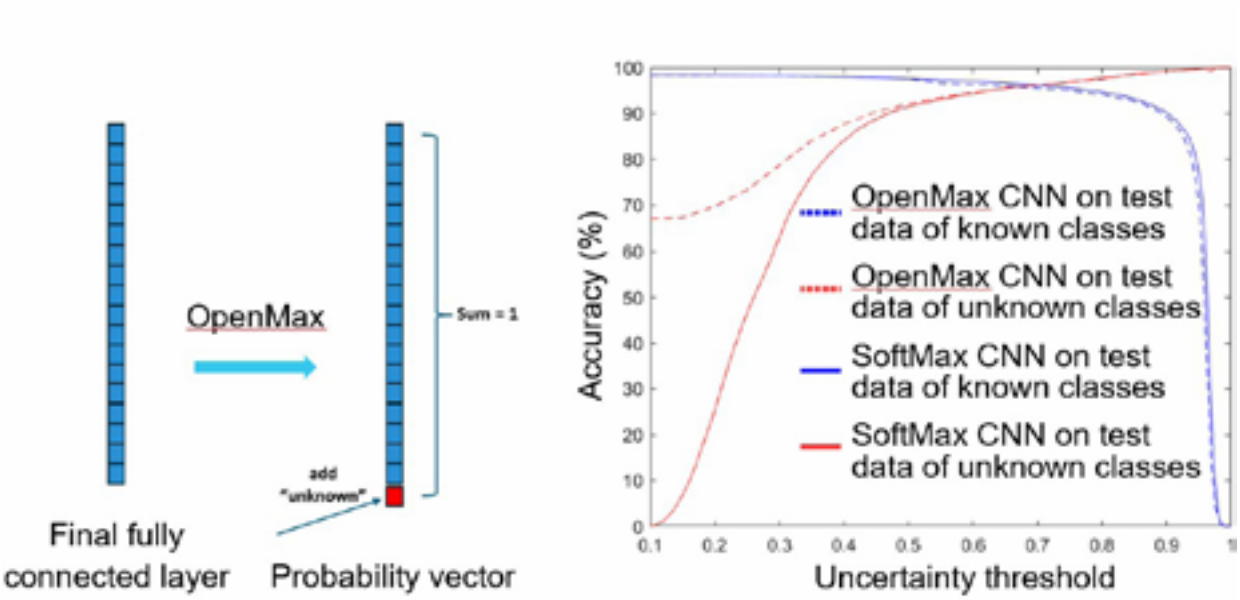
P01.05.37

SPECTRAL IDENTIFICATION OF ENVIRONMENTAL MICROPLASTICS BY OPEN SET CONVOLUTIONAL NEURAL NETWORKS

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Infrared (IR) spectroscopy has long been a cornerstone for identifying and analyzing microplastics (MPs), which are globally pervasive pollutants. In recent years, convolutional neural networks (CNNs) have been increasingly applied to spectral classification tasks in MP research due to their high throughput and remarkable accuracy. Here, we developed a CNN model specifically tailored for IR spectral classification of MPs derived from the environment. Considering the open-world nature of environmental MP detection, we incorporated OpenMax, an open set recognition technique, to address the closed-set limitation of traditional CNN models, where inputs are invariably forced into known classes (i.e., classes included during model training). To enhance the model's generalization, we focused on increasing intra-class variation by incorporating data collected using various IR instruments in our laboratory and publicly available online resources. Additionally, we designed a targeted data augmentation strategy to improve model performance across different spectral ranges, further enhancing intra-class diversity, hence generalization. Results showed that the trained model achieved a classification accuracy of 98.3% on a test set containing over 23,000 spectra from known classes, and 0% on a test set comprising over 9,000 spectra from unknown classes. By integrating OpenMax, the model attained an accuracy of 67.1% on the test set of data of unknown classes (categorizing them into a single 'unknown' class). When applied an uncertainty threshold: between 0 and 0.7, OpenMax demonstrated superior open set recognition performance. At 0.7, the model achieved balanced classification accuracy for both known and unknown classes at 95%. However, in the range of 0.7 to 0.9, OpenMax did not exhibit a significant advantage in identifying unknown classes. Furthermore, the targeted data augmentation strategy improved the classification accuracy for narrow spectral ranges by up to 3.1%. Our study advances the application of CNNs in the spectral identification of MPs from the environment. The ideas proposed here are potentially transferable to Raman spectral identification of MPs from the environment.



ACKNOLEDGEMENTS: Funding for this research was provided by the Science Foundation Ireland (SFI)-Irish Research Council Pathway Programme Proposal ID 21/PATH-S/9290.

Keywords
microplastics,environment,convolutional neural network,open-set recognition,OpenMax

P01.05.38

OPTIMIZING MOISTURE PREDICTION IN KRAFT COOKING DIGESTORS USING PORTABLE NIR SPECTROSCOPY

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Rapid on-site assessment of wood quality can significantly enhance decision-making and operational efficiency in the forestry and wood processing industries. Spectroscopic techniques offer a more consistent and reliable evaluation of wood properties than subjective visual methods. The application of advanced statistical methods improves defect detection, allowing for more accurate differentiation between varying wood conditions. This study focuses on using portable NIR spectroscopy to predict the moisture content in wood chips from Nordic wood species, intending to optimize the continuous cooking process in digesters by analyzing spectral patterns [1]. Additionally, the study compares the performance of the portable NIR model against a traditional benchtop NIR predictive model. We aim to develop a field-applicable predictive model for moisture content, reducing reliance on time-consuming laboratory analyses. To achieve this, we employed two linear calibration methods on NIR data: Partial Least Square Regression (PLSR) and Maximization of Orthogonal Cross-covariance (MOX). While PLSR effectively reduces data dimensionality using a machine-learning technique and performs regression simultaneously [2], MOX focuses on minimizing the number of latent variables in the model, potentially enhancing model simplicity and interpretability. An initial model was developed using benchtop NIR spectra from samples with different moisture levels, achieving an accuracy of R2=0.92. This model was then tested on fresh samples using a portable NIR device to evaluate the feasibility of field application. The results indicated that the portable prediction model performed decently to the benchtop-based model, demonstrating the possibility of creating similar accuracy while providing greater convenience and ease of use in field conditions.

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ACKNOLEDGEMENTS: The authors would like to acknowledge the financial support from the Knowledge Foundation. The authors would like to thank the members of the HÖG project "Homogeneity in High-Kappa Kraft Pulping" (HH-KK) consortium for their participation and in-kind contribution. This consortium consists of the following organizations: Mid Sweden University, Mälardalen University, Billerud Skog & Industri, as well as Mondi Dynäs AB. S. B. Lindström thanks research profile Neopulp, the Knowledge for his financial support.

Keywords
Portable NIR,Benchtop NIR,PLSR,MOX,Wood chips

P01.05.39

IDENTIFICATION OF EUCALYPTUS CLONES BY NEAR-INFRARED SPECTROSCOPY USING PORTABLE EQUIPMENT ON LEAVES

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Klabin S.A., Telêmaco Borba, BRAZIL

Efficient identification of Eucalyptus clone matrices in the field is essential for the success of genetic improvement programs. It enables the proper rescue and transfer of genetic material from the best trees to nurseries, where they will be used for seedling production. This process ensures that the rescued clone is the correct one and allows for the continuation of selecting superior traits, such as productivity and disease resistance, under controlled conditions. Additionally, traceability of the matrices in the field facilitates the optimization of management practices and maximization of genetic gains. For this, it is necessary to have a tool capable of monitoring and tracing clones in commercial and experimental areas. Near-infrared (NIR) spectroscopy has been used for fast, non-destructive, and efficient applications. The potential of NIR needs to be explored for clone identification. Thus, the objective of this study was to test and develop a methodology using portable NIR for identifying Eucalyptus clones through leaf analysis. For the study, 30 leaves from trees of 7 Eucalyptus clones were collected in the field, totaling 210 leaves, near Telêmaco Borba, Paraná, southern Brazil. A portable NIR (MicroNIR) device was used to collect the spectra. NIR spectra were recorded on the surface of wet, dry, and ground leaves. After spectral reading on the wet leaves, they were dried in an oven for 48 hours at 50°C. The spectra were then collected again on the dry leaves, which were subsequently crushed and sieved. Leaves with granulation retained on a 60-mesh sieve were used for a new spectral reading. The spectra were analyzed by principal component analysis (PCA). Models were developed using partial least squares discriminant analysis (PLS-DA). The dispersion of the scores in the PCA graph, formed by the first and second principal components, explained 100% of the data variability and formed three groups. PCA of the spectral information revealed that it is possible to distinguish spectra by moisture and granulometry (wet, dry, and ground leaves). Thus, models for distinguishing clones should be developed for each group separately. Partial least squares discriminant analysis (PLS-DA) allowed the prediction of species groups with a high degree of correct classification. The PLS-DA models developed from untreated NIR spectra for wet leaves achieved 93.9% accuracy. The models developed with dry leaf samples showed 95% accuracy in cross-validation. The model developed using spectra collected from dry and ground leaves classified the clones correctly 100% of the time in cross-validation. The results indicate that by removing moisture from the leaves, the models become more robust and capable of identifying the clones. However, even with wet leaves, the model showed a high accuracy rate. The advantage of using the wet leaf model and portable equipment is the ability to apply the technique directly to the tree leaves in the field, outside the laboratory. This study presents innovative results for the forestry sector. The portable NIR technique combined with multivariate statistics proved to be promising for discriminating Eucalyptus clones within the genetic improvement program.

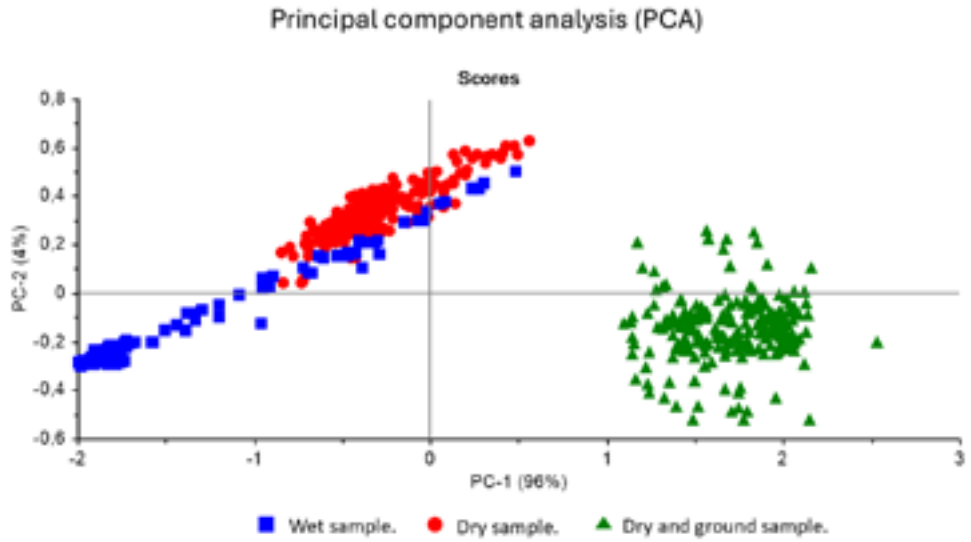


Figure: PCA of spectra from wet, dry and ground leaves of 7 Eucalyptus clones.

ACKNOLEDGEMENTS: Acknowledgments to Klabin S.A.

Keywords
Clone identification,Wood quality,Genetic improvement,NIRS,Cellulose

P01.05.40

MULTI-ANALYTICAL APPROACH TO EVALUATE CHEMICAL ALTERATIONS IN CANNABIS SATIVA EXPOSED TO PER- AND POLYFLUOROALKYL SUBSTANCES

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In recent years, water and soil contamination by per-and polyfluoroalkyl substances (PFASs) has become a pressing issue because of their toxicity to human health. PFAS are synthetic compounds known for their persistence, resistance to degradation, and extensive industrial applications. Since their introduction in the 1950s, nearly 900,000 substances in this category have been utilized across different industries [1, 2].

A primary concern is the contamination of agricultural soils, which facilitates the entry of PFAS into the food chain. Phytotechnology can be a viable sustainable solution to remove and/or manage PFAS contamination [3]. Recent studies have demonstrated the ability of Cannabis sativa L. to remove PFAS from water and soil by accumulating them in the aerial part [4]. The use of hyperaccumulator plants such as Cannabis sativa L., provides a valuable tool for the restoration of contaminated agricultural soils by preventing their accumulation in crops. In addition, hyperaccumulator species can be used as bioindicators for the presence of PFAS in soil. However, the timing and efficiency of PFAS accumulation are critical for the effectiveness of efforts to use Cannabis sativa L. in soil remediation. In this context, hyperspectral imaging (HSI), with can capture both spatial and spectral features, offers a promising approach for monitoring. Indirect detection of PFAS in plant tissues can be achieved by observing metabolic alterations [5] in contaminated plant tissues compared to the uncontaminated ones. In this study we developed an innovative approach using hyperspectral imaging (HSI) coupled with chemometrics to detect variations in PFAS-contaminated Cannabis sativa leaves (Figure 1). To this end dried and ground leaves, flowers, stems and roots were analyzed after 15 days of growth in hydroponic conditions in the presence or absence of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS). Hyperspectral images were acquired in the Short-Wave Infrared (SWIR: 1000–2500 nm) spectral range, using the SisuCHEMA XL™ Chemical Imaging instrument with a 31mm lens and a resolution of 150mm/pixel. The data were preprocessed to emphasize spectral variations between PFOS- and PFOA-contaminated plants and control (non-contaminated) plants. Exploratory data analysis was performed using the t-SNE algorithm, which reduces dimensionality and visualizes hidden patterns or clusters, enabling the identification of differences associated with the presence of PFAS and detecting variations between different plant tissues. To assess the variations observed by HSI, the samples were analyzed using Fourier transform infrared spectroscopy (FTIR) to evaluate potential chemical and metabolic differences in the tissues. In conclusion, the combined approach of SWIR-HSI and multivariate analysis, validated by FTIR spectroscopy, applied to Cannabis sativa, provides a valuable starting point for developing predictive models for monitoring the phytoextraction process of PFAS. This methodology, previously tested [6] on other contaminants such as arsenic, offers new opportunities to optimize contamination monitoring and enhance the effectiveness of phytoremediation.



Figure 1. Image of *Cannabis sativa L.* grown under hydroponic conditions in the presence or absence of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS).

ACKNOLEDGEMENTS: PRIN2022 Funded by the European Union- NextGenerationEU- under the National Recovery and Resilience Plan (NRRP), Mission 4, Component 2, Investment 1.1 call for tender No.104/2022 by the Italian Ministry of University and Research (MUR) - CUP B53D23017010006.

Keywords
PFOS,PFOA,hyperspectral imaging,chemometrics,cannabis sativa l.

P01.05.42

SPECTROSCOPIC ANALYSIS OF AUREOBASIDIUM PULLULANS BIOFILM FOR THE DEVELOPMENT OF LIVING COATING FOR ARCHITECTURE

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Fungal biofilms play crucial roles in ecology, medicine, and industry but remain relatively under-researched. Studying the biofilm of the yeast-like fungus Aureobasidium pullulans provides valuable insights into fungal biofilms with biotechnological potential. Additionally, spectroscopic analysis helps evaluate biofilm composition and structural properties, further enhancing our understanding. In this study, we analysed biofilms of three morphologically different strains of A. pullulans on four different solid media – Yeast Nitrogen Base (YNB), Potato Dextrose Agar (PDA), Sabouraud Agar (SA) and Synthetic Nutrient Deficient Agar (SNA). Depending on their morphology, the strains were inoculated onto solid media as a cell suspension or with a cork border and incubated at 25 °C for two weeks. Afterwards, the biofilms grown on solid media were analyzed using a Fourier transform near-infrared spectrometer (FT-NIR) MPA II and a Fourier transform infrared spectrometer (FTIR) Alpha (both from Bruker Optics GmBH). Three biological replicates of each biofilm sample were prepared and each sample was subjected to five measurements. Spectral pre-processing and data mining were performed using Opus 6.5 (Bruker, Ettlingen, Germany) and PLS_Toolbox (Eigenvector Inc, Manson, IA, USA), an extension of the Matlab package (Mathworks Inc, Natick, MA, USA).

Principal Component Analysis (PCA) of all spectra shows no major differences between the biofilms of the different strains. However, there are clear spectral differences in the nutrient-poor SNA medium compared to other media. This pattern is consistently observed in both spectroscopic methods and confirms that chemical variations in the fingerprint region of the IR spectra are also reflected in the NIR spectra, which capture overtones and combination bands. The analysis of loadings emphasises the importance of the bands associated with -OH groups (around 5000 cm-1 and 7000 cm-1) in the NIR range and -CH groups (around 1000 cm-1) in the IR range. Previous results suggest that all strains adopt a yeast form on SNA medium, which may influence the structure of the biofilm, but further investigation is required.

These findings suggest that biofilm composition and structure are influenced by nutrient availability, with potential implications for adhesion and functional properties in coating applications. The integration of FT-NIR and FTIR spectroscopy proves valuable in characterizing biofilms and can be further used to adjust the formulation of living coating. Future studies will explore the interplay between morphology, biochemical composition, and performance in real-world applications. These results provide new insight into A. pullulans biofilm structure and serve as a foundation for developing an optimal living coating for various materials, which is the main goal of the ARCHI-SKIN project.

ACKNOLEDGEMENTS: This research was funded by the European Union (ERC, ARCHI-SKIN, #101044468). Views and opinions expressed are, however, those of the author(s) only and do not necessarily reflect those of the European Union or the European Research Council. Neither the European Union nor the granting authority can be held responsible for them.

Keywords
Fungi,Biofilm,FT-NIR,FT-IR

FOOD & BEVERAGE

P01.06.45

FT-NIR SPECTROSCOPY FOR THE MONITORING OF SECONDARY SHELF LIFE OF GROUND COFFEE

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Coffee is one of the most consumed beverages worldwide, with more than 10 million tons consumed in 2022 [1] and a world market valued at 41.80 USD billions [2]. Because of its impressive economic value, the precious and highly valued flavor must be protected throughout the entire shelf life, including also the secondary shelf life after the package opening. One of the most impactful defects that causes coffee quality loss is staling, which is mainly due to lipid oxidation and hydrolysis, resulting in rancid and unpleasant off-flavors and aromas. Sensory analysis is considered the golden standard for identifying defects resulting from coffee oxidation, but the coffee industry needs faster analytical methods for decision-making processes. Thus, considering sensory analysis results as references, the aim of this study was to investigate the ability of FT-NIR spectroscopy in monitoring changes during secondary shelf-life of ground coffee exposed to stressful environmental conditions (i.e., high oxygen concentration and storage temperature).

Eighty soft-packs (250 g each) of ground coffee (100% Coffea arabica) were opened and stored in air (O2, 20.9%) at 25, 37, and 45 °C for a maximum of 42 days. At given sampling times, 3 opened packages were analyzed, collecting 50 g of coffee on the upper part of each pack. FT-NIR spectra were collected on ground coffee by using an integrating sphere, in a wavenumber range of 9000-3800 cm-1, with 8 cm-1 resolution, and 32 scans for both background and samples. Sensory analysis was carried out on brewed coffee, applying Quantitative Descriptive Analysis (QDA) performed by 8 trained sensory panelists. Principal Component Analysis (PCA) was applied to highlight sample patterns and relationships between variables.

As expected, samples stored at accelerated conditions were sensory described by high values of oxidation, acidity, and astringency attributes; fresh samples were associated to high values of body, odor intensity, and aftertaste attributes, which then decreased during storage, especially under accelerated conditions (i.e., 37 and 45 °C). Similarly, FT-NIR spectra, after SNV pre-treatment, well distinguished fresh samples from the stored ones, mainly due to OH-bonds of water, C-H bonds of fatty acids (affected by chemical oxidation and hydrolysis during storage), O-H and N-H overtones of chlorogenic acid and amino acids (involved in coffee staling phenomena and generating off-flavors). The combined PCA of SVN-spectra and sensory data revealed that samples stored up to 14 days at 25 °C were characterized by high values of odor intensity, aroma intensity, and body of brewed coffee, which were related to the spectral regions 9000-7200 cm-1 and 4800-4000 cm-1, mainly associated to bonds of fatty acids and amino acids. Samples stored at 45 °C were instead associated to higher values of oxidation and astringency, related to the spectral regions 7200-6000 cm-1 and 5300-4800 cm-1, ascribable to water, chlorogenic acid, and organic acids.

In conclusion, the work demonstrated that FT-NIR spectroscopy can be a valuable tool to monitor secondary shelf life of ground coffee, giving insights in the staling phenomena occurring during storage and described by sensory analysis.

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[2] Custom Market Insight. (2024). Global Roasted Coffee Market. https://www.custommarketinsights.com/report/roasted-coffee-market/

ACKNOLEDGEMENTS: This project was funded by Programma Operativo Nazionale "Ricerca e Innovazione" 2014-2022 – Action IV.5 "Doctorates on green topics" (PON) and Lavazza S.p.A.

Keywords
Coffee,Shelf life,Sensory analysis

P01.06.46

PREDICTION OF SUGARS IN RIPE BANANA PULP FROM SPECTRA OF UNRIPE FRUIT PEEL USING NEAR-INFRARED SPECTROSCOPY AND MULTIVARIATE CALIBRATION

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Bananas are one of the most consumed fresh fruits in the world, and their global market is worth around US$5 billion annually. In addition to their nutritional value, bananas are important economically and socially for many countries. Assessing banana sweetness during ripening is challenging using conventional methods like visual inspection of peel color. This study aims to utilize Near-Infrared Spectroscopy (NIR) and multivariate calibration to predict sucrose, glucose, and fructose concentrations in ripe banana pulp from spectra collected from unripe and ripe fruit peels. From a reliable method for early prediction of sugar concentrations in bananas, is it possible to define the harvest timing and improve post-ripening sweetness, benefiting consumers and agricultural producers. A total of 320 unripe bananas from 19 cultivars were harvested and analyzed. NIR spectra were collected using diffuse reflectance from the unripe and ripe banana peels. Using an adapted method, sugars were extracted from the ripe banana pulp and analyzed through High-Performance Liquid Chromatography (HPLC) with an evaporative light-scattering detector (ELSD). Partial Least Squares (PLS) regression models were built, and Ordered predictors selection (OPS) was used for variable selection. Sucrose, glucose, and fructose concentrations in ripe pulp ranged from 45.10–207.20 mg/g, 23.81–78.18 mg/g, and 21.60–85.26 mg/g, respectively. The PLS models for unripe peel spectra achieved RMSEP values of 15.18 mg/g for sucrose, 3.41 mg/g for glucose, and 8.7 mg/g for fructose, with correlation coefficients (Rp) of 0.93, 0.97, and 0.86, respectively. Models using ripe peel spectra yielded RMSEP values of 19.29 mg/g, 5.29 mg/g, and 6.65 mg/g, with Rp values of 0.85, 0.82, and 0.86, respectively. From the FeediOPS strategy, OPS significantly improved model accuracy by reducing the number of variables. Combined with multivariate calibration, NIR provides a robust, nondestructive approach for predicting sugar levels in bananas early on. The results demonstrate the potential to enhance agricultural practices by predicting sweetness at harvest and improving market quality and consumer satisfaction.

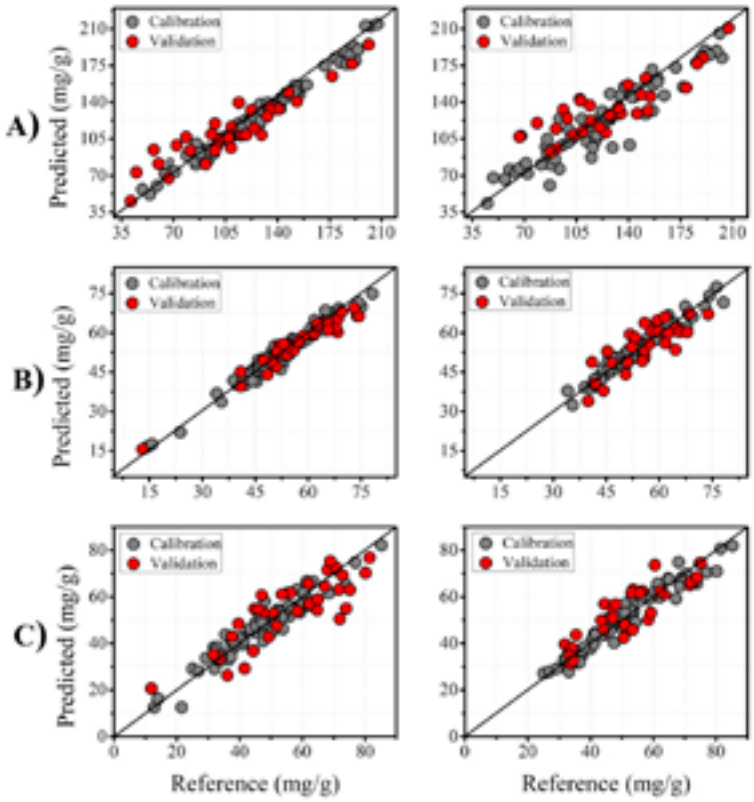


Figure 1. Reference and predicted sugars content in unripe banana on the left and ripe banana on the right for sucrose (A), glucose (B), and fructose (C).

ACKNOLEDGEMENTS: CAPES (Projetc 001), FAPEMIG, DAA-UFV

Keywords
glucose,fructose,sucrose,Early prediction,Banana

P01.06.47

RAPID QUALITY EVALUATION AND MARKET VALUATION OF CRUSH TEAR CURL BLACK TEA USING NEAR INFRARED SPECTROSCOPY

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The quality and market valuation of Crush Tear Curl (CTC) tea are traditionally conducted through professional sensory evaluation. A panel of tea tasters assigns a score ranging from 1 to 10, with higher scores signifying better quality and commanding higher prices. The valuation process hinges on three primary attributes collectively referred to as the LIQ value (liquor, infusion and quality), which is influenced by two major biomarkers present in black tea, theaflavins (TF) and thearubigins (TR). Theaflavin is responsible for the colour, brightness and briskness and directly influence the tasters' palate. Thus, combining TF estimation with tasters' scores offers a balanced approach to tea valuation. Human taste perceptions are inherently subjective and may be prone to inconsistencies, while chemical methods, though precise, require costly instruments, skill and are time consuming, thus do not qualify for routine commercial quality estimation [1,2].

In this paper, an attempt has been made to simultaneously predict the LIQ values and TF contents of commercial CTC black tea using near infrared spectroscopy for rapid valuation and decide the market price. For the study, 100 CTC black tea samples were gathered from local markets of Kolkata and Upper Assam. A taster panel evaluated the reference LIQ scores, while TF values were derived by standard spectrophotometric method. The NIR spectra of samples were obtained using a portable NIR spectrometer (Ayudyog Pvt. Ltd., India) with an InGaAs detector, operating in a diffuse reflectance mode [3]. 2 g of each tea powder was placed a quartz sample holder, and spectral scanning was performed over the wavelength range of 900–1700 nm with 16 scans.

Partial Least Square Regression (PLSR) model was employed to predict the component contents from spectra for sensory scores and TF values. To improve the performance of the prediction models, different spectral preprocessing methods including first derivative (1D), second derivative (2D), standard normal variate (SNV) and multiplicative scatter correction (MSC) were applied [3]. Two variable selection methods, namely random frog (RF) and competitive adaptive reweighted sampling (CARS), were applied to select characteristic wavelengths and eliminate irreverent wavelengths. The optimal spectral preprocessing methods for prediction models of L, I, Q and TF were SNV+1D, MSC, SNV+2D, SNV respectively.

The CARS–PLSR model for TF, liquor (L) and quality (Q) shows preferable predictive performance with higher RPD values, lower RMSEP compared with RF–PLSR model; while the RF–PLSR shows better performance for infusion (I). Notably, the CARS -PLSR model for TF yielded the optimal predictive performance, with the highest Rp of 0.983, and the highest RPD value of 5.39 compared to other models after using variable selection algorithms. Overall, the number of wavelengths used for the model establishment represents a reduction above 80%. The results show that the PLSR model using variable selection methods offers better results than the full spectra model, in terms of Rc, Rp, RMSEC, REMSEP, and RPD. The proposed method is feasible for rapid quality control and taste-related attributes evaluation and valuation of CTC tea.

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Keywords
theaflavin,liquor-infusion-quality (LIQ),CARS,random frog (RF)

P01.06.48

EXPLORING TEXTURIZATION HYPOTHESIS IN HIGH MOISTURE EXTRUSION USING HYPERSPECTRAL IMAGING AND MACRO LENS

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IRTA, Monells, SPAIN

Meat analogues are food products made from non-meat ingredients, eaten as a replacement for meat products. However, to mimic meat appearance and mouthfeel is challenging. In this regard, the High Moisture Extrusion Process (HMEP) combines mixing, extrusion-cooking, and cooling to create high moisture extrudates (HME) with a fibrous texture similar to animal muscle fibers, ideal for developing meat analogues. Despite the research conducted so far, there is still a limited knowledge about the fibrous texture development during HMEP. Two main mechanisms are used to explain the formation of anisotropic structures: the alignment of protein molecules and the existence of a multiphase system [1]. Another hypothesis suggests that the fibrous texture results from a syneresis phenomenon that occurs as the extrudate cools and solidifies in the cooling die [2]. As the protein matrix cools rapidly, it contracts and expels water, resulting in a layered structure in the extrudates, with a water-rich phase surrounded by a protein-rich phase. Previous works show that the water-rich phase can be imaged via X-ray microcomputed tomography, or via time-domain NMR The aim of this study was to analyse the distribution of water content in HME and explore the hypothesis that the phase separation, resulting in the formation of water-rich phases surrounded by protein-rich phases, plays a role in the formation of fibrous texture, by using hyperspectral imaging combined with a macro lens. To do so, HME with varying levels of fibrousness (anisotropic index ranging from 1.2 to 1.8) were produced under different extrusion conditions using a soybean protein isolate. Hyperspectral images of HME were acquired with a Specim FX17 camera (Specim Spectral Imaging Ltd.; Oulu, Finland) equipped with an OLES macro lens, giving a pixel dimension of 0.2 µm. Samples surface was scanned. Thereafter they were cut down in the middle transversely or longitudinally and the internal faces were also scanned. In parallel, soybean HME with a water content ranging from 60.7 to 76.5% were also produced and scanned using the same system to develop a water content predictive model. This model was used to calculate water content chemical images of the HME having different fibrousness levels. The water content predictive model developed had an error of 0.93% and R2 of 0.98, being accurate enough to be used to evaluate water distribution in HME. Chemical images of samples surface revealed a higher water content in the central part of the extrudate. The lower water content on the extreme sides is explained by the more intense and rapid cooling experienced by these parts due to the higher contact with the cooling die during production. Chemical images from cut samples revealed some water rich phases, but the inherent fibrousness interferes with image acquisition, causing shadows that might lead to incorrect conclusions. Another shortcoming could be that near-infrared light penetrates multiple water-protein-rich phases, which might overlap, resulting in a mean spectrum of several overlapping phases that makes impossible to distinguish their different compositions. It can be concluded that there is a different distribution of water inside the extrudate, but more experimental work is needed to confirm the water-protein rich phases hypothesis.

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ACKNOLEDGEMENTS: This work was financially supported by SENSANALOG [PID2021-122285OR-I00] and grants for doctorate's studies [PRE2022-103798] and FPU20/04009 funded by MCIN/AEI/10.13039/501100011033. Acknowledgements are extended to the consolidated research group (2021 SGR 00461) and CERCA program from Generalitat de Catalunya.

Keywords
meat analogues,texture,anisotropy index,high moisture extrusion,food structure

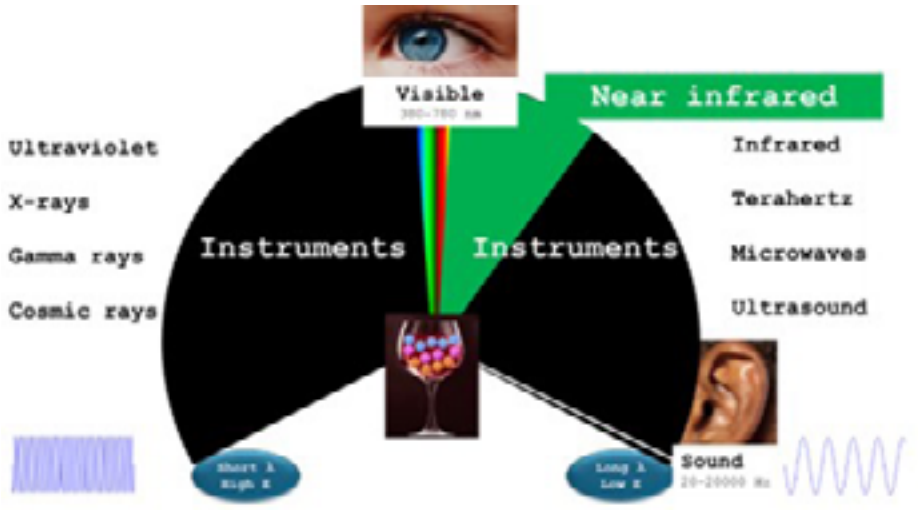
P01.06.49

APPLICATION OF PORTABLE NIR SPECTROSCOPY IN TABLE WINE ANALYSIS

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Wine quality and composition are key factors influencing sensory attributes such as flavor, aroma, mouthfeel and color, which shape consumer preferences. Accurate measurement of bulk components like ethanol, residual sugars, and acidity ensures product consistency and compliance. In EU regulations is has become obligatory to label wines with their nutritional energy. Classical analytical methods, while precise, are time-consuming and labor-intensive, prompting interest in rapid, non-destructive techniques like IR and NIR spectroscopy. This analytical technology enables real-time prediction of multiple chemical parameters, supporting efficient quality control while preserving the sample and meeting sensory and consumer demands. Additionally, NIR spectroscopy is an environmentally friendly green technology, as it requires no chemical reagents, generates no waste, and significantly reduces resource consumption compared to traditional analytical methods [1].



In this study, 235 wines from different countries, mostly red wines, were analyzed to obtain reference values for important chemical properties. These samples were also tested with a benchtop NIR spectrometer and two portable NIR instruments. The reference data were used to create Partial Least Squares (PLS) models for predicting bulk wine components like ethanol, glucose-fructose, total acidity, volatile acidity, pH, malic acid, and color spectra. Portable NIR instruments are not only cost-effective, but also lightweight and easy to transport, making them a practical solution for on-site measurements. Their accessibility and ease of use imply that they have the potential to be easily implemented as a reliable analytical method, even in smaller vineyards or wineries with limited resources.

In addition, consumer preference data were collected from a larger group of people who purchased these wines through Vivino and the main focus of this study will be on the relation between sensory attributes and consumer vocabulary and the NIR spectra, exploring how well the NIR technology can capture attributes that influence consumer choices.

ACKNOLEDGEMENTS: [1] T. Czaja, S.B. Engelsen, Spectrochimica Acta A, 2025, 325, 125028

Keywords
NIR,Handheld NIR,food,wine,consumer data

P01.06.51

REAL-TIME MONITORING OF PARTICLE SIZE REDUCTION IN DARK CHOCOLATE USING NEAR-INFRARED SPECTROSCOPY

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Refining is a critical step in chocolate production that reduces particle size, followed by conching, which develops flavour and texture [1,2]. Traditionally, these are separate processes, but melangers - machines that combine refining and conching into a single operation - are increasingly popular due to their scalability and relatively low initial capital investment. Despite their widespread use, the relationship between processing time in a melanger and particle size reduction, a key factor in determining the texture and quality of chocolate, has not been thoroughly studied. This study investigates the impact of processing time in a melanger on the particle size reduction of dark chocolate using near-infrared (NIR) spectroscopy. By examining how processing time influences particle size, the study provides valuable insights for optimising chocolate production, particularly for small-scale and artisanal chocolate makers who rely on melangers. Understanding this relationship can help producers achieve consistent quality, desired texture, and improved efficiency. Furthermore, the use of NIR spectroscopy offers a rapid, method for monitoring particle size, with the potential to enhance quality control in chocolate manufacturing.

Sampling was conducted at 15-min intervals throughout the particle reduction process until a particle size of 20 um was reached. Reference particle size measurements were obtained using a digital micrometer, and spectra of the molten chocolate were collected using two NIR devices: a benchtop NIR instrument (Molten benchtop; Büchi NIRFlex; 1000-2500 nm) and a handheld NIR device (Molten handheld, Viavi; 950-1650). Descriptive analyses of the reference particle size values showed a non-linear particle size reduction (due to the formation of agglomerates), over time and a 480-min particle size reduction period was needed to reach a particle size of 20 um.

Principal component analysis (PCA) effectively differentiated five particle size groups when using standard normal variate (SNV) and mean centering (MC) as preprocessing techniques. Clustering of these particle size groups was evident in the direction of PC1 for the Molten benchtop samples, while separation of the Molten handheld samples was clear in the direction of PC2, preprocessed with MC and SNV+MC, respectively.

Partial least squares (PLS) prediction models were developed using the calibration and validation sets using combined PCA and DUPLEX. Model optimisation was done with Venetian blinds cross-validation with 14 splits. The PLS prediction model for the Molten benchtop data set (preprocessed using SNV+MC) resulted in a root mean square error of prediction (RMSEP) of 5.40 um, a coefficient of determination (R2) of 0.97, and a ratio of performance to deviation (RPD) of 6.0. Similarly, for the Molten handheld matrix (preprocessed with MC), the RMSEP, R^2, and RPD values were 6.59 um, 0.96, and 4.9, respectively. The RMSEP-values were both in the range of the obtained standard error of laboratory (SEL) value (3.28 um), indicating reliable prediction capabilities. Although results indicated a higher prediction accuracy for benchtop instrument, it obtained a larger prediction bias of 2.38 compared to 1.83 for the handheld device. This work illustrates the use of NIR spectroscopy as an effective quality control tool, providing real-time data, during chocolate manufacturing.

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ACKNOLEDGEMENTS: Carli Pretorius gratefully acknowledge receiving funding from European Union's Horizon 2020 research and innovation programme under grant agreement (#101008228).

Keywords
Chocolate,Refining,Conching,Melangers,PLS regression

P01.06.53

FT-NIR SPECTROSCOPY IN SUPPORT OF THE EU AUTHORIZED HEALTH CLAIM FOR OLIVE OIL POLYPHENOLS

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The hydrophilic phenolic molecules are peculiar minor compounds of virgin olive oils particularly important as antioxidants and responsible for imparting the characteristic pungent and bitter attributes of the oil. Their protective action against oxidation is essential not only for better preservation of the product but also for providing potential health effects. The latter capability has been remarked by one health claim that can be used to label olive oils as follows: Olive oil polyphenols contribute to the protection of blood lipids from oxidative stress. The claim must be accompanied by the information that the beneficial effect is obtained with a daily intake of 20 g of olive oil containing at least 5 mg of hydroxytyrosol and its derivatives (e.g., oleuropein complex and tyrosol) [1]. The health claim is an important marketing lever in raising the willingness to pay for a product, but world producers do not take advantage of it because there are still obstacles to their use [2]. Among these, the lack of a lack of rapid and non-destructive method for determination of hydroxytyrosol and tyrosol derivatives (secoiridoids).

In this context, the presented research activity aimed at developing an analytical approach based on FT-NIR spectroscopy for a quick estimation of olive oil polyphenols related to the health claim.

The hydrophilic phenolic fraction of 42 virgin olive oil samples of different geographical origin and different storage times were extracted by means of a hydroalcoholic mixture followed by acid hydrolysis and ultra-high performance liquid chromatography (UHPLC-UV-Vis) [3].

Samples were characterized by a wide range of the sum of hydroxytyrosol and tyrosol (3.3 - 24 mg in 20 g of olive oil), however, the samples showing a content below the minimum threshold required to use the health claim were too few (7 out of 42) to cover the expected variability. The same samples were analyzed by FT-NIR spectroscopy in transmission mode by using a cuvette with 8 mm path-length, in a wavenumber range of 12500-4000 cm-1 with 8 cm-1 resolution, and 16 scans for both background and samples.

The reference analysis results and the FT-NIR spectral features were used to develop a Partial Least Square regression (PLS-R) model to predict the sum of hydroxytyrosol and tyrosol content.

The limited sample numerosity enabled the development of an internally validated PLS model (6-fold Venetian Blinds cross-validation). The model showed a coefficient of determination of 0.81 in cross-validation and a root mean square error of 1.8 mg/20 g of oil.

Despite the promising results, before model exploitation an increased number of samples will be necessary, especially those with low polyphenol content, to externally validate the model.

The proposed approach could allow producers and control bodies to quickly characterize the virgin olive oils, implementing simple and fast control systems to assess the conservation status and the current EU Regulation requirement for the health claim.

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ACKNOLEDGEMENTS: OoFoods - Research and innovation network on food and nutrition Sustainability, Safety and Security – Working ON Foods

Keywords
hydroxytyrosol,tyrosol,Partial Least Square regressio,PLS

P01.06.54

MONITORING ALCOHOLIC AND ACETIC FERMENTATION IN RED GRAPE MUST: A COMPARISON OF PLS MODELS OBTAINED ON THREE FT-NIR SPECTRAL RANGES

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Wine vinegar is a food product obtained through the dual fermentation of grape must. This process unfolds in two main stages: first, yeasts convert the sugars present in grapes into ethanol [1], and subsequently, acetobacteria oxidize the ethanol produced in the initial phase into acetic acid [2]. The full completion of this transformation can take several weeks, requiring periodic monitoring to track the progress of fermentation and measure key byproducts, such as ethanol and total acidity.

The growing demand for improved product quality and production efficiency in the food industry has driven a shift from traditional, destructive analytical techniques toward faster and more sustainable alternatives. Among these, Near-Infrared (NIR) spectroscopy stands out for its versatility, as it is both rapid and non-invasive. This makes it well-suited for online process control, facilitating automation. Numerous studies in the literature have explored alcoholic and acetic fermentation processes, utilizing NIR spectroscopy to quantify various analytes that indicate different fermentation stages [3, 4].

To our knowledge, no previous research has examined the entire progression of dual surface fermentation, covering both phases over an extended time frame. In this study, we monitored the simultaneous fermentation of two different batches of red grape must to evaluate the reproducibility of process kinetics when musts from distinct red grape varieties are subjected to the same environmental conditions. Fermentation was tracked over nearly four months by measuring ethanol content and total acidity, assessing their evolution over time.

Simultaneously, grape must samples were analysed in transmission mode using a Bruker Multi Purpose Analyzer (MPA) spectrophotometer across a broad spectral range of 12500–4000 cm-1 (equivalent to 800–2500 nm). Initially, the obtained spectra were examined using Principal Component Analysis (PCA) as an exploratory chemometric method. Subsequently, Partial Least Squares (PLS) regression models were developed to predict both ethanol content and total acidity. These models, created using the full spectral range, were compared with similar models built on two narrower spectral regions: 10526-6060 cm-1 (950-1650 nm) and 12500-9346 cm-1 (800-1070 nm). These spectral ranges are particularly relevant since they align with commercially available, more affordable, and easily miniaturized sensors, as opposed to the costly TE-InGaAs sensor required for analysing the full spectrum.

The monitoring of alcoholic and acetic fermentation in red grape musts by means of FT-NIR analysis has demonstrated its effectiveness in accurately determining ethanol content and total acidity levels. This applies both to the entire spectral range and the narrower 10526-6060 cm-1 region, where prediction accuracy was notably high (R^2Pred > 0.98). Results in the 12500-9346 cm-1 range were not as strong but remained acceptable (R^2Pred > 0.81), confirming the feasibility of employing simpler, cost-effective devices for real-time monitoring of fermenting must.

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Keywords
Red grape must,Fermentation,Process monitoring,Multivariate calibration

P01.06.55

CRYOGENIC FREEZING TO KILL ANISAKIS IN FISH FILLET AND NEAR INFRARED SPECTROSCOPY TO VERIFY ITS APPLIANCE: PRELIMINARY DATA

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To meet the demands of the seafood market, companies are constantly in search for new processing techniques aimed at offering adequate shelf life and safety of raw products.

Consumers' health can be endangered by the consumption of raw seafood due to the presence of bacteria and parasites, including Anisakis spp. To date, European legislation (Regulation (EC) No 853/2004 and Commission Regulation (EU) No 1276/2011) provides that fish intended for raw consumption must undergo a freezing treatment to guarantee the safety of the products.

The aim of this study was to evaluate the effect of cryogenic freezing on Anisakis spp vitality and to explore whether Near infrared (NIR) spectroscopy combined with the chemometric analysis can discriminate between fish samples subjected to cryogenic treatments and those not subjected to it.

Materials and Methods

Fish fillets (n=20) of farmed Sea bream (Sparus aurata) were divided in two groups, identified as infested (T) with three larvae of Anisakis spp and not infested (NT). All samples, except one from group T were subjected to cryogenic treatment (-70°C for 20 minutes).

NIR spectra acquisitions were performed with a portable NIR device (MicroNIR by VIAVI), PRE and POST cryogenic treatment. The raw data were analysed with Unscrambler software (VIAVI).

After every treatment the vitality of Anisakis was evaluated by using a specific protocol.

Results

All larvae extracted from fish fillets subjected to cryogenic treatment died, while larvae taken from a sample infested and not subjected to treatment were still alive.

As for chemometric analysis, supervised classification was performed with Linear Discriminant Anlaysis (LDA), and good discrimination was found between the samples before and after cryogenic freezing. In details, the LDA was able to efficiently identify the two classes of samples with an accuracy higher than 90%.

Discussion and Conclusion

The growing consumer demand for high-quality raw products becomes an opportunity to develop new strategies to ensure product safety in short time suggesting the cryogenic freezing as a valuable strategy to kill Anisakis spp. In addition, the preliminary results from NIR spectroscopy coupled with chemometrics suggest the ability of this approach to discriminate fish fillets treated from those not treated.

ACKNOLEDGEMENTS: This study was supported by the Italian Ministry of Health, under Grant nr. IZSPLV 04-23 - RC.

Keywords
Anisakis spp,food safety,cryogenic treatment,chemometrics

P01.06.56

PORTABLE NEAR-INFRARED SPECTROPHOTOMETERS FOR EVALUATING AVOCADO MATURITY

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Near Infrared Reflectance Spectroscopy (NIRS) was used as a non-destructive method for evaluating the quality of avocados. The avocado (Persea americana) is a highly valued fruit due to its rich nutritional profile, distinctive flavor, and aroma [1, 2]. Avocados are particularly prized for their high content of polyunsaturated fats, which help mitigate the risk of cardiovascular diseases, as well as their significant levels of monounsaturated fatty acids, such as oleic acid. The selection of the optimal harvest time is crucial, as it directly influences the pre-harvest quality and determines post-harvest quality [3]. Typically, mesocarp fat content, along with related parameters such as dry matter, are considered reliable indicators of maturity. These parameters are fundamental for determining the optimal time for harvesting [3, 4].

The objective of this study was to evaluate the performance of two portable NIR instruments for predicting dry matter and fat content in whole avocados as a non-destructive approach to assessing their maturity and determining their optimal harvest time. Two portable NIRS instruments were used: the MicroNIR™ Pro 1700 and the SCiO v.1.2, each with distinct optical characteristics and features. Predictive models for dry matter and fat content were developed using Modified Partial Least Squares (MPLS) regression. Calibration equations were developed using the entire dataset (N = 100). Spectra pretreatment Standard Normal Variate (SNV) + Detrending (DT) were applied for scatter correction [5], alongside various derivative mathematical treatments. The results confirm the feasibility of NIRS technology for predicting dry matter and fat content in intact avocados using portable devices. The SCiO device demonstrated superior predictive accuracy for both parameters, with standard errors of cross-validation (SECV) of 1.89% and 1.87%, and cross-validation coefficients of determination (r^2VC) of 0.86 and 0.87 for dry matter and fat content, respectively. These findings underscore the potential of NIRS as a reliable tool for decision-making in harvest timing and the evaluation of internal avocado quality.

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Keywords
avocado maturity,fat content,dry matter content,portable NIR sensors

P01.06.57

CHEMICAL AND PHYSICAL MARKERS FOR ANNURCA APPLE QUALITY ASSESSMENT BY AQUAPHOTOMIC

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The Annurca apple is a typical Italian cultivar of the Campania Region. Immediately after harvest, the fruit undergoes a maturation process known as "reddening", which involves exposing the fruit to the sunlight for 10-15 days. This practice, referred as "melaio", is essential to enhance the qualitative characteristics of the fruit (e.g. colour and aroma). After the reddening, and before marketing, apples are stored in air or controlled atmosphere to ensure the preservation of fruits' organoleptic characteristics and nutritional properties.

This study focused on the aquaphotomic approach to monitoring two chemical and physical markers of apple quality: acidity (malic acid) and firmness, both of which are directly correlated to apple quality during storage.

Annurca apples, whether reddened or not, were stored in either air or controlled atmosphere, as previously reported [1]. Fruit samples underwent: 1) NIR characterisation in reflectance mode by portable MicroNIR 1700 (VIAVI solutions, Italy) in the 900 – 1700 nm wavelength range; 2) titratable acidity expressed as malic acid [1]; 3) firmness assessment by an Instron Model 1140 Universal Testing Machine, High Wycombe, UK [1].

Data pre-processing was performed according to Tsenkova et al. [2] after transformation from reflectance to absorbance. Subsequently, focusing on the 1311-1559 nm range (1st overtone WAMACS), the dataset underwent PLSR and LASSO (Least Absolute Shrinkage and Selection Operator [3]) procedures, which worked on one training set containing 60% randomly chosen observations and a test set containing the remaining 40%.

If on the one hand, the PLSR model using 15 most impacting variables seemed to perform well in identifying the malic acid and firmness data (Rmalic train = 0.83; RMSE malic train = 0.05; R2malic test = 0.82; RMSEmalic test = 0.06; R2firmness train = 0.76; RMSE firmness train = 0.74 R2firmness test = 0.81; RMSEfirmness test = 0.78); the LASSO procedure allowed achieving comparable model performance using 11 variables for malic acid (R2malic train = 0.84; RMSE malic train = 0.05; R2malic test = 0.82; RMSEmalic test = 0.05) and 7 variables for fruit firmness (R2firmness train = 0.75; RMSE firmness train = 0.76 R2firmness test = 0.80; RMSEfirmness test = 0.82).

The Aquaphotomics approach allowed monitoring of the chemical changes that occur during fruit softening. Throughout the processes of ripening and storage, the WAMACS related to water structures with weak-hydrogen bonds increased, whereas the WAMACS related to more organized water structures with medium-strong hydrogen bonds decreased. These rearrangements suggested the establishment of new bonds with the remaining water molecules.

The results also point out the suitability of the LASSO procedure for chemical marker monitoring for the quality assessment of Annurca apple. This method significantly simplifies the chemometric process by identifying redundant wavelengths within the first overtone of the water absorbance region.

KEYWORDS: Apple quality; LASSO analysis; WAMACS; Malic acid; Fruit firmness

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ACKNOLEDGEMENTS: Authors gratefully acknowledge receiving funding from the PSR CAMPANIA 2014/2020 – Tipologia di Intervento 16.1.2 Az. 2– "Sostegno ai GO del PEI per l'attuazione di progetti di diffusione delle innovazioni nell'ambito del rafforzamento dell'AKIS campano" - TECNOMELA project.

Keywords
Apple,LASSO analysis,WAMACS,Malic acid,Fruit firmness

P01.06.58

A COMPARATIVE ANALYSIS OF GLASS AND PLASTIC PETRI DISHES FOR ASSESSING BUTTER QUALITY

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This study evaluated the proximate composition and fatty acid profiles of 102 butter samples using near-infrared (NIR) spectrometer with a wavelength range of 850–2500 nm. The main objective was to assess and compare the predictive performance of NIR calibration models when using two different petri dishes: reusable glass and disposable plastic. The butter samples were divided into a calibration set (81 samples) and a validation set (21 samples). Chemical analyses included measurements of dry matter (DM), moisture, ash, total fat, and various individual fatty acids ranging from short-chain to long-chain species. Principal Component Analysis (PCA) confirmed the influence of packaging materials on spectral properties, with clear clustering of samples stored in glass and plastic. This clustering is clearly due to the pronounces spectral absorption features of plastic petri dishes. Spectral data from glass and plastic petri dishes were used to develop partial least squares (PLS) regression models. Calibration models from glass generally yielded higher coefficients of determination (R^2) and lower root mean square errors of calibration (RMSEC) than those from plastic. (result shown in Table 3)

Prediction for both of Moisture and Fat had R^2 around 0.77 and RMSEP around 0.5%, for glass petri and slightly lower performances with plastic petri dishes (R^2 = 0.70 and RMSEP around 0.75%. With this accuracy, calibrations with glass or petri dishes would be suited to evaluate proximate butter composition.

Some fatty acids, such as C14:0, showed strong calibration performance (R^2 around 0.95–0.96) but had RMSEP/RMSEC values exceeding 2.0, indicating potential overfitting. Similarly, C18:3n6 and C10:0 exhibited high RMSEP/RMSEC ratios (>2.0), raising concerns about generalization. Conversely, ash and total fat predictions were poor, with low or negative validation R^2 values, suggesting that NIR may not be reliable for these constituents.

In conclusion, while glass petri dishes enhance calibration performance, both petri dishes types provide similar prediction accuracy for most chemical components in butter. However, certain fatty acids demonstrated overfitting, and ash showed weak predictive performance, highlighting NIR limitations. This study emphasizes the influence of packaging on spectral data and the need for refined NIR calibration models. Future research should explore advanced modeling approaches, such as machine learning and feature selection, to improve predictions for challenging components and enhance NIR applications in dairy product quality assessment.

ACKNOLEDGEMENTS: We ackoledge the gret work of dr. Federica Rela in sampling and laboratory work and the Naresuan University for the financial support during Saowaluk Rungchang internship in Padua

Keywords
Butter composition,Sample Presentation,Laboratory instrument

P01.06.59

COMPARATIVE ANALYSIS OF FAT, SUCROSE, AND COCOA CONTENT IN DARK CHOCOLATES USING LABORATORY AND HANDHELD NEAR INFRARED SPECTROMETERS

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Near-infrared spectroscopy (NIR) is a fast and eco-friendly method used to analyse various quality-related parameters in both cocoa and chocolate. Prediction models for chocolate analysis were developed by using laboratory instruments and various chemometric methods. Nowadays, portable handheld devices are becoming more popular, allowing for immediate on-site examinations during cultivation, production, and manufacturing.

This research aims to investigate whether handheld NIR spectrometers can be used to estimate quality-affecting parameters in dark chocolate, such as fat, sucrose, and cocoa content.

For the analyses, 49 commercially available dark chocolate samples were obtained, with cocoa content ranging from 46% to 100%. An integrated sample preparation method, validated in a previous publication (Benes és mtsai., 2025), was employed to determine the fat and sucrose content of the samples. Fat content was measured gravimetrically following petroleum ether extraction, while sucrose concentration was determined using high performance liquid chromatography. For cocoa content, the values indicated by the manufacturers on the packaging were used as the reference.

Spectra were acquired using both a benchtop and a handheld NIR spectrometer. A Fourier transform NIR (FT-NIR) device (MPA, Bruker Optics, Ettlingen, Germany) was employed to collect diffuse reflectance spectra within the 12500-4000 1/cm wavenumber range, at a resolution of 16 1/cm. Approximately 50 g of each sample was used in a rotating quartz cuvette, with the samples broken into smaller pieces. Additionally, a handheld NIR-S-G1 instrument (InnoSpectra Co., Hsinchu, Taiwan) was utilized to collect spectra in the 900-1700 nm wavelength range, at a resolution of 4 nm. The diffuse reflectance spectra of the samples were collected through low-density polyethylene bags.

For data analysis, 147 spectra were utilized. Principal component analysis with random seven-fold cross-validation was employed for exploratory data analysis. Various spectral pre-processing methods were applied to reduce data variability and extract chemical information. These analyses were performed using Unscrambler X 10.4 software (CAMO Software, Oslo, Norway). Prediction models were developed using partial least squares regression (PLSR) in MATLAB R2019b software (Mathworks Inc., Massachusetts, USA) with PLS_TOOLBOX v. 9.3 (Eigenvector Technologies, Research, Inc., USA). Model performance was evaluated through random seven-fold CV and an independent set of samples.

The fat content varied between 30.7 and 63.66 g/100 g, and the sucrose content of the samples ranged from 0.55 to 46.6 g/100 g. Considering the PCA results, the dataset did not contain spectral outliers based on the Hotelling-T2 and F-residual values. In PLS regression, the models were initially developed using only smoothed data. Generally, the models for fat, sucrose, and cocoa content had determination coefficients greater than 0.91 for the handheld device. In contrast, for the benchtop spectrometer, this value was 0.95 for the validated models. After applying various data pre-processing methods, the statistical parameters of the models were improved. The R^2 value increased, while the RMSE/RMSECV decreased compared to the initial models.

The results indicate that, in addition to the benchtop device, the handheld NIR spectrometer is also suitable to predict various parameters of dark chocolates.

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Benes, E., Matejka, G., & Fodor, M. (2025). Near-infrared spectroscopy for comprehensive analysis of dark chocolate composition. Food Chemistry, 469, 142562.

ACKNOLEDGEMENTS: EB thanks the support of the EKÖP-24-VI/MATE-13 Program of the Ministry for Culture and Innovation from the source of the National Research, Development, and Innovation Fund.

Keywords
dark chocolate,composition,quality,regression

FORENSICS

P01.07.60

APPLICATION OF PORTABLE MICRONIR SPECTROSCOPY AND CHEMOMETRIC ANALYSIS FOR THE RAPID CHARACTERIZATION OF COMMERCIAL CANNABIDIOL (CBD)

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The potential of handheld NIR instrumentation in the most diverse fields is attested by a consolidated bibliography, resulting a very useful tool for acquiring fast and accurate quali-quantitative analytical responses. Handheld MicroNIR devices offer the advantage of in-field analysis without the need for sample preparation, making them an attractive alternative to conventional laboratory techniques [1].

Cannabidiol (CBD) is actually at the center of a debate due to the significant difference in cost at a commercial level between the product extracted from inflorescences and the one obtained synthetically, which is much cheaper.

A joint study, based on handheld MicroNIR/Chemometrics approach validated by HPLC-MS/MS, has been conducted on samples from certified synthesis processes and samples deriving from certified extraction processes on inflorescences. Results from both the techniques are reported and propose a new approach for the characterization of commercial CBD.

This portable and handheld instrument has proven to be a fundamental aid for identifying illicit commercial conduct, aiding law enforcement in regulatory compliance. Additionally, it enables quality control in hemp-derived products by verifying the presence of target cannabinoids, naturally present after extraction and purification processes, or added to synthetic CBD to mask the real origin.

The proposed analytical approach highlights the MicroNIR/Chemometrics potential as a powerful tool for the analysis and monitoring of controlled substances, reinforcing its role as an innovative and efficient method for real-time chemical assessment.

ACKNOLEDGEMENTS: [1] G. Gullifa, L. Barone, E. Papa, A. Giuffrida, S. Materazzi and R. Risoluti, Front. Chem., 2023, 11

Keywords
Chemometric,MicroNIR

P01.07.61

A NEAR-INFRARED AUTONOMOUS SYSTEM FOR IN-SITU FORENSIC ANALYSIS OF ILLICIT DRUGS

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There is a strong call for forensic analysis to meet police force expectations for in-situ results that can assist in timely decision-making on the composition of seized samples. Near-infrared spectroscopy (NIRS) has succeeded in identifying and quantifying illicit substances. Compact low-cost spectrophotometers present all requisites to be deployed for in-situ analysis.

This work describes and evaluates an autonomous system based on the NanoNIR spectrophotometer, a laptop computer, and a friendly user software and interface written in Python to provide instantaneous identification and quantification of actives in illicit drugs. The system was evaluated for seized samples of cocaine and ecstasy.

The NanoNIR (Texas Inc. Instruments) is encapsulated in a 3D-printed case and connected to the computer's USB port. The case features a sample compartment where a glass vial (1 cm diameter x 5 cm length) containing the ground sample is fitted for spectrum acquisition. The system is shown in Figure 1a. The user interface is shown in Fig. 1b.

Principal Component Analysis (PCA) and Partial Least Square (PLS) models to identify and quantify seized cocaine and ecstasy samples were developed in a primary system as previously described [1,2]. The pertinent model parameters were transferred to the system files, allowing their stand-alone application. Two other secondary systems were standardized using Piecewise Direct Standardization (PDS) to distribute the models constructed using the primary instrument. The system interface asks for the insertion of the suspect drug type. It guides the user to obtain a valid spectrum, checking for the data quality before it is automatically transformed by the 2nd derivative (9 points, 2nd-degree polynomial) and presented to the qualitative and quantitative models. The connected instrument is identified automatically based on its serial number, and the proper standardization matrix is applied to the secondary spectrum. Common mistakes, such as lack of the sample in the vial, lack of vial in the sampling compartment, and lack of 100 % reflectance (Spectralon®) reference measurement before sample measurement, are prevented by the system software which alerts the user for procedure correction. The qualitative results are presented in three levels to the user (positive, negative, and suspect). Samples identified as suspect are recommended to be sent to the laboratory for more detailed analysis.

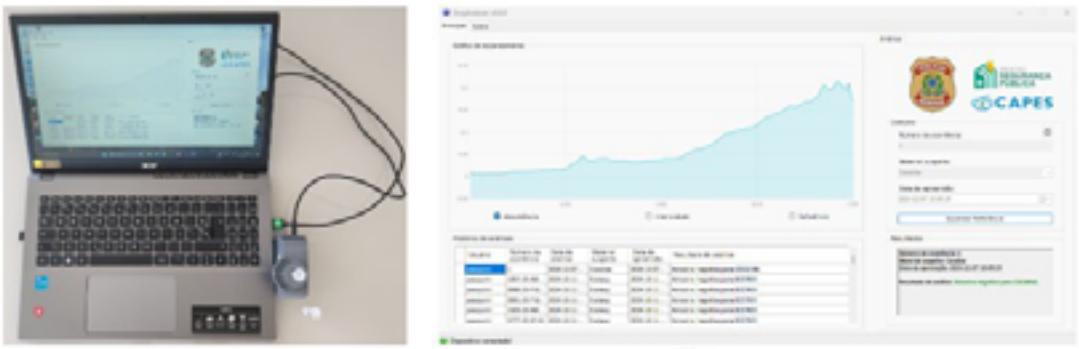
Presently, the system can identify and quantify the two most common forms of cocaine (base and hydrochloride) and the two most common forms of ecstasy (MDA and MDMA). After identifying the sample, regression models are employed to estimate the content of the illegal drug in the seized samples. The scope of applications can be extended due to the software's modular architecture.

The system performance was evaluated by analyzing independent sets comprised of 126 and 98 seized samples of cocaine and ecstasy. The qualitative and quantitative results are depicted in Table 1. The results corroborate the use of the autonomous system for screening analysis of seized samples.

The proposed system presents a total cost of about US$ 2.500,00; it is self-sufficient and can operate in the field for at least 6 hours. No web connection is required as the system may have to operate in locations where a connection is unavailable. The system represents a solution to apply the efforts of chemometricians and analytical chemists to real-world scenarios of forensic analysis.

[1] https://doi.org/10.1016/j.saa.2024.124163

[2] https://doi.org/10.1016/j.talanta.2019.03.091



(a) (b)

Figure 1. (a) Autonomous system; (b) User interface showing a negative result for cocaine

Table 1. Validation results for autonomous system

parameter	instrument					
	primary		secondary-1		secondary-2	
	cocaine	ecstasy	cocaine	ecstasy	cocaine	ecstasy
N	126	98	125	98	126	98
FN	14	0	12	0	13	0
S	7	8	19	11	8	5
FNR	11	0	10	0	10	0
RMSE (%)	5.4	7.5	6.1	7.6	5.3	6.7
R^2	0.776	0.900	0.784	0.896	0.815	0.898
RMSE−BIAS (%)	3.2	6.5	4.2	6.9	4.0	5.8

N: number of samples; FN: false negative; S: suspect; FNR: false negative rate; RMSE: root mean squared error; R^2: determination coefficient; RMSE/BIAS: error corrected for bias

ACKNOLEDGEMENTS: CNPq/INCTAA 465768/2014-8; FAPESP/INCTAA 2014/50951-4; CNPq 305649/2021-3, 304199/2019-2; CAPES/PROCAD-SPCF 88887.608496/2021-00, 88887.808376/2023-00, 88887.838359/2023-00

Keywords
autonoumos system,forensic analysis,cocaine,ecstasy,near infrared spectroscopy

FUTURE TRENDS

P01.08.62

EXPANDING HORIZONS OF NEAR-INFRARED SPECTROSCOPY THROUGH A SMALL DOMAIN-SPECIFIC LANGUAGE MODEL

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Near-infrared spectroscopy (NIRS) is becoming increasingly important in science, industry, and education, creating a growing demand for specialized tools that share and build upon domain-specific knowledge. This work introduces a small, domain-specific language model (DSLM) focused on NIRS. The model is based on the Llama 3.21 architecture, which features 13 billion parameters and provides state-of-the-art natural language understanding capabilities. It has been fine-tuned using 1.2 GB of curated datasets specifically related to NIRS.

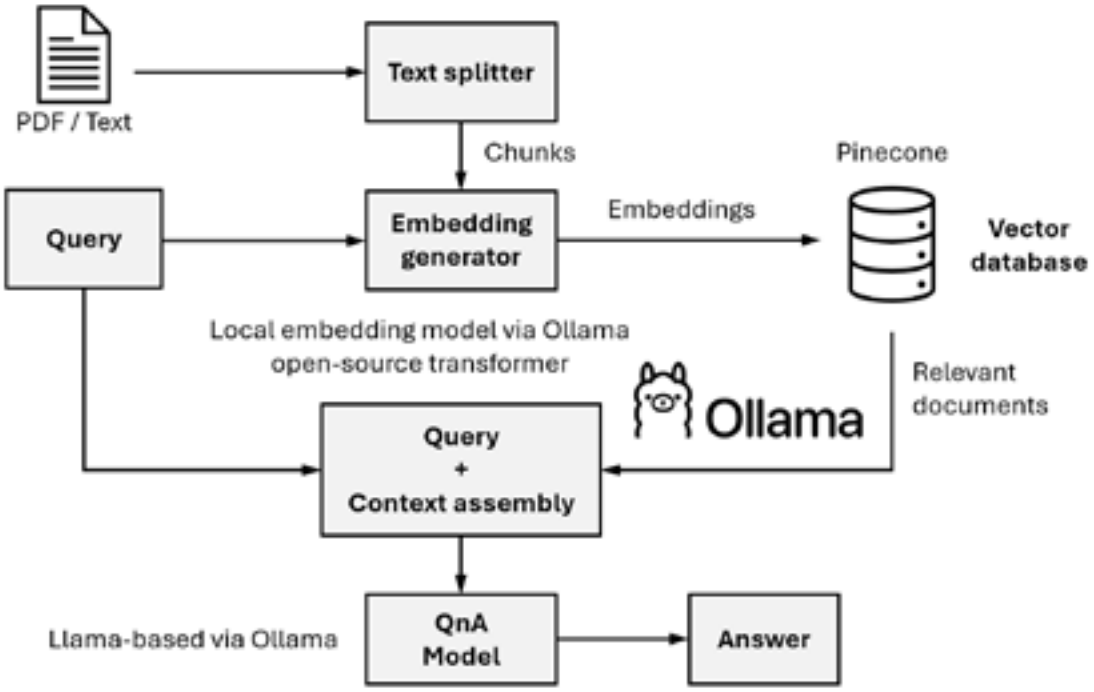
A central component of our approach is the Ollama framework, which provides a structured process for data ingestion, model training, and deployment. Ollama offers a command-line interface (CLI) that simplifies the tasks of gathering, cleaning, and organizing text data prior to training. With support for GPU acceleration, Ollama enables faster and more efficient model training, reducing convergence times by up to 40% compared to traditional training pipelines. It also includes tools for tracking different model versions, allowing developers to easily iterate on improvements. The modular design of Ollama supports separate pipelines for data preparation, model evaluation, and deployment, which can be tailored to meet the specific needs of NIRS related projects.

The model-building process started with a pre-trained Llama 3.21 model, using its 13 billion parameters to capture diverse linguistic and contextual nuances. This model was fine-tuned on 1.2 GB of book data, covering various aspects of NIRS to establish a strong foundation encompassing both fundamental theories and advanced methods. Subsequently, approximately 500 MB of additional domain-specific texts were integrated to refine the model's understanding of practical and technical topics. Ollama's modular workflows facilitated this process, to ensure that data preparation, evaluation, and deployment pipelines were seamlessly customized for NIRS-related tasks.

To accelerate training, we utilized three NVIDIA L40 GPUs, each equipped with 48 GB of GDDR6 memory, 18,432 CUDA cores, and a memory bandwidth of 1,536 GB/s. This high-performance hardware enables processing of larger datasets and efficient optimization of the model. Using the L40 GPUs, we reduced training convergence times significantly, allowing frequent updates to incorporate feedback from domain experts. These GPUs, specifically designed for AI workloads, ensured optimal utilization of computational resources while maintaining low power consumption.

During preprocessing, expert knowledge selection was applied to ensure that text sources were relevant and reliable. Guided by Ollama's built-in tools, this step eliminated irrelevant or ambiguous data and emphasized key terms, concepts, and relationships typical of NIRS literature. The improved data quality is reflected in the model's ability to maintain context in conversations and provide precise insights into NIRS procedures, analytical techniques, and best practices.

The final DSLM is lightweight, requiring less than 4 GB of memory for deployment, making it suitable for resource-constrained systems. It supports interactive learning by offering answers at varying levels of detail, whether users require high-level summaries or in-depth technical discussions. This versatility makes the model valuable in settings such as classrooms, small laboratories, and remote field operations. <FILE



ACKNOLEDGEMENTS: The development of this domain-specific language model was conducted mainly for educational purposes, aiming to enrich the learning and research opportunities for students and faculty at Mälardalen University.

Keywords
Domain-specific language model,Ollama framework,Fine-tuning,GPU-accelerated training,Near-infrared spectroscopy

P01.08.63

YOLOV8 MODEL FOR HYPERSPECTRAL CORE ANALYSIS

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Hyperspectral imaging (HSI) is a transformative tool in geological research, facilitating the analysis of drill core samples required for resource exploration and environmental studies. Despite its great potential, massive data volumes and complex processing workflows pose significant barriers to its routine application. To overcome these challenges, we developed a deep learning-driven framework that improves hyperspectral core analysis.

The core of the framework is an enhanced YOLOv8 model that enables automatic detection and precise localization of core storage boxes. Specifically, we adopted an enhanced YOLOv8 model, replaced the traditional C2F layer with a new Cross-Stage Partial Path Convolution (CSPPC) layer to optimize computational efficiency and gradient transfer, and introduced a Sigmoid Intersection-over-Union (SIOU) loss function to overcome the shortcomings of traditional bounding box regression, thereby significantly improving the model's convergence speed and detection accuracy. The model was evaluated using more than 900 hyperspectral images from multiple datasets, containing a variety of core storage box materials such as volume, metal, and plastic. Experimental results show that the enhanced YOLOv8 model has an average accuracy (mAP@0.5) of 99.5% and an F1 score of 0.999 at a 0.5 IoU threshold, which is higher than the standard YOLOv8. This work provides a solid technical foundation for the automated processing of hyperspectral core scanning, greatly reduces manual intervention, speeds up the geological data processing process, and promotes the widespread application of hyperspectral imaging technology in the field of earth science.

ACKNOLEDGEMENTS: The Geological Survey Ireland and University College Dublin supported this work. We gratefully acknowledge the Geological Survey Ireland for providing the drill core datasets and the University College Dublin for their support. Special thanks are extended to all colleagues and technical staff who contributed to developing and testing the enhanced YOLOv8 model and the overall hyperspectral core scanning framework.

Keywords
Deep Learning,Hyperspectral Core Analysis,YOLOv8,Object Detection,Geological Exploration

P01.08.64

AUTONOMOUS UAV SYSTEM FOR SUSTAINABLE VITICULTURE: INTEGRATING MINIATURIZED SENSORS AND MACHINE LEARNING FOR PRECISION MONITORING

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Recent advancements in proximal sensing for sustainable viticulture emphasize the integration of autonomous technologies, such as unmanned aerial vehicles (UAVs), with miniaturized sensors and machine learning algorithms to enhance the precision and efficiency of vineyard monitoring. These innovations aim to improve agricultural sustainability by optimizing resource use, reducing environmental impact, and providing real-time insights into vine health and grape ripening conditions.

This study is part of the COLIBRI' project (Collaborative integration of mini-UAV, miniaturized sensors, and machine learning for proximal sensing towards sustainable viticulture), which focuses on developing an autonomous UAV system designed to automate vineyard monitoring. The idea is to develop a drone equipped with sensor technologies, including a thermal camera to evaluate leaf water stress and a hyperspectral camera to detect anomalies in grape ripening. By integrating these advanced sensing capabilities, the system enables vineyard managers to assess plant health, optimize irrigation strategies, and predict harvest quality with unprecedented accuracy.

The UAV system is equipped onboard companion computer (Raspberry Pi), for real-time data analysis using machine learning models. This computational framework will facilitate instant classification of ripening conditions and stress levels, empowering decision-makers with timely and actionable information.

At the moment, the UAV operates autonomously, executing assigned for future monitoring missions with high reliability. It is equipped with a flight controller to ensure stability and regulate engine functions, while the companion computer manages onboard software and issues control commands. The system is complemented by a ground station, which will enable remote telemetry monitoring and mission data uploads. For precise navigation and object detection, the drone integrates a GPS receiver and an artificial vision system based on Aruco fiducial markers and the OpenCV2 library. Initial test flights have demonstrated the reliability and efficiency of the system, confirming its potential for further refinements and future applications in precision viticulture and sustainable agricultural management

ACKNOLEDGEMENTS: The authors would like to thank the University of Milan for funding the COLIBRI' project "Collaborative integration of mini-UAV, miniaturised sensors, and machine learning for proximal sensing towards sustainable viticulture" through the competitive call MyFirstSeedGrant.

Keywords
Drone,Optical Sensors,Machine Learning,Grape quality,Water status

P01.08.65

ENSURING OLIVE OIL QUALITY AND INTEGRITY WITH PORTABLE NEAR-INFRARED SPECTROSCOPY AND OPEN-SOURCE TOOLS FOR A GLOBAL MARKET

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The authentication and quality assurance of olive oils (OOs) are critical challenges in the global market. While international standards used in official inspections of producer and importer countries mandate rigorous testing of physicochemical and sensory parameters for OOs, the current official analytical methods are expensive and time-consuming, leading to a low percentage of inspected samples relative to total production. To address these limitations, the development of fast, economical, and portable analytical techniques is urgently needed to safeguard authenticity and strengthen consumer confidence [1]. Near-infrared spectroscopy (NIRS) has emerged as an effective and efficient tool for olive oil analysis, offering a promising alternative to traditional methods [2,3]. Over the last decade, significant advancements have been made in miniaturising NIRS instruments, enhancing their portability and ease of use. This study evaluates the potential of two portable NIRS instruments –MicroNIR Pro 1700 (908-1676 nm) and AlbaNIT (950-1650 nm)– for the on-site analysis of olive oils. A comprehensive dataset of 802 OO samples from different commercial categories (extra virgin, virgin, and lampante) was analysed in transmission mode. Both quantitative and qualitative models were developed using open-source Python software to predict the organoleptic characteristics (fruity, bitter, pungent, and median of defect) of OOs, enabling the determination of their commercial category. The quantitative models developed using these portable NIRS devices showed residual predictive deviation for cross-validation (RPDcv) values for the different sensory parameters tested ranged from 1.86 to 2.09. Moreover, qualitative models correctly classified more than 80% of the samples into their respective categories in validation. These findings underscore the potential of portable NIRS technologies, combined with the accessibility of open-source tools, for small and medium-sized producers. By enabling cost-effective and rapid on-site evaluations, these sensors make access more equitable to advanced quality control methods, enhancing traceability, strengthening consumer trust, and promoting authenticity in the global olive oil market.

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ACKNOLEDGEMENTS: This research was funded by the projects entitled 'NIRS technology and IoT platforms used to ensure the integrity of high-added value Spanish products: Iberian cured ham and Extra-virgin olive oil' (PID2019-111387RB-I00), 'NIROLEO: Innovation in olive oil quality control in olive mills using NIRS sensors' (GOPO-CO-20-0002) and 'Digitisation strategies based on NIRS sensors for the authentication of olive oil and the characterisation of its nutritional properties (AuthenOleo)' (GOPO-CO-23-0005). It is also part of the research work designed in the first author's doctoral thesis. The authors wish to thank all the technical support staff involved in this research for their collaboration and dedication.

Keywords
Olive Oil quality,portable NIRS sensors,cost-effective analysis,authentication,consumer confidence

P01.08.66

IMPLEMENTATION OF A PORTABLE NIR SPECTROMETER FOR AUTHENTICATING QUALITY IN VACUUM-PACKED AND UNPACKAGED SLICED IBERIAN HAM

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The adoption of rapid and non-destructive technologies such as Near-Infrared (NIR) spectroscopy has revolutionised quality control and authentication processes in the food industry, particularly for high-value products like Iberian ham [1-3]. This study explores the potential of a new-generation portable NIR device, the MicroNIR™ Pro 1700, for in situ analysis in static mode (punctual readings) of vacuum-packed and unpackaged sliced Iberian ham. A total of 232 sliced Iberian ham samples were used to develop models for the prediction of the major fatty acids (FA) [4] using MPLS regression, while 259 sliced Iberian ham samples were used for classification purposes to distinguish between premium (acorn-fed) and non-premium (feed-fed) samples. For the unpackaged samples, the best models achieved residual predictive deviation for cross-validation (RPDcv) values of 1.39, 1.25, and 1.35 for palmitic, stearic, and oleic acids, respectively. For the vacuum-packed product, the RPDcv values for the three fatty acids studied ranged between 1.23 and 1.33. Models developed using Partial Least Squares 2-Discriminant Analysis (PLS2-DA) successfully classified 100 % of the samples by feeding regime in validation, in both vacuum-packed and unpackaged conditions. Therefore, the models developed to predict FA showed a limited predictive capacity, being necessary to increase the variability of the calibration set. However, the qualitative approach to classify the product into commercial categories highlights the potential of this new portable NIR sensor, not only facilitates real-time quality control and authentication directly through packaging, maintaining product integrity and hygiene, but also enhances traceability and transparency within the Iberian ham supply chain, offering consumers additional assurances about the product they consume.

ACKNOLEDGEMENTS: This research was funded by the project entitled 'NIRS technology and IoT platforms used to ensure the integrity of high-added value Spanish products: Iberian cured ham and Extra-virgin olive oil' [PID2019-111387RB-I00], and by the project 'Digitalisation of the authentication and labelling systems in the Iberian ham production chain, using NIRS sensors and artificial intelligence' [PID2023-152903OB-I00]. The authors wish to thank all the technical support staff involved in this research for their collaboration and dedication.

Keywords
Sliced Iberian ham,In-situ analysis,New-generation NIR sensor,Quality authentication,Commercial categories

IMAGING & REMOTE SENSING

P01.09.67

DISCRIMINATION OF MAN-MADE VITREOUS FIBRES AND ASBESTOS FIBRES USING HYPERSPECTRAL IMAGING

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Man-made vitreous fibres (MMVFs) are synthetic materials made from processed rock, glass, or minerals, characterized by non-crystalline inorganic silicates with a length-to-width ratio of at least 3:1. Depending on their chemical and geometrical properties, particularly their respirable characteristics critical for lung penetration, MMVFs can be classified as either hazardous or non-hazardous [1]. These fibres have widespread use in thermal and acoustic insulation as well as fire protection. However, distinguishing MMVFs from hazardous asbestos fibres remains a significant challenge, especially during building demolition. This study aims to evaluate hyperspectral imaging (HSI) as a non-destructive and non-invasive method to classify and predict the presence of asbestos within MMVF samples, providing a rapid, efficient alternative to traditional methods.

The study employed hyperspectral imaging (HSI) to analyse mixed samples containing known percentages of asbestos and MMVF. Hyperspectral data were processed using chemometric techniques, specifically principal component analysis (PCA), to develop a predictive model for asbestos and MMVF's identification [2]. This job will show the preliminary analytical results obtained using HSI integrated by PCA and traditional SEM images coupled with EDS on the same samples for distinguishing both asbestos and MMVF's fibre and to compare their data.

Unlike traditional scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS), which requires meticulous sample preparation and skilled personnel [3], HSI integrates imaging and spectroscopy to facilitate the rapid identification and spatial mapping of MMVF and asbestos components without manipulating the sample.

The application of hyperspectral imaging demonstrated its capability to accurately and efficiently classify MMVF and asbestos components. By using PCA in the chemometric analysis, the method showed promise in predicting asbestos presence within mixed samples, effectively distinguishing hazardous elements.

Hyperspectral imaging presents a reliable, non-invasive, and preparation-free alternative to traditional SEM-EDS techniques for MMVF analysis. This approach not only reduces analysis time and exposure risks but also enhances the rapid and accurate characterization of fibrous materials. The findings support the potential of HSI to improve waste management practices and address the environmental and human health risks associated with hazardous materials in demolition and construction waste.

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ACKNOLEDGEMENTS: The study was developed in the framework of the collaboration between INAIL (National Institute for Insurance against Accidents at Work) and Sapienza University of Rome.

Keywords
Hyperspectral imaging,Man-made vitreous fibres,Spectroscopic analysis,Asbestos,CDW

P01.09.68

POST-HARVEST DETECTION OF IMPURITIES AND CONTAMINANTS IN WHEAT KERNELS USING NEAR-INFRARED HYPERSPECTRAL IMAGING

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Wheat grain purity is an important criterion of quality impacting price, storage and further processing. Despite going through the usual sorting stages for food uses, harvested kernels may still contain various undesirable impurities and potential contaminants. Conventional assessment methods are generally performed using visual observations and chemical methods which are time consuming and tedious. Moreover, the presence of particles containing toxic compounds such as mycotoxins, ergot sclerotia or datura seeds is strictly regulated in the EU. In recent years, near-infrared hyperspectral imaging (NIR HSI) has shown its potential as an efficient, non-destructive and fast tool for wheat quality evaluation and impurities detection [1,2]. While the use of instrument working in the NIR range below 1700 nm has proved effective for few selected impurities [3], the further NIR range (1000–2500 nm) offers benefits for the detection of a broader range of impurity types [4]. Additionally, linescan technology allows for the analysis of large-scale bulk samples increasing batch representativeness compared to conventional sampling.

For this study, various impurities from discarded sorting fractions of common wheat samples cultivated in Wallonia (Belgium) were gathered and visually identified. This collection consists mainly of foreign weed seeds (e.g., Galium aparine L., Lolium perenne L.), contaminants (e.g., datura seeds, ergot sclerotia, mycotoxins), diseased wheat kernels (e.g., fusarium damaged kernels, bunt-affected kernels), harvest residues (e.g., straw, husk), entomological material and inorganic material (e.g., soil, pebbles). All collected samples were measured with a linescan NIR hyperspectral camera (SWIR model, SPECIM, Finland) working in the spectral range from 1000 to 2500 nm and coupled with a custom-built conveyor belt (RMA Techniek, The Netherlands). The images acquired were used to build spectral libraries of impurities and to develop machine learning classification models (PLS-DA). Specific models were developed for the detection of contaminants of interest while more generic models were made for purity assessment. Wheat samples artificially contaminated with several impurities were then measured and subjected to the different developed models applied in a dichotomic way.

NIR HSI proved to be a promising tool to provide a fast assessment of sample purity. This method allows for an efficient detection of the tested contaminants including hard to notice particles thanks to characteristic spectral differences. Indeed, specific spectral features found above 2000 nm such as lipids bands were of particular interest for contaminants detection. NIR HSI could be used as a preliminary screening step to inform about the need for further cleaning or the implementation of more precise quantification methods in the case of regulated contaminants.

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ACKNOLEDGEMENTS: This work was performed as part of the ValCerWal project funded by the Walloon Recovery Plan (D65-1427). The authors wish to thank the CRA-W teams from the "Crop production" and "Valorization of agricultural products, biomass and wood" units for providing and sorting the samples and from the "Quality and authentication of agricultural products" unit for identifying the impurities and performing the image acquisitions.

Keywords
wheat impurities,contaminants,imaging,NIR,PLS-DA

P01.09.69

AUTHENTICATION OF MEAT PRODUCTS FROM EXTENSIVE REARING SYSTEM BY NIR SPECTROSCOPY, HYPERSPECTRAL IMAGING, AND CHEMOMETRICS

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Rearing system, breed, geographical origin are some of the quality features able to drive consumer's choice in meat and meat products purchase. The same aspects are marketing leverage from the producer's side. However, the compliance of a specific product with the declared - or supposed - quality features is mostly of a documentary nature, resulting in a possible distrust. Indeed, according to the Alert and Cooperation Network, meat and meat products are the third most reported category concerning non-compliance and suspicions fraud notifications, mostly related to faulty labelling or claims [1]. Bearing this framework in mind, both authorities and consumers could profit from the development of rapid, non-destructive solutions to certify meat and meat products with claims of quality. In this context, NIR spectroscopy could represents an efficient analytical solution to evaluate the authenticity of products. As a first step of this study, fat samples (n = 61) from extensively and intensively reared pigs were collected and analysed by NIR spectroscopy using a benchtop instrument. After an exploration of the dataset, an authenticity model for the "extensive" class was developed using DD-SIMCA [2]. The results showed that a model based on 4 PCs preprocessed using SNV allowed to reach perfect sensitivity and specificity by using only the fat portion in unprocessed meats [3]. However, when moving to processed products, minced and fermented such as ripened sausages, the things start to get complicated. Indeed, these products have usually highly non-uniform surfaces, with both fat and lean portions irregularly mixed and the analysis of the fat portion by single spot NIRS is often technically impossible. In the attempt to overcome this issue, as a second step of the work, the applicability of NIR HyperSpectral Imaging (HSI) to authenticate more complex products such as fermented cured meats was evaluated. Samples (n = 12) from extensive and intensive farming were collected and hyperspectral images of samples slices collected. Three distinct images of the samples were acquired. After background removal and data exploration, K-means clustering was used to separate the pixels of each sample into two clusters, corresponding to the fat and lean fraction. Subsequently, the NIR signal of the fat fraction of each salami was averaged, obtaining an average spectrum for each sample (12 x 140 matrix). The same approach was followed for the three images, obtaining a final matrix of 36 x 140. Preliminarily, an exploratory analysis was carried out by PCA (Principal Component Analysis). By means of PCA, a distribution of samples along PC1 according to the rearing system was highlighted in the score plot. Subsequently, SIMCA and PLS-DA models were built, showing promising errors in cross-validation, suggesting the potential of this technology. Nonetheless, the need to improve the numerosity of the dataset emerged.

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ACKNOLEDGEMENTS: This research was carried out within the Agritech National Research Center and received funding from the European Union Next-GenerationEU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR)–MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.4–D.D. 1032 17/06/2022, CN00000022). This manuscript reflects only the authors' views and opinions, neither the European Union nor the European Commission can be considered responsible for them.

Keywords
Authenticity,Fraud,Classification,Imaging

P01.09.70

A LOW-SWAP DRONE-INTEGRATED ADAPTIVE SPECTRAL SENSING SYSTEM

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We present a novel drone-integrated spectral imaging system that uniquely combines discrete wavelength sensors and a MEMS tunable near-infrared (NIR) spectrometer module, fabricated using standard semiconductor technology. This innovative approach enables precise, scalable, and cost-effective solutions for airborne applications in precision agriculture, remote sensing, and environmental monitoring.

The system incorporates a MEMS tunable optical filter integrated with an InGaAs photodiode, leveraging standard semiconductor wafer-level fabrication processes for volume production and affordability. The system design allows a flexible combination of discrete wavelengths and MEMS tunable microspectrometers to meet diverse application needs. This flexibility enables many configurations of sensor wavelengths, optimizing costs for specific applications. For the proof-of-concept (POC) instrument, discrete visible wavelengths were selected at 530 nm, 570 nm, 660 nm, 705nm, and 850 nm to cover key vegetation indices such as PRI, NDVI, and NDWI. A tunable MEMS NIR spectrometer (825–1700 nm, FWHM optical resolution of 20 nm) further enhances versatility, making the system adaptable to a wide range of scenarios.

The airborne imaging system features a whiskbroom scanner with a reflecting mirror and a single refracting plano-convex lens (focal length 100 mm), achieving a 1 m ground resolution at 100 m altitude with a 6° field of view. Designed as a unified low-SWaP product, the drone and instrument share GPS, IMU data, and power from a single battery module, enhancing flight efficiency and reducing operational costs.

Weighing under 500 g and measuring 120 mm in length and 65 mm in diameter, the sensor system is lightweight, portable, and well-suited for integration into drone platforms. Field trials have demonstrated the system's reliability and versatility across various applications.

This innovative design enables a turn-key solution for drone-based spectral imaging, eliminating third-party integration costs while delivering a highly affordable product. . By offering a flexible combination of discrete sensors and MEMS tunable technology, this system sets a new benchmark for scalable, precision-driven remote sensing solutions.

Keywords
Hyperspectral Imaging,NIR Spectroscopy,Precision Agriculture,MEM Spectrometer

P01.09.71

CLASSIFICATION OF TEXTURIZED VEGETABLE PROTEIN INGREDIENTS USING HYPERSPECTRAL AND MULTISPECTRAL IMAGING AND CHEMOMETRICS: DETECTION OF ALLERGENIC SUNFLOWER PROTEINS

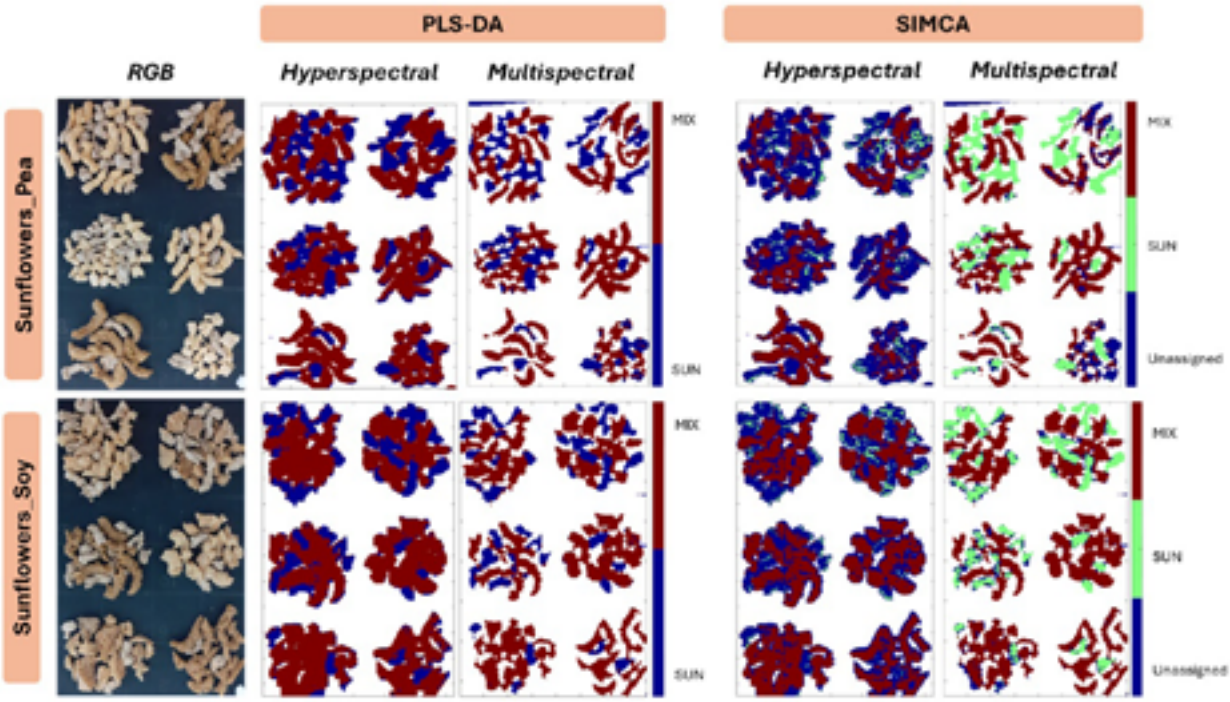
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In recent years, plant-based meat-like products (PBM) have gained prominence as sustainable alternatives to animal meat. PBM main ingredients are texturized vegetable proteins (TVP) that can derive from different sources. Some of them, like soy proteins have allergenic potential. In this study, two imaging systems were employed: a hyperspectral camera (900–1600 nm), which provides a detailed characterization of the optical and chemical properties of the samples, and a multispectral camera (340–1100 nm), which enables faster and more efficient data acquisition but with lower spectral resolution. Classification models were developed using Soft Independent Modeling of Class Analogy (SIMCA) and Partial Least Squares Discriminant Analysis (PLS-DA) to differentiate different types of TVP, with an emphasis on those made from sunflower seed protein due to its potential allergenicity (Taylor et al., 2021).

Two classes of TVP were considered: (1) sunflower and (2) a combination of soy and pea, both dry and rehydrated. SIMCA and PLS-DA models, built using hyperspectral data, showed error rates of 0% and 4-5%, respectively. In validation, SIMCA demonstrated high sensitivity, specificity, and precision, achieving 100% accuracy in the classification of both dry and wet TVP using multispectral images. These results highlight the potential of spectroscopy, combined with chemometric tools, for the efficient classification of TVP. The application of this approach contributes to the development of quality control and food safety systems, providing key information for the industry and consumer protection.

Taylor, S. L., Marsh, J. T., Koppelman, S. J., Kabourek, J. L., Johnson, P. E., & Baumert, J. L. (2021). A perspective on pea allergy and pea allergens. Trends in Food Science & Technology, 116, 186–198. https://doi.org/10.1016/j.tifs.2021.07.017



ACKNOLEDGEMENTS: The authors would like to thank the following projects: ANID national doctoral scholarship n° 21221771, Chile. Dirección de postgrado Universidad de Concepción.

Keywords
texturized vegetable proteins,Allergenic Sunflower Proteins,Hyperspectral spectroscopy,Multispectral spectroscopy,Classification models

P01.09.72

EARLY DETECTION OF LEAF BLOTCH DISEASE IN WHEAT USING HYPERSPECTRAL IMAGING AND ONE-CLASS MODELLING

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Wheat (Triticum turgidum L.) is the second most widely cultivated cereal and a key staple in many regions. However, it is highly susceptible to Leaf blotch (Lb) disease, caused by the fungal pathogen Zymoseptoria tritici, which threatens yield and quality. Thus, early disease detection is crucial for effective management strategies, such as disease control, yield improvement and agriculture cost reduction. However, traditional diagnostic methods are often subjective, time-consuming, and labor-intensive. To limit disease spread, economic losses, and chemical use, innovative techniques like hyperspectral imaging (HSI) have been developed. HSI is a rapid, cost-effective, and non-destructive technique, collecting both spectral and spatial information from samples, enabling early disease detection in plants.

The present study investigates the use of HSI combined with a supervised classification method to detect Lb infection in wheat leaves before visible symptoms appear. A hyperspectral dataset of infected and non-infected wheat leaves acquired in the visible-near infrared (VNIR) spectral range [1] was used. Measurements were performed over 18 time points, from one day before inoculation to 20 days post-inoculation. A total of 588 hyperspectral images of wheat leaves provided spectral data at the pixel scale, capturing both visual and spectral changes associated with the onset of Lb symptoms.

For classification, a one-class modelling approach was employed to distinguish non-infected and infected leaves by capturing significant spectral differences before symptom development. Soft Independent Modelling of Class Analogy (SIMCA), known for its flexibility and applicability, was applied using a 'rigorous' approach [2]. Such one-class modelling methods define a boundary around a target class in the variable space, identifying regions where class members are most likely to be found. To determine the best classification strategy of the samples, four modelling approaches were compared:

(i) independent SIMCA models for each date,

(ii) a single SIMCA model including all dates,

(iii) a moving window approach across dates, and

(iv) a cumulative approach progressively integrating new dates.

The classification models yielded promising prediction performances, with variations in accuracy depending on the modelling strategy. The cumulative modelling approach demonstrated the best ability to classify non-infected leaves while rejecting the non-targeted ones at an early infection stage. This indicates that integrating temporal spectral information enhances classification performance by monitoring Lb disease symptom emergence.

This study confirms the potential of HSI for the early detection of Lb disease in wheat leaves. SIMCA proved to be an effective method for developing classification models based on daily infection status using temporal spectral data. The acquired hyperspectral images offer valuable insights for tracking fungal disease progression over time and improving precision agriculture strategies.

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ACKNOLEDGEMENTS: This work was supported by the Convergences Digital Agriculture Institute ("DigitAg") under the reference 16-CON-0004.

Keywords
hyperspectral imaging,VNIR,class modelling,plant disease detection,wheat

P01.09.73

NIR AND MAPPING IR STUDY ON AMYLOSE-AMYLOPECTIN MIXTURE FOR EVALUATION OF HYDRATION OF STARCHES

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The NIR and mapping IR spectroscopy and Chemometrics analysis for mixture of amylose and amylopectin in various ratios have been investigated. Since the molecular structural changes for gelatinization and retrogression of starch are very complicated, the basic study on hydration mechanisms of molecular components of starch is important. Therefore, we focused on the various ratios of mixtures for commercial reagents of amylose and amylopectin.

The mixtures of amylose and amylopectin were heated for gelatinization with water. NIR spectra and mapping IR of the samples for gelatinization states were measured.

The obtained NIR and IR spectra varied during the retrogression process. The intensity of a band at 4770 cm-1 in NIR spectra, assigned to combination of OH stretching and bending modes of the amylopectin molecule, is decreasing with progression of rehydration. The feature of IR spectra about 1000 cm-1 also suggests that the gelatinization of starch is proceed and molecular structure has been changed in this process. In the mapping IR, the spectral changes are vary by starch granules suggest that the gelatinization process proceeds gradually.

Thus, the NIR and IR technique, including mapping and chemometrics, are useful for hydration structural study of starches.

Keywords
Starch,Rehydration,Molecular Structure,Gelatinization,Mapping IR

P01.09.74

PLASTIC MATERIAL DISCRIMINATION USING PIXEL-WISE STANDARD DEVIATION OF SPECTRAL INTENSITY IN HYPERSPECTRAL IMAGING

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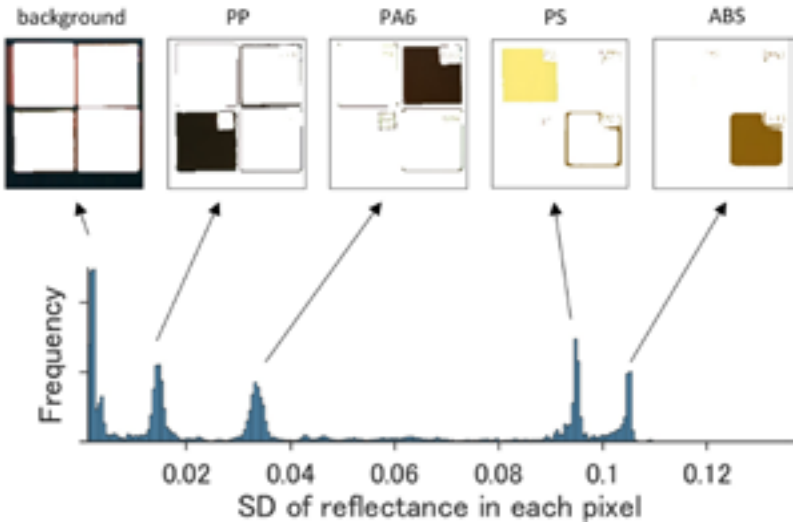
The pixel-wise standard deviation of spectral intensity (px-wise SD method) was previously proposed for hyperspectral image segmentation [1], and its effectiveness in identifying and removing background pixels was reported [2]. The px-wise SD method is also applicable to the segmentation of non-background regions, and can be used for discrimination by appropriately setting the thresholds. This paper reports the application of the px-wise SD method for plastic discrimination.

The test specimens (50×50×2 mm) were polystyrene (PS), nylon 6 (PA6), polypropylene (PP), and acrylonitrile-butadiene-styrene (ABS). The specimens were placed on a low-reflective sheet, and diffuse reflectance spectra were obtained in the range 950–1650 nm at 5 nm intervals using a near-infrared (NIR) hyperspectral camera. Spectral analysis was performed using MATLAB software without spectral pre-processing, and the SD of the reflectance was calculated for each pixel.

For the hyperspectral images, the px-wise SD of the reflectance was calculated over the wavelength range 950–1650 nm at 5 nm intervals. A histogram was then created, revealing 5 peaks within the SD range 0–0.1. When thresholds are set between these peaks, the peaks correspond to the background (low-reflective sheet), PP, PA6, PS, and ABS in increasing order of SD. This result demonstrates that plastic classification is feasible based on the px-wise SD of reflectance without multivariate analysis. In addition, when the wavelength range for calculating the standard deviation is changed to 1050–1250 nm, the peak positions of the SDs for ABS and PS are reversed. Nevertheless, plastics can still be distinguished. Furthermore, the classification was achievable even when the wavelength interval increases from 5 to 40 nm in the 1050–1250 nm region. This method offers the advantage of low computational load without multivariate analysis. Additionally, it can be applied to spectra obtained with conventional spectrometers and in fields beyond hyperspectral imaging

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ACKNOLEDGEMENTS: This research was supported by JSPS KAKENHI (Grant Number: JP23K18068).

Keywords
NIR,hyperspectral imaging,plastic discrimination,pixel-wise standard deviation

P01.09.75

INVESTIGATING THE PENETRATION DEPTH OF NIR RADIATION THROUGH HYPERSPECTRAL IMAGING AND CHEMOMETRICS

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The two-year research project INSIDE, funded by the Italian Ministry of Universities and Research – MUR, investigates the penetration depth of incident near-infrared (NIR) radiation coupled with hyperspectral imaging (HSI) to identify the depth reached by this radiation, depending on materials analysed and experimental settings.

To this aim, ad hoc samples in the shape of 1×1×1cm and 1×1×3cm (square base × height) parallelepipeds were designed and built employing a multi-material 3D printer, resulting in specimens composed with strata of different thickness of polymers with distinct spectral signatures. In particular, glucose-modified polyethylene terephthalate (PETG) and polylactic acid (PLA), were employed to produce biomaterial columns with different proportions of the two plastics, as represented in Figure 1.

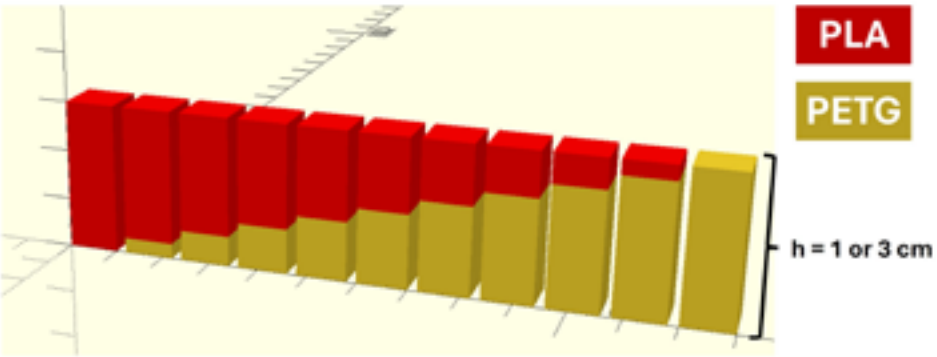


Figure 1: graphical representation of the column samples used in the study. The step was either 0.5mm when h = 1cm, or 2mm when h = 3cm. The number of columns in the image does not correspond to the reality, wich was n = 21 columns when h = 1cm and n = 16 when h = 3cm.

The samples were then analysed via a NIR-HSI camera (Specim, Finland) with either polymer facing the objective, and the obtained spectra were used for data analysis.

The obtained data matrices underwent chemometric analysis in the Python and Matlab® environments, using both PLS Toolbox (Eigenvector Research Incorporated, Washington) and in-house scripts. Exploratory analysis through principal component analysis was exploited to observe the penetration within different materials, which resulted in being non-linear, and ordinary least squares were then applied to deconvolute the spectral signatures of the two components, providing an unsupervised quantification of each polymer in each pixel.

Subsequently, the amount of different components in terms of height in mm of the considered polymer was quantified, and the precision of the quantification in terms of root mean square error in prediction (RMSEP) was investigated, both in relation to the type of polymer facing the objective and the algorithm used to reach the quantification.

In particular, partial least squares regression (PLS) was initially carried out on the mean spectrum of each column, and the height of either PLA and PETG was predicted, with a number of latent variables varying between 4 and 5, depending on the polymer, and an RMSEP ~ 1 mm (10% of total range). Then, regression by means of convolutional neural networks (CNN) was performed on the full image, with an RMSEP ~ 0.5 mm (i.e., 5% of total range). The better performances obtained using CNN were ascribable to the ability of this method to better account for non-linear behaviours. Surprisingly, RMSEP was not significantly different between the two polymers, thus suggesting that NIR radiation can penetrate for at least 1 cm into both PETG and PLA, and in general, proving its potential for being exploited as a 3D technique.

ACKNOLEDGEMENTS: Financial support provided by the Italian Ministry of Universities and Research – MUR (Research Project PRIN 2022 n. 20223WBTH8, CUP: D53D23008950006) is gratefully acknowledged.

Keywords
Penetration depth,Hyperspectral imaging,Artificial neural networks,PLS regression,Multimaterial sample

P01.09.76

BENEFITS OF SPARSITY IN THE SPATIAL DIRECTION FOR THE ANALYSIS OF NIR HYPERSPECTRAL IMAGES

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Near Infrared Hyperspectral imaging (NIR-HSI) couples the advantages of classical NIR spectroscopy with the possibility of representing the spatial variability of spectral data within an image, allowing the visualization of the chemical distribution of the sample surface. Despite the great potential of this technique, data-richness is a double-edge sword: on the one hand, a large amount of data permits a detailed representation of the analysed samples but, on the other hand, it negatively affects data handling, analysis and storage [1, 2].

Multivariate Image Analysis is mandatory to extract relevant information from hyperspectral data. Due to the large amount of data contained in HSI, pixel-level data reduction is often performed by selecting only the pixels that are most representative of the variability of the acquired samples.

In this study, we evaluated the benefits of applying sparse Principal Component Analysis (sPCA) with sparsity in the spatial direction to perform spatial features selection on NIR-HSI data. sPCA is an extension of classical PCA where the sparsity constraint is applied to the model coefficients (i.e., loadings and/or scores) [3, 4].

To extract meaningful spatial features from the images (i.e., pixels), sparsity was applied to the score vectors using the Least Absolute Shrinkage and Selector Operator (LASSO). In this manner, only a limited subset of pixels contributes to each principal component, while the score values of noisy or uninformative pixels are forced to be equal to zero [3].

The effectiveness of sPCA was tested considering different well-known datasets with increasing complexity. The first dataset consisted of plastics pellets of different polymers, while the second dataset consisted of stinkbugs on mixed vegetal backgrounds [4, 5]. The elaborations were performed evaluating the impact of different sparsity levels in the final pixel selection, since it represents a key aspect to refine the selection of the Regions Of Interest (ROIs).

The results suggest that spatial feature extraction performed with sPCA can help in reducing redundant information, improving interpretability and eliminating noise. In addition, this approach has a significant impact on image data compression by reducing storage and computational efforts thanks to the selection of the most informative spatial regions.

As a further development, sPCA can be used as an alternative approach to select representative pixels from classes of interest to develop supervised classification models.

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ACKNOLEDGEMENTS: The research was partially supported by the Innovation Fund Denmark under the project HyperSort – Hyperspectral Imaging for Advanced Textile Sorting and Food Control, which contributed to the work of one of the authors.

Keywords
Sparsity,Pixel selection,ROIs selection,Hyperspectral imaging,sPCA

P01.09.77

PART 2: COMPARATIVE SPECTRAL IMAGING FOR IDENTIFICATION AND CLASSIFICATION OF AIR-LIQUID INTERFACE BIOFILMS ON FOOD PROCESSING SURFACES: DATA FUSION ANALYSIS

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Biofilms forming at air-liquid interfaces [1] pose a significant challenge in food processing due to their resilience and difficulty in detection [2;3]. This study explores the potential of spectral imaging techniques—Fourier Transform Infrared (FT-IR), Raman, Short-Wave Infrared (SWIR), and Visible-Near Infrared (VNIR)—for characterizing biofilm formation on food processing surfaces. Five bacterial species (E. coli, B. subtilis, C. sakazakii, P. fluorescens, and L. plantarum) were cultivated on stainless steel (AISI 304 and 316) and aluminum substrates under milk and broth conditions to assess biofilm growth and spectral variability.

This study compares micro and macro spectral imaging systems to evaluate their effectiveness in biofilm detection and classification. PCA revealed that macro-scale spectral imaging (VNIR and SWIR) effectively distinguished biofilm presence, with SWIR outperforming VNIR in biofilm detection and Gram classification. Moreover, for macro-scale imaging, we assessed the impact of analyzing entire surfaces versus applying a masked region specifically targeting the biofilm area. While masking improved classification performance in some cases, particularly on stainless steel, results varied depending on the growth medium and bacterial species.

Additionally, data fusion techniques (SO-PLS-LDA) integrating SWIR, VNIR, FT-IR, and Raman improved classification accuracy. SWIR-VNIR fusion yielded the best performance in bacterial strain identification and Gram classification, reducing variability across test sets and enhancing predictive robustness.

Our findings highlight the complementary potential of micro- and macro-scale imaging approaches, with macro-scale techniques offering a rapid and non-invasive solution for biofilm detection in food processing environments.

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ACKNOLEDGEMENTS: This work was supported by Science Foundation Ireland (SFI) [Proposal ID 15/IA/2984–HyperMicroMacro]. Prof. Aoife Gowen acknowledges funding from European Research Council (ERC) under the 472 starting grant programme ERC-2013-StG call-Proposal No. 335508-BioWater.

The authors gratefully acknowledge Dr Ian Reid (NIMAC-UCD) for the SEM images and Dr Emmanuel J. Ekoi for the optical profilometry analysis.

Keywords
spectral imaging,multi-block,biofilms,chemometrics,food safety

P01.09.78

COMPARISON OF BENCHTOP INFRARED SPECTROSCOPY DEVICES USED TO PREDICT PHYSICAL AND CHEMICAL PROPERTIES IN WHOLE WHEAT

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Wheat (Triticum aestivum L.) is among the most essential cereal crops globally. Assessing the physical and chemical properties of wheat is integral for determining its commercial grading, milling efficiency, and suitability for end-use products. For the plant breeders baking properties are also important when breeding the next generation. Benchtop NIR spectrophotometers have proved itself to be successful in measuring important physicochemical properties of wheat and flour, however, these devices are bulky and expensive and restricted to lab-based analyses. Technology has allowed for the development of handheld NIR devices and with further miniaturisation of these optical components leading to the commercial rise in miniaturised NIR devices. This work evaluated three categories of NIR devices, i.e. benchtop, handheld, and miniature spectrometers with regard to their calibration accuracy, predictive performance, cost-effectiveness, and overall feasibility for wheat physicochemical quality assessment. The instruments differ in wavelength ranges and resolution, and therefore a data fusion approach, combined dimensional PCA, was used to make results between instruments comparable. Calibration models were developed using partial least squares (PLS) regression and standardised laboratory reference methods. Coefficient of determination (R^2) and RMSEP (Root Mean Square Error of Prediction) for model calibration and validation were used as figures of merit. The models were first established using the spectra of wheat samples obtained by the Benchtop NIR device. The reference methods that yielded successful calibrations on the benchtop device were: protein whole wheat (PWW) (0.68% RMSEP, 0.75 R2), protein flour (PF) (0.68% RMSEP, 0.77 R2), Mixograph absorbance (MA) (0.94% RMSEP, 0.75 R2), Farinograph development time (FDT) (3.68 min RMSEP, 0.31 R2), and Hectolitre mass (HM) (1.84 kg/hL RMSEP, 0.65 R2). Model development was subsequently done for the handheld and miniature instruments. The handheld yielded the following results: PWW (0.94 % RMSEP, 0.53 R2), PF (1.01 % RMSEP, 0.51 R2), MA (1.13% RMSEP, 0.63 R2), FDT (3.67 min RMSEP, 0.29 R2), and HM (2.16 kg/hL, RMSEP, 0.46 R2). The miniature device yielded the lowest results: PWW (1.44 % RMSEP, 0.01 R2), PF (1.50 % RMSEP, 0.01 R2), MA (1.91% RMSEP, 0.01 R2), FDT (4.32 min RMSEP, 0.02 R2P), and HM (2.87 kg/hL RMSEP, 0.05 R2).

Variables important in selection (VIP Scores) were utilised to identify important wavelengths. For protein whole wheat content, the benchtop NIR device showed peaks at approximately 1200 nm, 1410 nm, 1675 nm, 1892 nm, 2168 nm, 2270 nm, and 2453 nm compared to the peaks identified from the recorded spectra using the handheld (963 nm, 1162 nm, 1248 nm, 1366 nm, and 1459 nm). Protein or N-H bonds absorb in the ranges 1500-1570 nm and 2050-2300 nm.

While the benchtop instrument maintained a marginally higher accuracy, their cost and lack of portability may limit their adoption in certain field-based scenarios. The handheld NIR device yielded promising predictive performance results for rough screening. The miniature NIR device—though practical and cost-effective for rapid decision-making—yielded the lowest predictive results with few important wavelengths identified due to its low resolution. Further investigation and robustness of optical components are necessary for the theoretical promises of miniature NIR devices to be put into practice.

ACKNOLEDGEMENTS: Department of Food Science, Stellenbosch University, Stellenbosch, South Africa for providing a post-graduate scholarship for 2024.

Keywords
NIR,handheld NIR device,miniature NIR device,whole wheat,PLS Regression

P01.09.79

EXPLORING THE USE OF HYPERESPECTRAL IMAGING FOR DETECTING LOW-DENSITY FOREIGN OBJECTS IN DEHYDRATED BOLETUS POST-HARVESTING

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One of the major challenges the food industry has faced over the years is the presence of foreign objects in finished products [1]. Technologies like metal detectors and X-rays are effective for detecting metallic objects or those with different densities than the product. However, these methods are often not enough. The origin, composition, and size of foreign objects can vary greatly. Depending on the type of foreign object, the food matrices in the final product, and the production process, detection can become very challenging.

This project explored the use of hyperspectral imaging technology to solve a problem for a food company: the detection of foreign objects in four categories of dehydrated boletus mushrooms. In this case, the complexity of the problem lies in the nature of the boletus itself (notable differences between the cap and the stem) and the wide variety of foreign objects of plant origin that are very similar to boletus: dried leaves, twigs, fragments of tree bark, and others. The company has a dedicated room specifically for detecting foreign objects in the product. This is an initial preprocessing step before the product moves to production lines, where variables like conveyor speed, lighting, and camera height are not an issue once optimally set.

The project used the FX17 hyperspectral imaging camera, with a range of 900 to 1700 nm. Fifty samples of dehydrated boletus from the same variety but different suppliers were collected, categorized by size: granular, small, medium, and large. Additionally, the company provided a wide collection of foreign objects from their production and 20 plant-based materials were selected out of them for their similarity to boletus matrix.

The images were processed pixel by pixel, identifying the variety of spectra that make up the matrix of a product or foreign object. Various spectral preprocessing methods were applied, such as Fourier transform, along with linear and non-linear models, neural networks, and combinations of spectral and morphological data.

The best model trained produced excellent results, detecting most of the foreign objects regardless of their size in all the four boletus categories. This shows that hyperspectral imaging is an effective solution for these complicated cases of foreign bodies detection.

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ACKNOLEDGEMENTS: This project has received funding from the European Union's Digital Programme under the grant agreement No 101083411. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union (DGCNECT). Neither the European Union nor the granting authority can be held responsible for them

IRIS EDIH provides services through PADIH thanks to funding from the European Union through the funds of the Recovery. Transformation and Resilience Plan - Nest GenerationEU

Keywords
Hyperespectral imaging,Foreign object,Dehydrated boletus,Morphological,Fourier transform

INDUSTRY & PAT

P01.10.80

MILK COAGULATION MONITORING USING A NIR PROBE (II): IDENTIFICATION OF THE OPTIMAL SPECTRAL RANGE TO PREDICT COAGULATION PROPERTIES

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In the Italian dairy PDO production, there are several traditional cheeses with artisanal production processes where not standardized raw milk is used and milk renneting occur in small vats. In these processes, the success of milk coagulation phase is closely tied to cheesemakers' skills, that based on their personal expertise, determines the coagulation time and the curd cutting time by visual and manual curd firmness evaluation. The absence of standardization can be problematic, particularly regarding the management of curd cutting and syneresis that can affect both cheese yield and final product quality. To support cheesemakers in the management of curd cutting, is possible to perform a real-time milk coagulation monitoring through a method based on NIR spectroscopy. The aim of this work is to identify the optimal spectral range for milk coagulation monitoring to anticipate the prediction of coagulation time, simplifying the implementation of the NIR probe directly in dairy coagulation vat; furthermore, is a preliminary study for the development of low-cost NIR probes with narrowband emission, that will be possible thanks to the increasing availability on the market of light-emitting diodes (LED) with emission peak around specific wavelengths.

Laboratory tests were performed on fourteen coagulation batches obtained using milk skimmed by natural creaming at different acidification levels (obtained adding different volumes of natural whey starter), and different calf rennet concentrations; milk temperature was maintained at 35°C and coagulation was monitored with both mechanical lactodynamograph (Formagraph, Foss Electric, Hillerød, Denmark), as reference method, and a portable NIR spectrometer (MicroNIR, Viavi Solutions, Chandler, AZ, United States) mounted on a food-grade probe, placed in direct contact with 220 ml of coagulating milk. Full NIR spectra (125 wavelengths ranging from 908 to 1676 nm) were recorded continuously with 13.5 seconds interval for more than 30 minutes after coagulant addition. The data acquired are related to the variation of backscattering due to casein micelles aggregation during milk coagulation. Backscattering depends on the ratio between the particle optical diameter and the incident wavelength, when the particle optical diameter is bigger than the wavelength, as the Mie theory predicts, the forward scattering predominates causing an increasing in the pseudo-absorbance in diffuse reflectance; conversely, when the wavelength is comparable to the particle optical diameter, isotropic scattering occurs. Observing the apparent absorbance curve during coagulation, is possible to find an inflection point due to a change in backscattering related with coagulation starts; this point can be easily identified finding the minimum of the first derivative. This behaviour, that affect absorbance at each wavelength, was investigated. The spectra acquired were processed to remove the absorption of milk macro-constituents, specifically water, fat, protein, and lactose, highlighting the scattering variation associated with milk coagulation process. The data obtained were used to calculate the minimum of first derivative for coagulation time prediction at each wavelength. Longer wavelengths are associated with an increase in signal due to greater backscattering compared to shorter wavelengths. In this regard, 1601 nm band was used achieving a coagulation time prediction 1.38 ± 0.68 minutes in advance compared to 1304 nm wavelength, maintaining a comparable correlation (R^2 0.868) with the reference data (LDG r parameter).

Keywords
Milk coagulation,Near-infrared spectroscopy,On-line monitoring

P01.10.81

REAL-CASE IMPLEMENTATION OF ON SITE NEAR INFRARED SPECTROSCOPY SENSORS FOR SELF-CONTROL IN AN ANIMAL RENDERING PLANT

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Renderers convert deceased animals and animal by-products into high-quality animal-based feed ingredients. This recycling process is carried out by the rendering industry, yielding Processed Animal Proteins (PAPs) and feed-grade animal fats. However, the use of PAPs has been significantly restricted due to regulations implemented since 2001 in the fight to eradicate Bovine Spongiform Encephalopathy (BSE) [1]. Since then, various projects and publications have demonstrated the key role of Near-Infrared Spectroscopy (NIRS) in ensuring the production of high-protein PAPs and compliance with current regulations. Nevertheless, the transition from research findings to real-world industrial applications is far from straightforward. This process involves overcoming substantial scientific and technical challenges to adapt commercially available NIRS devices to the existing infrastructure of rendering plants. End-to-end implementations, including both at-line and on-site applications, are particularly valuable, as they enable comprehensive quality control across the entire process – from raw material receipt to production and final shipment. This ensures that both purchased raw materials and finished PAPs meet customer specifications and regulatory standards [2].

This work aims to present a successful case study of the implementation of several NIRS devices for self-control at critical points in the rendering process. The main scientific and technological tasks addressed through over 20 years of close University-Industry collaboration will be summarized. The focus will be on the current deployment of an on-site multiplexed instrument (monitoring three distinct production lines) and a portable device in use for the PAP prediction of moisture, crude protein, ash, and fat content. Special attention will be given to the protocols developed for installing, transferring, and monitoring the predictive models at each control point, as well as the integration of the system into the industry for ongoing quality assessment.

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ACKNOLEDGEMENTS: The authors are grateful to the R & D & I project "Digitization systems for self-control in the production of Processed Animal Proteins (PATs) using NIRS Technology and Artificial Intelligence" funded by (CTA). The authors wish to thank M. Carmen Fernández for their technical assistance.

Keywords
SELF-CONTROL,ON-SITE SENSORS,PROCESSED ANIMAL PROTEINS,ANIMAL RENDERING

P01.10.82

OUT WITH THE OLD AND IN WITH THE NEW: REPLACING INSTRUMENTS IN AN NIR NETWORK

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Near infrared (NIR) instruments are widely used for analytical purposes and have applications in many industrial environments. In many larger businesses focused on processing there are networks of interlinked NIR that share calibrations for optimal efficiency1, 2. The performance of these NIRs is continually monitored remotely covering both the performance of the instruments themselves and how their calibrations are predicting composition when compared to laboratory results.

When the NIR instruments reach the end of their life and must be replaced this involves a two-stage process. The replacement NIR is selected and then all the instruments in the network are replaced. This process is a major capital investment decision for a company, and it is essential to ensuring continuity of performance while also minimising business risk. This paper gives some results from Fonterra's most recent installation and validation of a two new at-line NIR networks for used for dairy product (milk powder, cheese, butter) composition across 40 instruments and 11 production sites. The two NIR systems measure quite different matrices, have different wavelength ranges and one instrument class uses transmission while the other operates in reflectance mode.

The process of NIR replacement involves an initial instrument evaluation to ensure that both the individual instrument performance and stability is well understood and appropriate for the expected use. In addition, we assess that there is a degree of compatibility with existing NIR spectral data/calibrations that can be utilised when developing calibrations for the new instruments.

Once Fonterra's new NIR instruments were installed in production environments, a careful and consistent validation and calibration updating process was carried to prior to routine use. For both new NIR networks this showed up some variations in performance of individual instruments that in turn had an impact on the predictive performance of calibrations for moisture, fat and protein. We present data on how these variations were identified and addressed to create robust predictive calibrations that were deployed across the two new NIR networks.

This initial validation and calibration update has been followed by routine process monitoring to ensure consistent predictive performance for each instrument, and again the assessment of stability albeit over a longer time-period. We contrast the different experiences found when introducing two types of new NIRs for dairy product compositional analysis and the systems and analytics that underpin an effective NIR network replacement project.

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P01.10.83

NEAR INFRARED SPECTROSCOPY AND CHEMOMETRICS FOR WHEAT GLUTEN CLASSIFICATION AND ADULTERANT QUANTIFICATION

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Wheat gluten is an important ingredient in human and animal nutrition. It is used as a fortifier for bread dough, as a vegetarian meat substitute, but also as a filler in animal food. Gluten from different geographic origins and wheat cultivars vary in their protein content and composition. Depending on the protein content, gluten is tailored to specific applications. Generally, high protein content is traditionally associated with superior quality. This parameter is usually determined using nitrogen-based techniques such as Kjeldahl digestion or the Dumas combustion. However, due to the limited specificity of those techniques, melamine and cyanuric acid have been used in the past to adulterate gluten for animal food to mimic and falsify a higher protein content [1]. Therefore, raw material testing is essential to ensure the quality, purity and safety of wheat gluten products used in various applications. In this context, near-infrared spectroscopy (NIRS) offers a simple, rapid, cost-effective, and non-destructive analytical method for predicting the quality parameters of gluten samples [2].

This study investigates the potential of NIRS for the quantification of adulterants such as melamine or cyanuric acid in wheat gluten using partial least squares (PLS) regression models. Evaluation of preliminary results shows a relative prediction error of approx. 5%, indicating a good feasibility of the applied approach. A further goal is the classification of high-quality gluten vs. low-quality gluten as well as adulterated gluten samples. For this purpose, different classification techniques such as PLS-Discriminant Analysis (PLS-DA), statistical method for supervised classification of data (SIMCA) and support vector machine (SVM) will be tested and compared.

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Keywords
NEAR INFRARED SPECTROSCOPY,CHEMOMETRICS,WHEAT GLUTEN CLASSIFICATION,ADULTERANT QUANTIFICATION,partial least squares

P01.10.84

PREDICTION OF POL CONTENT IN SUGARCANE JUICE EXTRACTED BY DIVERSE EXTRACTION TECHNIQUES USING LOCALLY WEIGHTED REGRESSION

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Determining the optimal harvest maturity of sugarcane within a specific geographical region or nation presents an annual challenge for the sugar society. This difficulty arises because farmers and raw sugar factories employ different extraction methods as well as conventional techniques to determine the amount of sugar content. These varying extraction methods yield different sugar content, necessitating the development of individual predictive models for accurate harvest maturity estimation in each region. However, creating multiple models is costly and may affect operation efficiency. Developing a calibration model capable of accommodating diverse extraction methods would enhance measurement accuracy by minimizing sample biases. Non-linear regression is considered more suitable in building such a model compared to linear regression. The objectives of this study were to investigate the pol content in sugarcane juice extracted by direct extraction method (DEM) and shredded extraction method (SEM) by chemical analysis. And to develop a local partial least square (PLS) model using a locally weighted regression (LWR) to predict sugarcane harvest maturity and support harvest decisions. Mature sugarcane billets were collected from farmers' fields and sugar factory demonstration fields in sugarcane-producing regions. Samples collected from each field were evenly divided into DEM and SEM groups and were transferred to the laboratory in a cool box. For DEM samples, 106 non-defective sugarcane samples were directly squeezed using a hydraulic press machine at 25.5 MPa to obtain juice. Similarly, 106 non-defective SEM samples were first shredded using a cutter grinder before being squeezed with a hydraulic press machine at 25.5 MPa. The juice samples were then purified with lead acetate and transflectance spectra data were measured using a benchtop spectrophotometer with a wavelength range of 400-2500 nm. Purposive sampling was conducted, ranking DEM and SEM datasets separately from maximum to minimum pol values. The top three samples were assigned to the training set while the fourth sample was allocated to the test set, giving 75% and 25%, respectively. Furthermore, the DEM and SEM training sets were combined to develop a unified calibration model, which was evaluated using the test sets of DEM, SEM, and their combination (mixed). Models were built using raw spectra data with PLS regression and LWR. The results of the chemical analysis showed that DEM yielded 0.67% higher pol content in sugarcane juice than SEM, with statistical significance at $p<0.05$. The unified calibration models developed using LWR accurately predicted pol content in juice, yielding root mean square error of prediction (RMSEP) of 0.44%, 0.30%, and 0.38% for DEM, SEM, and mixed juice, respectively, In comparison, PLS models yielded RMSEP values of 0.66%, 0.38%, and 0.70%, respectively. These results indicate that the LWR calibration model is more robust for monitoring pol content in sugarcane juice extracted by diverse extraction techniques, thereby supporting harvesting planning. Additionally, farmers and sugar mill technicians should consider the extraction method employed to assess juice content.

ACKNOLEDGEMENTS: The authors express gratitude to the sugar factories in Okinawa and Kagoshima prefectures for providing the sugarcane samples used in this research and to The United Graduate School of Agricultural Sciences, Kagoshima University, for partially funding the research.

Keywords
benchtop spectrophotometer,extraction methods,harvest maturity,locally weighted regression,sugarcane juice

P01.10.85

QUANTITATIVE ANALYSIS OF NEAR-INFRARED SPECTROSCOPIC TECHNIQUES BASED ON MULTI-FEATURE FUSION

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Existing applications of near-infrared spectroscopy using linear or non-linear means for quantitative analysis of protein content in cereals, maize, wheat and milk are not well developed and mostly rely on complex data pre-processing steps. Therefore, this paper proposes a multi-feature fusion-based quantitative analysis model (MFFM) that aims to simplify the data processing process and improve the detection performance.

The MFFM combines a one-dimensional Convolutional Neural Network (CNN) and a Long Short-Term Memory Network (LSTM) in order to achieve regression prediction of protein content in near-infrared spectral data. The model first extracts the spatial features in the dataset through the CNN module, which consists of three branches to extract feature information at different levels. All the extracted spatial features are spliced and fused by the Concatenate function, and then input into the LSTM network for temporal feature extraction to capture the dynamic changes of different channel features. Finally, the output of the last time step of the LSTM is selected and passed to the fully connected layer to generate the final prediction value through nonlinear regression. During the model training process, Batch Normalization Layer (BN Layer) and Dropout structure are introduced to speed up the convergence speed and prevent overfitting during model training. Meanwhile, techniques such as learning rate decay, early stopping strategy, and L2 regularization are employed to ensure the accuracy and robustness of the model.

Near-infrared spectral data of cereals, maize, wheat and milk and their corresponding protein contents were used for the experiments. Four models, Partial Least Squares (PLS), CNN, LSTM and MFFM, were applied to the data under five different preprocessing methods and predicted the protein content of the test set. The results show that the MFFM outperforms the other three models in each sample without preprocessing. The root mean square error (RMSE) of the MFFM in the cereal test set was 2.5027, which was 11%, 0.3% and 80% lower than PLS, CNN and LSTM, respectively; the RMSE in the maize test set was 0.0942, which was 1.92%, 9.59% and 8.34% lower than PLS, CNN and LSTM, respectively; the RMSE in the wheat test set was 0.3943, which was 11.71%, 31.19% and 16.21% lower than PLS, CNN and LSTM, respectively; and RMSE in the milk test set was 0.1538, which was 11.13%, 14.16% and 44.97% lower than PLS, CNN and LSTM, respectively. By comparing the model performance under different preprocessing methods, it is found that the deep learning model has a low dependence on data preprocessing, and there is a large difference in the impact of different preprocessing methods on model performance. The ablation experiments further confirmed the advantages of the MFFM in terms of feature extraction and model complexity, which enabled it to show stronger generalization ability and higher accuracy in the prediction of multiple samples.

The MFFM proposed in this paper enables fast and accurate quantitative analysis of the protein content of four samples, namely cereals, maize, wheat and milk, without relying on data preprocessing. Compared with traditional PLS, CNN and LSTM models, the MFFM has a significant advantage in prediction performance and provides a new solution for quantitative analysis of nutrient content in food and crops by near-infrared spectroscopy.

ACKNOLEDGEMENTS: This research was supported by theDevelopment and Application of MEMS-Based Near-Infrared Sensors (No.2024GH17).

Keywords
near-infrared spectroscopy,feature extraction,multi-feature fusion,MFFM,quantitative analysis

P01.10.86

NIR SPECTROSCOPY FOR THE STUDY OF THE ENVIRONMENTAL VARIABILITY OF COFFEE BEANS

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Coffee is a worldwide significant commodity, with its quality and characteristics influenced by various factors, including environmental conditions [1-2]. This study investigated the chemical variations in coffee beans resulting from different environmental conditions, using Near-Infrared (NIR) spectroscopy [3].

Samples were collected across different production stages in a single year: green coffee beans (June-October), roasted coffee (April-October) and final product, i.e. capsules, (February-October). Both 100% Arabica blends and blends containing Robusta were analyzed.

NIR spectra were acquired using two different spectrometers: one operating from 4000 to 10000 cm-1 for both the green coffee and the final product and the other one operating from 5500 to 12500 cm-1 for the roasted coffee.

Data analysis was performed using chemometric tools [4] such as preprocessing methods and Principal Component Analysis (PCA) on the SNV-corrected and mean-centered data to evaluate spectral variations ascribable to changes in chemical properties. After a global range investigation, specific spectral ranges were analyzed, including common wavelength ranges between the two spectrometers and focused spectral regions containing peaks related to important chemical components, to highlight differences among samples.

The results indicate that environmental variability has no detectable effect on green coffee beans, but it becomes evident in roasted coffee and final product. Further analysis of NIR spectral intervals suggests that variations in lipid, protein and moisture contents are correlated with the industrial process.

Overall, these findings demonstrate the potential of NIR spectroscopy for monitoring and understanding the impact of environmental factors and process parameters on coffee quality during processing.

This approach can enable targeted adjustments to maintain consistent product characteristics.

Acknowledgments: we thank Luigi Lavazza S.p.A. for providing coffee samples and instrumental analysis for the collection of the spectroscopic data.

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Keywords
Coffee beans,NIR spectroscopy,Chemometrics,Environmental variability,Quality

P01.10.87

NON-DESTRUCTIVE TWO-DIMENSIONAL MONITORING OF ADHESIVE CURING PROCESS INSIDE RESIN STRUCTURE

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Introduction:

Adhesives are often used to join plastic parts. Adhesives can easily join parts together and have excellent environmental performance, such as being lightweight and low cost. In particular, cyanoacrylate adhesives can firmly bond various types of plastic parts in a short time, so they are used not only in industrial applications but also in everyday applications such as at home. However, after the parts are bonded together, the adhesive condition cannot be visually confirmed from the outside. Although it can be bonded in a short time, the evaluation of the adhesiveness must rely on adhesive strength testing by sampling some parts or destructive testing. In this study, we developed a new imaging method that utilizes the scattering phenomenon of near-infrared light and attempted to nondestructively and noncontactly monitor the curing process of adhesives when joining plastic parts.

Experimental:

Cyanoacrylate adhesive was applied to an ABS resin plate to create a 'resin part bonding model.' The progress of the adhesive hardening was observed in real time from the outside in a non-destructive and non-contact technique (Fig. 1), and the obtained image was binarized to obtain a discrimination image.

Results and Discussion:

Near-infrared light penetrates resins and adhesives well, so the adhesive appeared transparent immediately after bonding, but as the adhesive hardened, the area that appeared black gradually increased (Fig. 2). It is considered that as the adhesive contracted with hardening, cracks appeared, and the near-infrared light was scattered only at the interface of the cracks, causing the adhesive to appear black. This study demonstrated that the new imaging method can be used to non-destructively, non-contactly, and in real time to monitor the curing state of the adhesive (adhesion status), even for resin parts that have been bonded together with adhesive.

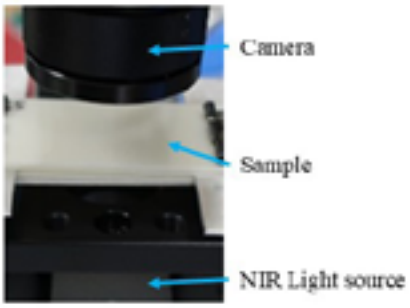


Figure 1 Monitoring by new imaging method.



Figure 2 Image of adhesive curing process over time.

Keywords
Near Infrared,Adhesive,Non-destructive observation,Monitoring,Scattered light

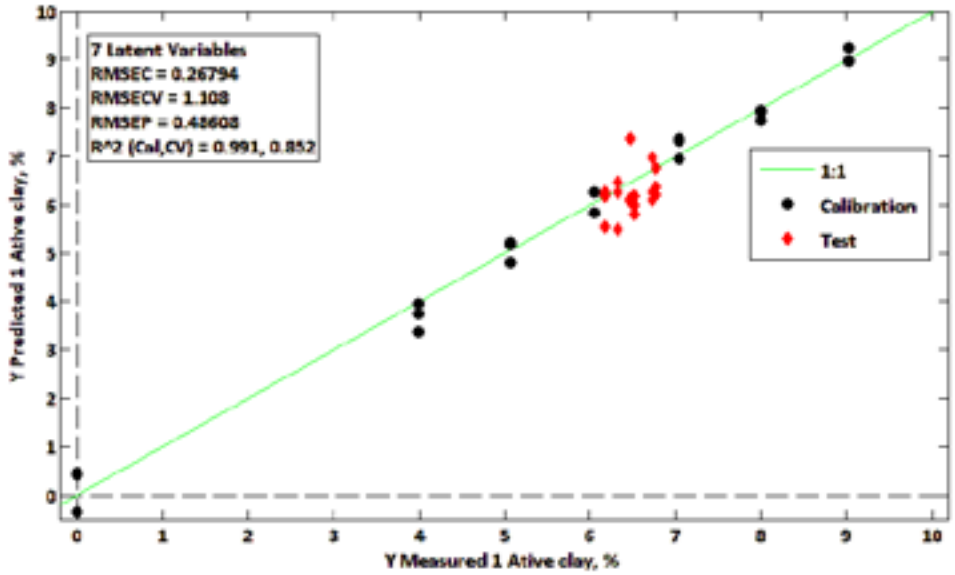
P01.10.88

CLEANER ANALYTICAL METHODOLOGY FOR QUANTIFYING ACTIVE CLAY IN FOUNDRY SAND USING NEAR-INFRARED SPECTROSCOPY

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The implementation of cleaner production processes is a critical strategy for industrial development, providing a competitive organizational advantage through optimized results, reduced resource waste, minimized environmental impacts, and ensured quality and safety in outcomes. Specifically, in the quality control of foundry sand, parameters such as granulometry, mechanical strength, refractoriness, and water retention are essential to guarantee product reliability. Traditionally, the foundry industry relies on classical analytical principles for quality control, which often require large volumes of chemical reagents, generate waste, and are time-consuming (ABNT NBR 16062). To address these limitations, this study aimed to develop an analytical methodology for the quantification of active clay in foundry sand using near-infrared (NIR) spectroscopy and multivariate calibration methods. The analytical curve was prepared using mixtures of bentonite, sand, and coal, resulting in a concentration range of 0 to 10% bentonite (active clay). Near-infrared spectra were acquired with a portable spectrophotometer designed for solid sample analysis. This equipment utilized a DLP NIRscan Nano EVM spectrophotometer (Texas Instruments®, USA) to capture infrared spectra in the range of 900 to 1,700 nm, with a resolution of 4 nm, 8 scans, and in absorbance mode. To protect the spectrophotometer and facilitate spectrum acquisition, a protective case and sampling chamber were produced using 3D printing technology (3D Pro, GTMAX3D Core A2V2, BR) with polylactic acid (PLA) filament. The spectrophotometer was connected to a smartphone via Bluetooth® using the Nanometrix app, developed by our research group, for data acquisition and storage. The smartphone also provided power to the spectrophotometer through a micro-USB cable.Partial least squares (PLS) calibration models were developed using SOLO+MIA software (Eigenvector Research, Inc.) for clay determination. Various preprocessing tools and combinations were investigated to optimize model performance. The number of latent variables (LV) and the best preprocessing strategy were determined based on the lowest cross-validation error (root mean square error of cross-validation, RMSECV) and prediction error (root mean square error of prediction, RMSEP), respectively. These models were applied to quantify active clay in real samples obtained from the foundry process in collaboration with Cercena Metallurgical Industry in Brazil (https://www.cercena.com.br/). The optimal preprocessing combination included the first derivative, Savitzky–Golay smoothing filter (polynomial order 2 with 21 data points), and mean centering. This approach resulted in RMSEC and RMSEP values of 0.26 and 0.48% (w/w), respectively. The portable near-infrared methods have proven to be satisfactory for the direct determination of active clay in foundry sand. These results highlight the great potential for developing methodologies aligned with white analytical chemistry principles due to their analytical performance, functionality, and sustainability.



ACKNOLEDGEMENTS: To National Council for Scientific and Technological Development (CNPQ) - protocol and 302816/2023-2, Coordination for the Improvement of Higher Education Personnel (CAPES) - code 001, 88881.710390/2022-1 and Foundation for Research of the State of Rio Grande do Sul (FAPERGS).

Keywords
Foundry sand,Metallurgical industry,Clay determinati,Active clay

INSTRUMENTATION

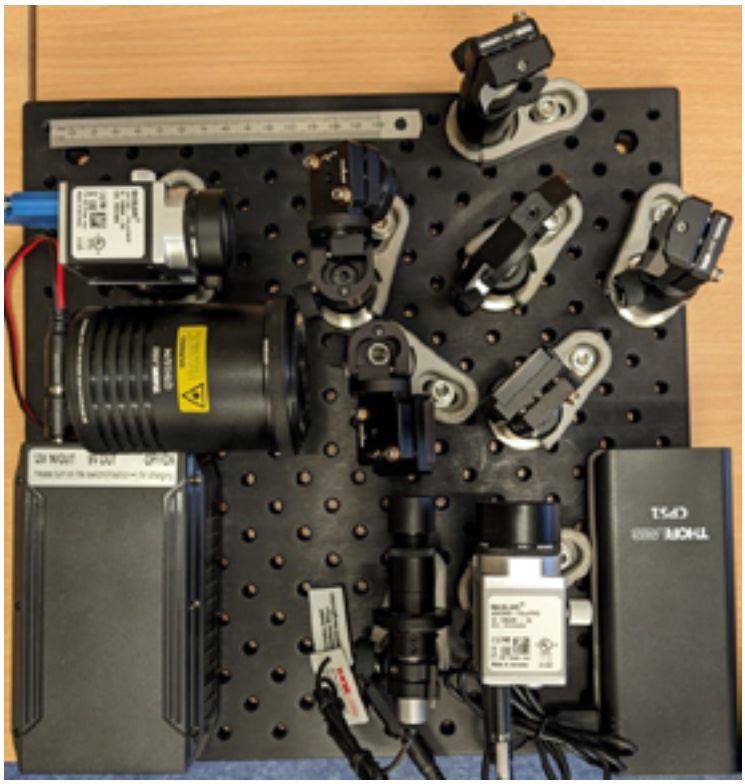
P01.11.89

A COMPACT BROADBAND STATIC FOURIER TRANSFORM SPECTROMETER

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Most Fourier Transform (FT) spectrometers use 2D optical plane interferometry to produce an interferogram by either a moving mirror (Michelson) or a static curved mirror (Sagnac). However, these spectrometers only use 50% of incoming light and the rest is redirected towards the source. We present a new 3D optical layout for a modified Sagnac interferometer using all incoming light. This innovative design has no moving parts and uses tilted mirrors to raise the optical plane above the incoming light path. Many benefits can be gained from this configuration and here we demonstrate extending the spectral bandwidth. The spectrometer is aligned and calibrated using two laser diodes of known wavelengths; 404.0nm and 833.7nm. When a broadband tungsten-halogen light source is used, two complimentary interferograms are simultaneously measured from visible (400-1000nm) and SWIR (800-1700nm) spectral regions using two different detector arrays. The spectrometer performance was tested using multi-peak spectral bandpass filters and results are in good agreement with the manufacturer specifications. We achieved a spectral range of 400-1700nm with resolution of <1% of center wavelength with an accuracy of <3nm. This technique can be applied to other spectral regions by changing the main optical components.



ACKNOLEDGEMENTS: Science and Technology Facilities Council (STFC) Proof of Concept Fund.

Keywords
Instrumentation,Fourier Transform,Spectrometer,Broadband

P01.11.90

ADVANCED PHOTONIC MEMS FT-NIR SPECTRAL SENSING WITH AUTOREFERENCING, ENHANCED STABILITY AND DURABILITY

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Büchi (Switzerland)

Photonic Microelectronic Mechanical System (MEMS)-based Fourier Transform Near Infrared (FT-NIR) spectroscopy has been shown as a competitive technology in the spectral sensors market due to the low cost and high performance [1]. This type of sensor has been used in different applications in different fields such as health and beauty, feed analysis, food analysis and oil and gas industry. The applications can be further extended to the field of robotics, process analysis and industry 4.0 by integrating autonomous features and improving the sensor immunity to environmental effects. In this work, we present a novel sensor that combines FTIR features using photonic MEMS technology with the autonomous features and built-in calibrations needed for uninterrupted operation in the above mentioned fields. The sensing system module, shown in Fig. 1, consists of the optical head containing the light source and the built-in reference, the sensor containing the core MEMS chip and ASIC chips of the sensor, the powering circuitry, and the controlling system. The system durability ensures a long-life time of 60,000 operational hours. Autonomous features have been integrated into the sensor to achieve continuous, non-interruptive and human-free operation. The autonomous features include self-referencing using a highly-stable and long-life mechanical shutter as well as a factory-calibrated temperature sensor and a compensation algorithm to significantly reduce baseline and wavelength drifts across the temperature range -10 – 60 degree C. Drift of power level is minimized to be less than 2% across the temperature range -10 – 60 degree C, as shown in Fig.2. In addition, wavelength shifts are corrected against humidity variations using self-correction technique. This technique reduces the humidity effect on the electronic components to meet the wavelength accuracy specs. Fig.3 shows the typical wavelength shifts after wavelength temperature compensation. The wavelength range of operation is 1350-2550 nm with 16 nm resolution at 1550 nm. The optical design from top to bottom is optimized to achieve a Signal-to-Noise Ratio (SNR) of about 10,000:1 for a scan time of 5 sec. Pipelining operation mode is introduced to measure the new sample in parallel to data processing and transfer of the previous measurement. In addition, the optical design does not necessitate a physical contact between the sample and the optical window with an optical power rate of change of less than 2 % / mm. Furthermore, with respect to previously reported sensors in the literature [1, 2], the optical head together with a novel specular reflection elimination algorithm lead to a reduction of the specular rays contribution by about an order of magnitude leading to enhancements in the photometric accuracy by >5 times. The sensor is provided with different options of communication interfaces, equipped with a sealed enclosure that also passively dissipates the thermal energy of the electronics components and the light source. We believe this sensor is a significant contribution to the sustainable development goals using NIR technology.

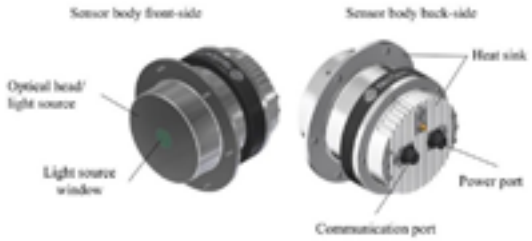


Figure 1: The sensing system module with built-in reference for autonomous operation

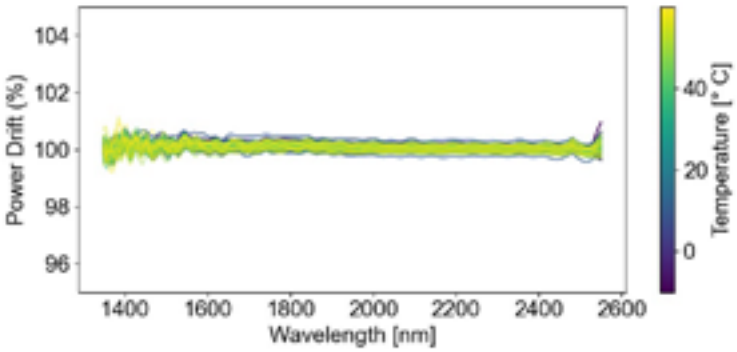


Figure 2: The improvement of temperature compensation on the power drift

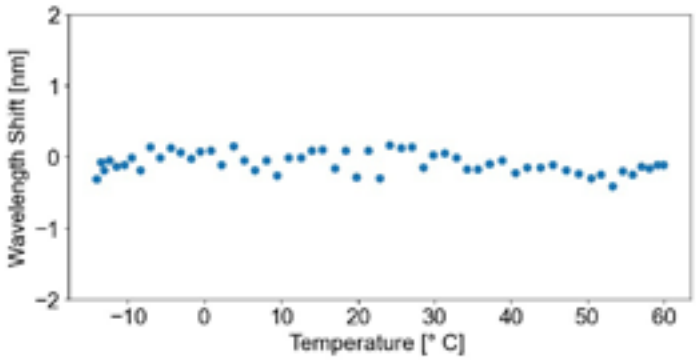


Figure 3: The wavelength shifts after wavelength temperature compensation

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Keywords
Microspectrometer,Industry 4.0,Autonomous sensor,Environmental drift,Process analysis

P01.11.91

COOKING UP HYBRID SAUSAGE: BREAKTHROUGHS WITH NIR HYPERSPECTRAL IMAGING

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The development of hybrid sausage products, combining meat with plant-based ingredients, is a growing trend in the food industry, driven by consumer demand for healthier and more sustainable options. However, achieving consistent quality in these complex formulations presents challenges, particularly in maintaining optimal texture, flavor, nutritional value, and uniform composition [1].

This study explores the potential of Near Infrared Hyperspectral Imaging (NIR-HSI) to support enhanced product design. Spectra were acquired for different recipes and sausage parts using a Specim Fx17 line scanner, ranging from 900 to 1700 nm with 3.5 nm of spectral resolution and coupled with two different light sources: traditional halogen lamps and a supercontinuum light source (NKT Photonics A/S, Denmark). The acquisition setup was designed to optimize the spectral data quality while adhering to practical constraints. The optical mount used for the supercontinuum light source was custom-designed and optimized to be lightweight and compact, given constraints imposed by laser optics, camera housing/objective, and optomechanical mounts. The laser was operated at 37% power, balancing spectral performance with compatibility to the FX17 camera settings. The data were processed using multivariate analysis for exploration and clustering, enabling the identification of distinct patterns within the samples. Spectral characterization, combined with multivariate error analysis, allowed for the assessment of variability in the data, including uncertainties arising from the use of different light sources.

The ability of NIR-HSI to capture molecular characteristics enables precise identification of key quality markers, such as lipids, proteins, and salt content, while also evaluating variability in raw materials, formulations, and batches [2]. A key innovation in this study is the application of the supercontinuum light source [3], which minimizes sample heating—a common issue in food analysis—preserving the integrity of sensitive food matrices during measurements. This capability enhances the reliability of spectral data, making it a valuable tool for the food industry.

This study demonstrates how NIR-HSI can enhance product development by supporting quality control and offering a non-destructive method for assessing food composition and variability. Ultimately, this approach contributes to the creation of superior hybrid sausage products, with potential benefits including improved sustainability, reduced waste, and innovative quality assurance practices in the food industry.

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ACKNOLEDGEMENTS: This research was supported by the Innovation Fund Denmark under the project "HyperSort - Hyper-spectral imaging for advanced textile sorting and food control." Additionally, funding was provided by the Elkartek Programme of the Basque Government (Spain) under the SMART-EYE project (KK-2023/00021).

Keywords
Product development,NIR imaging,Supercontinuum light source,Sausages,Quality markers

P01.11.93

HOW NIR AND REFLECTANCE UV SPECTROSCOPY SUPPLEMENT EACH OTHER IN NOVEL FOODS ANALYSIS FOR TOXICOLOGY STUDIES

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Recently, numerous novel food registration attempts have been submitted to EFSA (European Food Safety Authority), which requires strict toxicology testing, including concentration and homogeneity analysis in rat diet. The nature of these biological origin multicomponent foods does not allow the application of traditional techniques such as chromatographic analysis, particularly in dietary studies. Therefore, Near Infrared (NIR) spectroscopy has been a preferred method due to its non-destructive nature and rapid analysis capabilities in our company when we entered this quickly growing market. To meet the requirements of clients accustomed to relevant chromatographic standards, we must build calibrating models of HPLC quality and comply with accepted validation criteria of the chromatographic performance parameters. This is impossible in the NIR analysis of solid samples without developing very strict - and time-consuming - drying and grinding protocols and highly standardised sample preparation. The model building also has its challenges: there is a very narrow boundary between overfitting data and meeting the acceptance criteria of the guidelines required.

With many successful projects behind our back, our recent endeavours at Science Port Ltd. have focused on implementing Reflectance UV spectroscopy, utilising an integrating sphere UV spectrophotometer as a robust and sensitive technique for extending our NIR capabilities. Although UV spectroscopy is mainly applied as a univariate method, applying traditional chemometric techniques, such as principal component analysis (PCA) and partial least squares regression (PLSR), has enabled us to extract and interpret complex data sets with higher precision.

Both techniques have advantages and drawbacks. Reflectance UV spectroscopy is less universal and requires the presence of chromophores, while the lack of water absorption interferences and the generally higher sensitivity in the UV range lead to building more rugged calibration models.

In the first part of our presentation, we will take a quick look at the challenges, followed by comparative NIR and Reflectance UV spectroscopy analyses in modelled toxicological assessments. This comparison does not aim to point to a winner between the two methods - we believe there is no ultimate solution - but to show how we built a comprehensive strategy for the novel food analyses in Science Port's portfolio.

P01.11.94

ASSESSMENT OF IN-LINE SIMULATION SYSTEMS USING PORTABLE NIR SPECTROPHOTOMETERS FOR THE PREDICTION OF MOISTURE CONTENT OF INDUSTRIAL WOODCHIP

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Among the current European climate actions, the increasing interest toward renewable energy resources paves the way for the use of woody residues in the bioenergy sector. Due to its natural origin and immediate availability for use, industrial woodchips significantly enhance the energy efficiency of the entire supply chain associated with thermal and electric energy production. However the inherent heterogeneity of woodchips can negatively impact combustion efficiency, as well as technical performance, environmental sustainability, and economic viability [1]. Above all, moisture content (MC) is a crucial parameter during energy conversion, due to its influence in reducing the calorific value of the biomass. As a consequence, it is essential the qualitative characterization of the biofuel that leads to a continuous control and responsive management, ensuring cost-effectiveness and a sustainable approach [2]. Nevertheless, the current ISO standard requires the use of a thermo-ventilated oven and an extensive amount of time to assess the MC of the biomass, limiting the fast and convenient monitoring. Moreover, the destructive method of ISO standard makes repeatability of analysis impossible. Thereby, Near Infrared Spectroscopy (NIRS) represents an affordable substitute to standard analysis, due to its rapid and intuitive approach, both in lab and in-field conditions. The diffuse application of portable spectrophotometers promotes real-time results and in-line implementation directly in power plants [3]. Thus, the aim of this study is to assess the performance of a portable NIR instrument in predicting the MC of the industrial woodchip in a in-line simulation. Three Partial Least Squares regression (PLS) models have been developed and compared according to three different data collection approaches using 104 samples, in details: i) each sample is averaged across the 9 replicates (MET1), ii) each replicate is considered a single sample (MET2), and iii) each scan is assumed as a single sample (MET3). Considering each approach, the predicted MC obtained by the best PLS model is aligned with the laboratory MC value, computed according to ISO 18134. Statistics, Tukey's Test and Tolerance Limit's have been carried out to investigate the material variability, as well as the replicates weight and influence for the MC prediction efficiency. As last, all the results have been compared with the outcomes obtained with the application of a different portable NIR instrument, simulating the same in-line condition [4]. The results confirm the relevant variability of woodchip material, proving the importance of collecting enough replicates to guarantee an appropriate qualitative representation of the sample. Thus, the NIRS implementation along the material flows in power plants could enhance a rapid and sustainable prediction of the most critical parameters, ensuring economic advantages and technical supervision. Currently, this potential improvement is prevented by the lack of a dedicated ISO standard that could establish the endorsed application of NIRS.

Keywords
near infrared spectroscopy,biofuel,PLS,renewable energy,variability

P01.11.95

DIRECT STANDARDIZATION FOR CALIBRATION TRANSFER BETWEEN PORTABLE NIR SPECTROMETERS IN MILK ANALYSIS

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In the context of Precision Livestock Farming (PLF), portable NIR instruments offer a versatile and effective solution for the monitoring of various aspects of the production chain, ranging from forage to dairy products. At the same time, the replacement of an existing device or the acquisition of a new one results in significant financial investment for developing a calibration from the beginning. Spectral discrepancies can arise due to variations in optical components, light sources, or detectors. A more sustainable and cost-effective alternative is calibration transfer that compensates for such differences and ensures measurement consistency, thereby enabling the utilization of a unified model across multiple instruments, a feature that is particularly advantageous in industrial and research contexts. Calibration transfer facilitates the application of calibration models from a primary instrument, designated as the 'master', to secondary devices, referred to as the 'slave'. The present study evaluated the feasibility of calibration transfer for predicting fat and protein content in milk between different portable NIR instruments. MicroNIR (VIAVI), equipped with a linear variable filter (LVF)-based monochromator and a NANOSENS accessory for transflectance spectral acquisition (1 mm optical path), was used as the master instrument. This accessory enables the thermostatic control of samples. The two slave instruments tested were: AlbaNIR (GraiNit) a device based on DLP (Digital Light Processing) technology performing measurements in transmittance (1 mm optical path); and Polispec, a diode array-based instrument, that collects spectra in transflectance mode (1 mm optical path). Two calibration transfer methods were assessed: Direct Standardization (DS) and Piecewise Direct Standardization (PDS).

For this study, a total of 170 samples of individual raw milk and semi-skimmed milk were analysed. These comprised 115 samples that were used for the development of the calibration model and 55 samples, which were used for subsequent calibration transfer analyses.

The calibration model was developed using PLS regression based on 115 spectra acquired with MicroNIR. For fat content, the spectra were preprocessed with Savitzky-Golay (first derivative) and autoscaling. For protein content, in addition to these preprocessing methods, the GLSW filter (optimized for alpha = 0.001) was also applied. The remaining 55 samples were analyzed with both MicroNIR and the two slave instruments, AlbaNIR and Polispec and divided into two group sets with Kennard-Stone DUPLEX: 20 samples were used to build the standardization matrix, while the remaining 35 were employed as the test set.

The initial calibration models on MicroNIR showed the following statistics: for fat, R^2 (CV) = 0.97 and RMSECV= 0.19; for protein, R^2 (CV)= 0.67 and RMSECV = 0.16. The best calibration transfer performance was achieved using DS, giving the following results: AlbaNIR showed for fat R^2(prediction) = 0.88 and RMSEP = 0.36; for protein, R^2 (prediction) = 0.14 and RMSEP = 0.38. Polispec, instead, for fat R^2 (prediction) = 0.91 and RMSEP = 0.45; for protein, R^2 (prediction) = 0.56 and RMSEP = 0.44.

The calibration transfer demonstrated to offer a cost-effective solution, especially regarding fat prediction. However, the accuracy of protein prediction was found to be lower, thus indicating the need for further optimization. It is recommended that future research explore advanced standardization techniques and larger datasets, with a view to enhancing the robustness and applicability of calibration transfer in the analysis of milk composition.

Keywords
Calibration Transfer,Portable NIR spectrometers,Direct Standardization,Milk analysis

PETROCHEMISTRY, POLYMERS & TEXTILE

P01.12.97

GENETIC ALGORITHM FOR BAND SELECTION IN MICROPLASTIC DISCRIMINATION WITH HYPERSPECTRAL IMAGING

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Imec, Leuven, BELGIUM

Hyperspectral imaging combines the characteristics of computer vision and point spectroscopy by obtaining an image with both spatial and spectral information. Therefore, in combination with microscopy, it can increase material discrimination possibilities with respect to regular microscopy. We explore the potential to discriminate microplastic contamination material with hyperspectral microscopy. For this purpose, a hyperspectral short wavelength-infrared (SWIR) camera [1] is used in combination with reflection microscopy for pellet material identification. This camera provides high spectral and spatial resolution in the 1100-1650 nm range, 100 spectral bands and up to 640x512 spatial resolution and high acquisition speed. The analysis performed shows potential to accurately discriminate the 22 types of plastic pellets tested. The identification of the most relevant bands can drive customized sensor development to achieve cost-efficient sensors with fewer bands [2] and reduce simultaneously memory and processing requirements. For this purpose, we present a genetic algorithm [3] that performs the band relevance analysis. Genetic algorithms are one of the most common band selection methods [4], based on iteratively improving the band selection according to the computation of the fitness of already available solutions (band selections). The fitness of each band selection is then given as the classification accuracy for that specific band subset. The results show that a few spectral bands (<10) are sufficient for accurate discrimination of the considered plastic pellets. Therefore, a cost-efficient multispectral sensor could lead to a faster contamination assessment than traditional techniques used for microplastic discrimination.

Band selection with genetic algorithms is a well-researched topic, however, to our knowledge an analysis of the parameter impact, classifier methods, or class prioritization mechanisms has not been well addressed yet. This paper explores the impact of all these aspects in the algorithm evolution and convergence. The results show that the genetic algorithm can be steered through parameter selection for a faster convergence and higher classification accuracy. We can conclude that by using more simple classifiers in a first phase of iterations and more advance classifiers in the last iterations the algorithm can converge to good solutions while reducing its execution time. Finally, we compare different weight-based and prioritization methods to steer the band selection towards a fairer distribution of the classification accuracy between multiple material classes. By adapting the fitness computation, we can achieve a more balanced accuracy for all different material classes.

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ACKNOLEDGEMENTS: We want to acknowledge the Eximious project, European Union's Horizon 2020 research and innovation program under grant agreement No 874707.

Keywords
Hyperspectral,Genetic algorithm,Band selection,Microplastics,Microscopy

PHARMA & BIOTECH

P01.13.98

LIGHTING THE WAY: NEAR-INFRARED SPECTROSCOPY AS THE FUTURE OF CANNABIS SATIVA L. ANALYSIS

María del Carmen Díaz Liñán, María Teresa García Valverde, Carlos Ferreiro Vera

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Despite the controversy associated with its recreational used, Cannabis sativa L. has been extensively used in different fields, such as food production, material manufacturing and, the textile industries. It has also demonstrated multiple medicinal applications, serving as a potential palliative measure for various conditions such as chronic pain or multiple sclerosis, among others. For this reason, the current medical cannabis industry focuses on the development of Cannabis and Cannabis-derived products for medical purposes. Consequently, quality control in this industry has increased exponentially, and new, strict regulations have been established in order to obtain safe and efficient pharmaceuticals products.

Phytoplant Research S.L.U. is a company in Spain dedicated to cannabis research, from genetic improvement and cultivation to the extraction and isolation of the plant's active compounds for medicinal purposes. This generates a large volume of plants that need to be assessed in order to obtain valuable information and comply with the legislation.

Until now, all the relevant parameters of the Cannabis plant, i.e., cannabinoids, terpenes, moisture content and micro/macronutrients, have been determined via traditional techniques such as liquid and gas chromatography, Karl-Fischer, Kjeldahl, and atomic absorption spectrometry, among others. These techniques are associated to time-consuming processes and high costs, as well as requiring specialized personnel to perform sample treatment preparation prior its analysis. However, we have introduced Near-infrared spectroscopy (NIRS) for the quality control of the cannabis plant, and it has streamlined our daily processes while also cutting down analysis costs.

One of the most attractive advantages of this technology is its portability, which allows real-time and on-site measurements. We are currently employing the GraiNit Aurora NIRS, which is a portable and handheld device that can provide quantitative and qualitative analysis of the Cannabis plant. Through its very intuitive calibration software, i.e., INPROCESS, we have developed multiple predictive models for the determination of cannabinoids, moisture content in dried and fresh vegetal material and micro/macronutrients, and we are currently elaborating new equations for the determination of terpenes and water activity, while also improving the other models.

In this presentation, the current regulatory framework of Cannabis will be outlined and the potential of NIRS technology for the quality control of Cannabis will be highlighted. To this end, we will briefly discuss the predictive models that have been developed in our laboratory with the Aurora NIRS, emphasizing the capabilities of this particular instrument.

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Keywords
cannabis,near-infrared spectroscopy,cannabinoids,terpenes

P01.13.99

COMPARING NON-LINEAR AND LINEAR REGRESSION FOR NIR METHOD DEVELOPMENT

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1- Introduction

This presentation will examine non-linear regression algorithms and machine learning approaches, assessing their potential in PAT (Process analytical technology) applications for continuous manufacturing. By comparing these advanced techniques with established PLS methods, we aim to highlight the benefits and possibilities they offer for optimizing model performance and advancing pharmaceutical production.

2 - Material and methods

To enhance the accuracy of the final model, we conducted a thorough comparison of different preprocessing methods and settings. Once the data preprocessing was complete, we applied a range of non-linear regression techniques to the dataset. These included decision tree (DT), Random Forest, K-Nearest Neighbors (KNN), Support Vector Machine (SVM), and Artificial Neural Networks (ANN).

By employing these advanced regression techniques, we aimed to uncover the true potential of the dataset and identify the most effective model for our analysis. Furthermore, we compared the results obtained from the standard PLS regression approach against the outcomes derived from the non-linear regression techniques.

3 - Results and discussion

In this study, we conducted a comprehensive evaluation of various non-linear algorithms to assess their potential in enhancing prediction accuracy. The calibration accuracy of these algorithms was measured using Standard Error of Calibration (SEC) and Standard Error of Prediction (SEP), and the results were compared to the benchmark technique, linear Partial Least Squares (PLS).

By analyzing and comparing the SEC and SEP values, we aimed to identify the algorithms that demonstrated the greatest potential for improving prediction accuracy. This systematic comparison allowed us to gain insights into the strengths and weaknesses of each algorithm and make informed decisions regarding algorithm selection for optimal prediction accuracy improvement.

Additionally, we highlight the findings specific to neural networks in this study. Notably, neural networks exhibited a significant improvement in the standard error of prediction, reducing it by a factor of two. These results underscore the efficacy of neural networks in enhancing the precision and reliability of predictions within our research domain.

Overall, this study provides valuable insights into algorithm selection for improving prediction accuracy, with neural networks emerging as a promising approach for achieving superior results.

4- Conclusion

Near Infrared Spectroscopy (NIRS) has gained widespread popularity as a valuable technique for online Process Analytical Technology (PAT) applications. In this realm, statistical models utilizing spectral data have traditionally been calibrated using the widely adopted partial least squares (PLS) regression algorithms. However, to further enhance the accuracy of models employed for PAT applications, this project aimed to compare the performance of classical PLS regression with non-linear regression and machine learning algorithms.

By exploring these more advanced algorithms, we sought to unlock their potential in achieving higher accuracy for NIR calibration, specifically for the quantification of Active Pharmaceutical Ingredients (APIs) in drug products. The objective was to elevate the precision and reliability of models used in PAT applications, thereby improving process control and overall product quality.

This comparative analysis serves as a steppingstone towards leveraging the capabilities of non-linear regression and machine learning algorithms in the realm of NIR calibration. By embracing these more sophisticated techniques, we aspire to push the boundaries of accuracy and establish new benchmarks for PAT applications in the pharmaceutical industry.

Keywords
Near infrared spectroscopy,Process Analytical Technology,non linear regression,Pharmaceutical industry

P01.13.100

LOW CONCENTRATION QUANTITATIVE ANALYSIS OF LEVOTHYROXINE IN TABLETS USING PORTABLE AND BENCHTOP NIR SPECTROMETERS WITH PLS REGRESSION

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Near-infrared spectroscopy (NIR) and Partial Least Square Regression (PLSR) are powerful, non-destructive techniques for analyzing pharmaceutical solid dosage forms. However, their application to samples with low levels of active ingredients remains challenging. This study developed PLSR models using benchtop and portable NIR spectral data to quantitatively determine levothyroxine in commercial tablets from different manufacturers containing 25-150 µg/tablet of the drug. This study will evaluate which preprocess selection would obtain the optimal PLSR model for benchtop and portable NIR instruments. Standard Normal Variate (SNV), Savitzky-Golay derivatives, and Orthogonal Signal Correction (OSC), among others, will be tested. In addition, benchtop and portable instruments were compared to each other to look for significant differences between the predictions obtained by the models from these two instruments.

The significance of this research lies in its demonstration of the potential for portable NIR devices to perform comparably to benchtop devices. This advancement is crucial for the pharmaceutical industry, as it enables more flexible, efficient, and accessible quality control processes, particularly for products with very low concentrations of active ingredients. By validating the effectiveness of portable NIR spectrometers, this study paves the way for broader adoption of these tools, ultimately enhancing the ability to ensure the safety and efficacy of pharmaceutical products.

ACKNOLEDGEMENTS: The authors would like to thank the Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Mahidol University for research facilities. Global Dynamics (THAILAND) CO., LTD. is acknowledged for supporting the portable NIR facility in this study.

Keywords
levothyroxine tablet,partial least square regressio,near infrared spectroscopy

P01.13.101

RAPID QUANTIFICATION OF TOXIC DIETHYLENE GLYCOL (DEG) CONTAMINANT IN GLYCERIN BASED COUGH SYRUP USING NEAR INFRARED SPECTROSCOPY

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Diethylene Glycol (DEG) is a low-cost, sweet-tasting industrial solvent commonly used in antifreeze solutions, brake fluids, and cosmetics. However, it is highly toxic and can cause renal failure, peripheral neuropathy, encephalopathy, coma, and even death if ingested [1].

In contrast, Glycerin (or glycerol) is a widely used alcohol with applications in over 2,000 products in pharmaceutical, food, and personal care industries. Glycerin is typically derived from fats and oils or obtained as a by-product of biofuel production. Due to the rising global demand for glycerin, some unethical manufacturers illegally substituted it with DEG in pharmaceutical products such as toothpaste, fever medications, and cough syrups, because of DEG's similar physicochemical properties to glycerin and propylene glycol.

To detect and quantify DEG contamination in glycerin, various analytical methods are currently in use, including gas chromatography (GC), gas chromatography-mass spectrometry (GC-MS), and thin-layer chromatography (TLC), among others. However, these techniques are often limited by high costs, time-consuming sample preparation, and sample destruction. As a result, alternative low-cost detection methods have been emerging, offering advantages in situ detection without the need for reagents [2].

This work explores the applicability of NIR spectroscopy in detecting potential DEG contamination in complex commercial products containing glycerin. Fifty-four glycerin-based syrup samples were spiked with known amounts of DEG ranging from 3 to 50% DEG. The resultant samples were vortexed to perform near-infrared spectroscopic measurements. The absorption at 881 cm−1 was mostly due to DEG, with minimum contribution from the other constituents. The spectra of syrup were acquired with a Metaspeq spectrometer (Ayudyog, Kolkata, India) in transmission mode from 900nm to 1,700 nm. Each sample, in a 1 mm thick quartz cuvette (20 mm x 20 mm), was scanned with 35,000 µs exposure time and 16 scans averaged to produce the final spectrum. Data acquisition, and model establishment were all performed using SpectraFind software (Ayudyog Pvt Ltd., Kolkata, India).

To measure DEG percentage, a regression model was developed using the PLS algorithm on NIR spectra from 54 samples. The number of PLS components and the choice of preprocessing methods were optimized simultaneously through leave-one-sample-out cross-validation during model calibration [3]. The model's performance was evaluated using the root mean square error of cross-validation (RMSECV), root mean square error of prediction (RMSEP), and correlation coefficients (R2). The PLS regression with 44 samples in the training set and 10 samples in the testing set resulted in the performance parameters as RMSECV = 0.134, RMSEP = 0.069, rcv2 = 0.869, rp2 = 0.65, and RPD = 5.626 for five number components.

This study shows the efficacy of NIR spectroscopy for estimation of paracetamol content in tablets. This methodology can also be applied for quality control and content uniformity in other liquid pharmaceutical production.

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ACKNOLEDGEMENTS: The authors acknowledge the funding support of the Department of Biotechnology.

Keywords
GLYCERIN,DIETHYLENE GLYCOL,NEAR INFRARED SPECTROSCOPY

SUSTAINABILITY & RECYCLING

P01.14.102

INTEGRATION OF MICRONIR AND PRESSURIZED LIQUID EXTRACTION (PLE) IN SUGARCANE BAGASSE VALORIZATION

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Sugarcane bagasse (SCB), a byproduct of sugarcane production, offers potential for circular economy applications. Pre-treatment methods, such as pressurized liquid extraction (PLE), enable the selective recovery of lignin, cellulose, and hemicellulose. These compounds serve as precursors for platform chemicals like glucose, polyols, furfurals, and biofuels, supporting sustainable development. [1]. However, accurately profiling the composition of solid and extract phases is complex due to variable analyte diffusion and desorption rates. For instance, measuring PLE sample composition is laborious, requiring offline lab analysis and multiple operations such as dilution, dissolution, drying, evaporation, precipitation, and chromatography, introducing long-time delays [2, 3].

In this work, a MicroNIR spectrophotometer (1700 ES, Viavi Solutions), operating from 908 to 1676 nm, was used to quantify cellulose, hemicellulose, and lignin in the solid phase obtained along the PLE process [4, 5]. Each spectrum represented an average of 100 scans in reflectance mode. The PLE unit included an extraction vessel, pump, heater, cooler, pressure gauge, and control valves. Experiments were conducted at different temperatures (160, 180, and 200 °C), holding times (5 to 60 minutes), and solvents (ethanol, water, and ethanol/water mixtures). The obtained PLE results show effective extraction of compounds from SCB with increased temperature and time, with cellulose ranging from 35% to 75%, hemicellulose from 0% to 45%, and lignin from 5% to 30%, according to standard wet chemistry techniques applied (NREL/TP) [6, 7]. These experimental data were used as input for the development of MicroNIR calibration models for composition of residual SCB after PLE. Several preprocessing techniques were employed to reduce noise and eliminate variations not related to the component concentrations [8]. The techniques compared were Standard Normal Variate (SNV), Multiplicative Scatter Correction (MSC) and Savitzky-Golay (SG) first derivatives with either 5 or 7 points. Preprocessing and Partial Least Squares (PLS) calibration were performed using PLSToolbox (Eigenvector) and Unscrambler X 10.5 (AspenTech) multivariate analysis software. The SPXY algorithm was applied to split samples into validation (30%) and calibration (70%) groups, leading to models for lignin, cellulose, and hemicellulose contents in the biomass. The SG7 preprocessing proved most effective, yielding the lowest latent variables and validation errors: 5 latent variables with an RMSV of 1.05% for lignin, 6 latent variables with an RMSV of 1.32% for cellulose, and 3 latent variables with an RMSV of 1.88% for hemicellulose. Finally, the MicroNIR followed the progressive and smooth removal of compounds with temperature, being able to identify the end of extraction, even when different solvents were employed.

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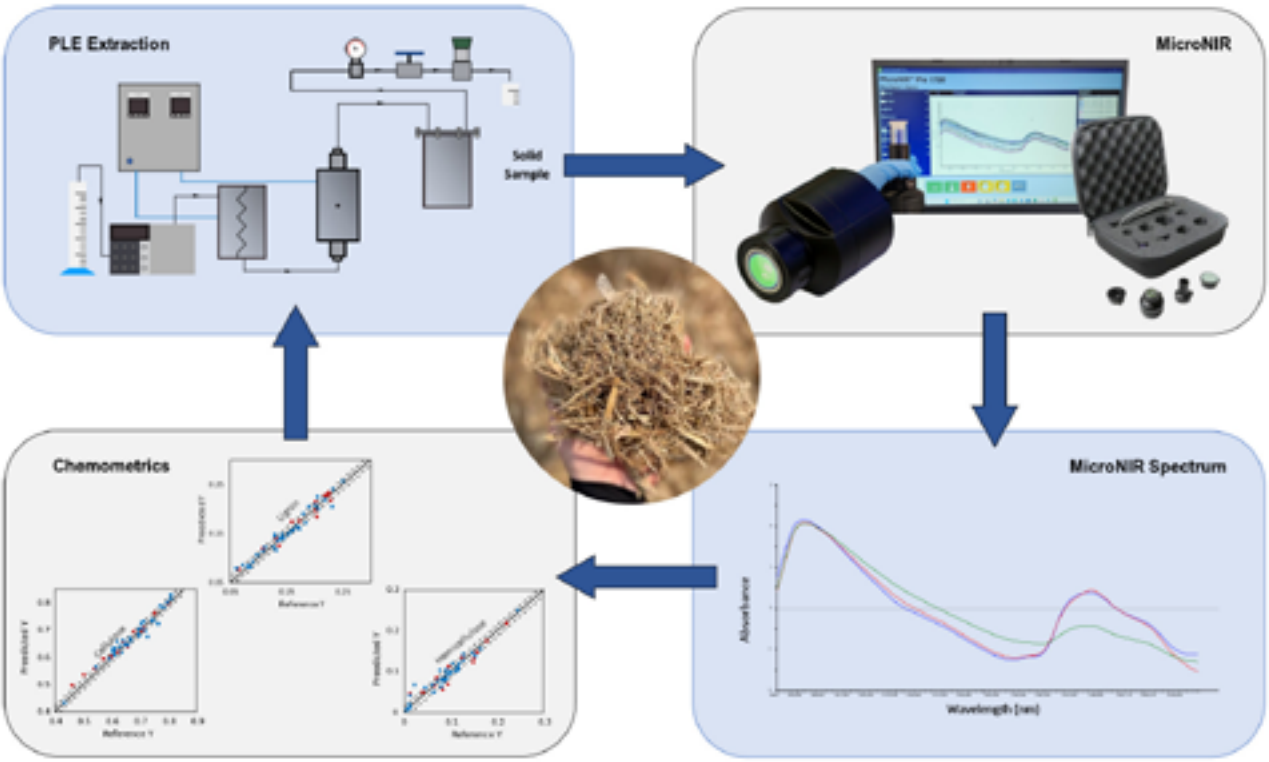
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ACKNOLEDGEMENTS: Authors wish to acknowledge the Brazilian funding agencies Finep and CNPq.

Keywords
MicroNIR,PLE,biomass

P01.14.103

ASSESSMENT OF BIOPOLYMER OF DEGRADATION USING NEAR-INFRARED HYPERSPECTRAL IMAGING AND MACHINE LEARNING METHODS

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The accumulation of waste is an environmentally concerning issue, driving extensive research into the development of biodegradable plastics derived from a wide variety of formulations to reduce their persistence in disposal environments. These formulations range from simple compositions that use a single polymer and a plasticizer to complex mixtures combining distinct polymers and hydrophilic compounds in optimized proportions. However, despite being biodegradable materials, it is necessary to understand how the components of polymeric matrices degrade under different environmental conditions, as this knowledge is crucial for the development of improved formulations. The literature highlights the use of specific international standards to assess the degradability of these materials through CO2 release processes (Beltrán-Sanahuja et al., 2021). However, such methods require controlled and labor-intensive conditions, which hinder their application to real-world scenarios. Based on this premise, this study focused on evaluating the degradation process of plastic films made of cassava starch, cellulose, and oregano essential oil in different soil types. Soil composition was closely linked to the microbial activity necessary for polymer decomposition. And, therefore, this property could have accelerated the biodegradation of biopolymers, helping to reduce the accumulation of non-degradable waste.

Near Infrared Hyperspectral Imaging (NIR-HSI) was employed to characterize and understand the degradation processes, as well as to investigate the variables that influenced the most on these processes. For this purpose, after being molded and dried, the plastic films were placed in direct contact with two types of soil (e.g. farm soils normally used for farming). NIR-HSI and RGB Images were acquired in the spectral range of 1000 to 2500 nm every two days, until the films completely disappeared. Subsequently, the data was analyzed by using exploratory methods such as principal component analysis (PCA) and multivariate curve resolution (MCR). The distribution of degradation was assessed through spatial mapping, allowing the identification of chemical trends resulting from the interactions of these polymers, thereby enhancing the understanding of the processes involved.

ACKNOLEDGEMENTS: This research was supported by FAPESP (Processes Nos. 2019/27354-3, 2019/03812-2 and 96/08366-5, 2014/50951-4, 2015/24351-2, 2023_07385-7, São Paulo Research Foundation - FAPESP), Coordination for the Improvement of Higher Education Personnel – Brazil (CAPES) - Financial Code 001,

Keywords
Sustainability,Biodegradable plastics,Chemometrics,Imaging

P01.14.104

SORTING THE UNSORTED: NIR SPECTROSCOPY AND CHEMOMETRICS IN WASTE RESOURCE VALORIZATION

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The global waste management challenge has become one of the most pressing issues of our time, driven by increasing urbanization and consumption patterns. In this context, the residual fractions from waste sorting processes, often dismissed as marginal, represent a valuable and underexplored resource. This study focuses on the application of NIR spectroscopy combined with advanced chemometric techniques to characterize mixed plastic and non-plastic materials originating from waste sorting residues.

The primary objective is to develop a rapid, precise, and non-destructive analytical approach capable of distinguishing the diverse materials within this complex matrix. Such characterization is critical for optimizing the end-use potential of these residues, whether as secondary solid fuel (SRF), feedstock for chemical recycling, or input for pyrolysis processes. The integration of NIR spectroscopy with multivariate data analysis enables the extraction of meaningful chemical and physical information from heterogeneous samples, overcoming traditional limitations associated with manual or slower analytical methods.

Our results demonstrate the feasibility of NIR-based methods to reliably classify and quantify key components within the waste matrix. Key chemometric approaches, have been employed to classify unsorted objects (by partial least squares-discriminant analysis, PLS-DA) and to quantify compounds of interest in mixed ground materials (by multivariate curve resolution, MCR). Additionally, this methodology aligns with the principles of Process Analytical Technology (PAT), promoting sustainable and efficient waste valorization pathways.

This research underscores the significance of revisiting waste sorting residues as an intriguing matrix rather than just a terminal disposal problem. By leveraging state-of-the-art spectroscopic and chemometric tools, this study contributes to the broader goal of sustainable resource management, supporting the transition to a circular economy. Future work will focus on scaling this approach to industrial applications and integrating real-time process monitoring capabilities, possibly with the use of hyperspectral imaging.

Keywords
waste sorting residues,secondary solid fuel,chemometrics,sustainable resource managemen

P01.14.105

NEAR-INFRARED SPECTROSCOPY AND MACHINE LEARNING FOR QUALITY MONITORING IN MEDICAGO SATIVA BIOREFINERY SUB-PRODUCTS

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The development of green biorefineries for the sustainable extraction and valorization of compounds from biomass calls for robust and reliable analytical quality control methods to assure standardization and safety. In this study, diffuse reflectance near-infrared spectroscopy (NIR) was applied for the analysis of Medicago sativa biorefinery end-products characterized by complex chemical compositions. Working on a limited number of samples (dataset of ninety observations), this work represents a critical investigation focused on the importance of data preparation and model evaluation when dealing with small datasets. Data pre-processing, feature selection methods, and different algorithms were compared for their impact on prediction accuracy. Models capable of capturing non-linear relationships and complex patterns resulted as the most effective, such as ensemble models and neural networks, which resulted in a Root Mean Square Error of Prediction of lower than 3%. Useful results have also been obtained with feature selection methods, highlighting the importance of determining the best candidate input features. Finally, the NIR spectra were used for product recognition using a multi-class classification model. Synthetic Minority Over-sampling Technique was used for class balancing, and 100% of classification accuracy was obtained. The at-line quality monitoring process strictly requires the inclusion of a higher number of samples and variabilities, however the data here presented are a proof-of-concept that this technique can reliably support innovative and sustainable biorefinery processes.

Keywords
biorefinery,data analysis,Medicago sativa,near-infrared spectroscopy,process monitoring; protein content

P01.14.106

INTEGRATION OF 2D HYPERSPECTRAL TECHNIQUES AND 3D RECONSTRUCTION FOR THE PRECISE DIFFERENTIATION OF MATERIALS IN CONSTRUCTION AND DEMOLITION WASTE RUBBLE

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Advances in analytical techniques have enabled significant progress in various fields, including industrial processes where rapid and precise material measurement is essential for quality control and recycling. In this context, NIR spectroscopy and hyperspectral imaging (HSI) have become crucial tools for extracting detailed information on the composition and structural characteristics of materials. In parallel, integrating these approaches with 3D reconstruction techniques offers an enriched spatial perspective, allowing spectral information to be linked with the actual geometry of the object under study.

This study explores the transformation of 2D hyperspectral images into a three-dimensional metric space, with the aim of comparing the accuracy and representativeness of 2D measurements with the reality obtained via 3D reconstruction. We have worked with rubble derived from construction and demolition waste, focusing on differentiating two key components: cement paste and natural aggregate. The experimental methodology included the acquisition of multiple hyperspectral captures from different angles using a SWIR line-scan camera and the generation of a point cloud enriched with spectral and geometric information through Structure from Motion (SfM) and Neural Radiance Fields (NeRF) algorithms.

The successful integration of these data required meticulous prior calibration of the camera to correct radial distortions and optimize intrinsic parameters, ensuring the convergence of the 3D reconstruction algorithms. Additionally, a structured-light stereo sensor was used to scan the rubble, providing geometric reference data that validated the accuracy of the reconstruction derived from hyperspectral images. The synchronization and fusion of both data sets allowed the alignment of spatial coordinates with spectral information, facilitating the application of spectral unmixing techniques to isolate and identify cement paste and natural aggregate.

Preliminary results show that, although 2D hyperspectral measurement is capable of partially differentiating between the materials, the incorporation of 3D reconstruction significantly improves the correlation between the spectral signature and the spatial distribution of the rubble. This integrated approach reduces reprojection errors and offers a more faithful representation of the structure and heterogeneity of the object, which is fundamental in industrial applications that demand rapid and precise measurements. Moreover, the study aims to evaluate the deviation between the 2D measurements performed on the material surface in an industrial setting with a conveyor belt and the 3D reconstruction, providing a quantification of discrepancies and determining the viability of 2D measurements under real process conditions.

In conclusion, the fusion of hyperspectral techniques and 3D reconstruction methods constitutes a robust approach for the characterization of materials in construction and demolition waste rubble. This novel system not only optimizes the identification of critical components but also lays the groundwork for future applications in industrial fields, enhancing classification, recycling, and quality control processes in an efficient and sustainable manner.

ACKNOLEDGEMENTS: We appreciate the collaboration of Tecnalia and the Departments of Analytical Chemistry and Automation at the University, whose cooperation has been essential for the development of this study.

Keywords
Hyperspectral Imaging (HSI),Near Infrared (NIR),Stereo-photometry (SP),3D reconstruction,Integration

P01.14.107

CHALLENGES IN SAMPLE PREPARATION TO BE SUITED FOR NIR ANALYSIS IN PLASTIC WASTE MANAGEMENT

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The proper identification and quantification of materials comprising the plastic solid waste is pivotal to improve the efficiency of the separation process in order to address the product for applications with the most "high value" use, fostering economic and environmental benefits. Such applications may include re-extrusion, mechanical recycling, chemical recycling and as fuel for energy recovery [1]. NIR spectroscopy among other analytical techniques has proven to be a suitable tool to achieve this goal.

Besides the intrinsic composition of materials entering the separation plant, environmental and storage conditions can affect the quality of plastic waste inputs. In our study, a minimum sample preparation was required to make materials suitable for NIR analysis in order to provide data portraying the actual material.

Several sources of plastic waste were considered in the current work. Sampling of batches pertaining five waste producers was carried out according to the DoE methodology taking into account the number of producers and the timeframe devoted to the project [2]. The diverse materials and samples planned to be tested featured different amounts of water, depending on the environmental conditions, that hindered the potentiality of NIR to provide accurate results. Consequently, samples prior to analysis have been set out in a sunny ambient and subjected to heat treatment at low temperatures.

Because of the preparation activity, the spectral data relevant to materials under testing were lacking from the interference of water thus improving the effectiveness of the methodology to properly label and quantify the unknown materials based on chemometric models developed on purpose [3].

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Keywords
Sample preparation,Design of experiments,Near infrared spectroscopy,Waste management

P01.14.108

APPLICATION OF LIFE CYCLE ASSESSMENT (LCA) FOR THE EVALUATION OF THE ENVIRONMENTAL BENEFITS OF THE USE OF VIS/NIR SPECTROSCOPY: A CASE STUDY ON GRAPE (VITIS VINIFERA L.) QUALITY MONITORING

Valentina Giovenzana [1], Alessio Tugnolo [1], Andrea Casson [1], Riccardo Guidetti [1], Roberto Beghi [1]

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By measuring grape quality parameters, it's possible to determine the optimum harvest timing, ensuring the production of high-quality wines. These parameters are usually obtained through traditional analytical methods, implying the use of chemicals. As a solution to destructiveness and slowness problems, optical methods can be suitable alternatives to monitor the technological maturation of grapes.

The work aims to evaluate and compare the environmental impact of three different types of analyses carried out to assess technological parameters (total soluble solids content, pH, and total acidity) using three different approaches: the wet-chem method, an optical method using a benchtop vis/NIR device, and a second optical method using an innovative and cost-effective vis/NIR prototype. The Life Cycle Assessment (LCA) methodology was used to identify the most sustainable solution in a 'from-cradle-to-grave' approach, according to the ISO 14040-14044 international standards. The functional unit was identified by executing the analyses necessary to obtain the three technological parameters.

Results demonstrate how, first, the wet-chem analyses should be carried out in triplicate to obtain technological parameters, while for the vis/NIR benchtop and the innovative proto-type, the three quality parameters can be obtained with only one single analysis. Moreover, the optical analysis carried out with the cost-effective prototype results in a solution with a lower impact and is 3.2 times more sustainable than the wet-chem analysis. If an innovative optical prototype can be identified as the best solution, its performance may not be so reliable in obtaining precise and trustworthy results. For this reason, a performance factor was included, and results have been normalized; nevertheless, results confirmed what was obtained in the previous scenario.

In conclusion, the study demonstrates how innovations in agriculture and the development of smart optical solutions could provide advantages both in monitoring the quality of agri-food products in an industry 4.0 approach and in proposing green solutions.

Keywords
Vis/NIR device,prototype,environmental impact,sustainability,technological maturation

THEORY & FUNDAMENTALS

P01.15.109

SHORT WAVE-NIR SPECTROSCOPY FOR TRANSMISSION AND DIFFUSE REFLECTION MEASUREMENTS USING INTEGRATING SPHERES: APPLICATIONS IN POWDER AND GRANULAR SAMPLE ANALYSIS

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Near-infrared spectroscopy (NIRS) is a non-invasive, non-destructive optical analytical method widely used in process analytics for the analysis of pharmaceutical, chemical, agricultural, and food products. NIRS offers various measurement techniques, particularly for diffuse reflection measurements of powdered and granular solids [1]. Recent advancements in portable, handheld spectrometers equipped with suitable accessories have enabled their use in process and mobile lab analytics. For several years, a commercial handheld spectrometer with an integrating sphere has been utilized for the quantitative analysis of cereal grains using discontinuous intra-cavity measurements in the SW-NIR spectral range [2].

For intra-cavity measurements, an NIR-transparent sample holder was used to position samples within the center of the integrating sphere. For optically thin layers of solid materials, with the radiation penetration depth exceeding the layer thickness, a transmission measurement is realized. With every part of the sample interacting with the incident radiation, it becomes possible to determine the total sample mass from the recorded spectra. In transmission measurements with small sample absorption coefficients, absorption in so-called Elterman units is expected to be proportional to the analyzed sample mass [3]. For layer thicknesses exceeding the photon penetration depth, the concept of hidden mass arises. Transmission measurements using integrating spheres are well-suited for quantitative spectroscopy. Applications include areas from pharmaceutical, chemical or food industries. We utilized this setup to investigate photon penetration depths in powdered and granular materials.

Furthermore, integrating spheres also allow for diffuse reflection measurements with a specific segment of the sphere containing an IR-transparent window for sample presentation. Two different sample illumination methods are available for this setup [4]. In the first approach, the radiation source is directed directly onto the sample, a configuration commonly found in the literature [5]. The second approach involves directing the radiation source at the wall of the integrating sphere or a baffle, causing the sample to be illuminated indirectly by diffusely scattered radiation. In particular for continuous process monitoring, this arrangement offers several advantages, which are discussed.

Specifically, the SW-NIR spectral range, accessible with Si-detectors with significant advantages over currently employed spectrometers with InGaAs detectors, was examined particularly in view of photon penetration depths and selectivity for moisture measurements. The objective of this work is to explore the fundamental aspects of integrating spheres for the analysis of powders, with a particle size analysis carried out by laser scanning microscopy, on photon penetration depths, dependent on different wavelength ranges, and consequences for quantitative analysis.

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ACKNOLEDGEMENTS: I would like to express my gratitude to several individuals who have supported this research. My thanks go to Ms. N. Skulimma from the South Westphalia University of Applied Sciences for her dedicated lab work. I am also deeply grateful to Professor H.M. Heise and Professor A. Schaum for their guidance and support. As my doctoral advisors, their expertise and encouragement have been instrumental in advancing my research. Thank you for your support and mentorship.

Keywords
Integrating Spheres,Diffuse Reflection,Short Wave-NIR,Photon penetration depths,Wavelength ranges

P01.15.110

DIFFUSE REFLECTION SPECTROSCOPY OF POWDERS USING DIFFERENT ACCESSORIES WITH A STUDY OF SPECTRA DEPENDENT ON WAVELENGTH DEPENDENT ABSORPTION COEFFICIENTS AND PARTICLE SIZE DISTRIBUTIONS

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For decades now, NIR spectroscopy has been used for rapid and non-destructive quantitative analyses of different powders as available, for example, in the pharmaceutical, food and agricultural industries [1]. A powerful measurement technique is diffuse reflection spectroscopy, which can be applied to disperse samples and powders, requiring little preparation. Diffuse reflectance spectra have been measured using different accessories, i.e. based on reflection optics with a rotational ellipsoidal mirror for focusing back-reflected photons [2], an integrating sphere for intra-cavity measurements and a fiber-optic probe [3]. Spectral measurements were carried out using FT-NIR spectrometers from Bruker Optics (Ettlingen, Germany). The spectra of different powders, such as sugars (e.g., glucose, fructose) and other hydrocarbons (starch, cellulose), lignin and others, for which the particle size distribution was measured by laser scanning microscopy, were analyzed in dependence of different sample thicknesses and sample mass, with the latter especially of interest for intra-cavity measurements within the short-wave near-infrared spectra range, realized with a GrainSense spectrometer, equipped with a linear variable filter and a Si-photo-detector array. The spectra were analyzed for wavelength-dependent penetration depths of the NIR-radiation within the complete NIR spectral range, i.e. from 12000 – 4000 cm-1. Consequences for quantitative analytical chemistry are discussed.

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ACKNOLEDGEMENTS: I would like to acknowledge the support of my colleagues and peers who provided helpful discussions and encouragement.

Keywords
Diffuse Reflection,Particle Size,Absorption Coefficients,Powders

POSTER SESSION B

WEDNESDAY JUNE 11, 2025 **13:15-14:00**

AGRICULTURE & FEED

P02.01.01

NON-DESTRUCTIVE DETECTION OF MICRO-CRACKS IN PROCESSED EGGS BASED ON MULTISPECTRAL IMAGING

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Processed eggs, such as boiled, baked, and smoked variants, often develop micro-cracks that pose a significant challenge to food safety and quality. These micro-cracks can compromise the structural integrity of the eggs, increase the risk of contamination, and reduce shelf life. Unlike raw eggs, which can be effectively inspected using conventional acoustic or candling techniques, thermally processed eggs have solidified interiors and hardened or darkened shells that reduce acoustic signal sensitivity and disrupt light transmission due to optical changes, making inspection more challenging. Therefore, an innovative and effective inspection method is required to detect micro-cracks in processed eggs. This study addresses these limitations by employing multispectral imaging in the near-infrared (NIR) spectral range of 700–1000 nm as a non-destructive method for detecting micro-cracks. The NIR spectral range is particularly effective as its longer wavelength than the visible spectrum allows it to penetrate solidified interiors and darkened shells, thereby enhancing the contrast between intact and cracked regions. Imaging data were acquired using a 3-CMOS multispectral imaging camera covering the 400–1000 nm range, and a deep learning-based image classification model was developed for detecting micro-cracks in processed eggs. The model achieved an accuracy of over 90%, demonstrating its capability to identify even fine cracks that are difficult to detect with conventional techniques. This study demonstrates the potential of employing multispectral imaging in the NIR region for detecting micro-cracks in processed eggs. Furthermore, the application of a deep learning-based classification model paves the way for developing fast and accurate automated inspection systems, ultimately providing consumers with safer and higher-quality processed eggs.

ACKNOLEDGEMENTS: This research was funded by Chungnam National University, Republic of Korea.

Keywords
Micro-crack,Food safety,External quality,Processed egg,Deep learning

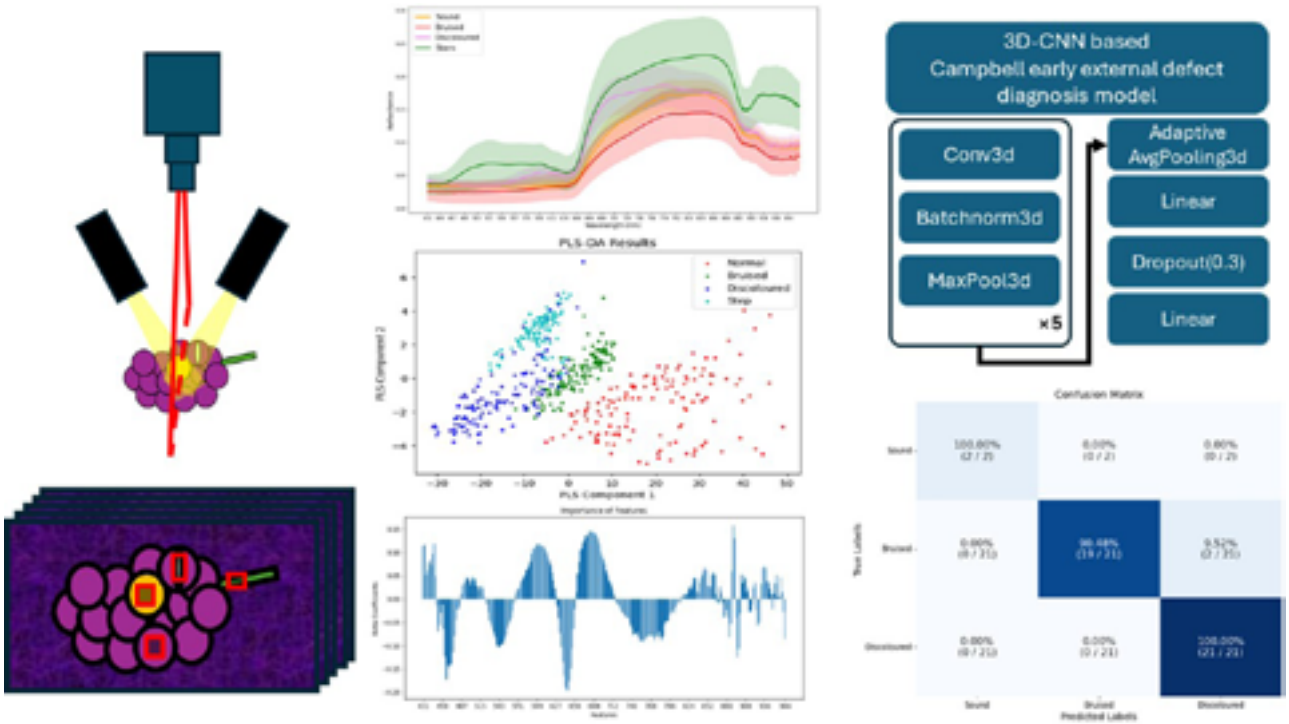
P02.01.02

GRAD-CAM-BASED CNN MODEL FOR EXTERNAL DEFECT ASSESSMENT IN CAMPBELL EARLY GRAPES USING VNIR HYPERSPECTRAL IMAGING

HongGu Lee [1], Jeong-Woo Han [1], Su Ho Tae [1], In-Geun Hwang [3], Cheon Soon Jeong [1,2], Changyeun Mo [1,4]

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Grapes are a globally cultivated crop, extensively grown in Europe, Americas, and Asia, with an economic value of approximately 10.8 billion USD. Campbell Early, a grape variety, was accounted for more than 30% of grape production in the Republic of Korea. The external quality of grapes serves as a crucial and intuitive factor influencing consumers' purchasing decisions. These external quality can be evaluated based on several attributes, such as color, shape, and the presence of defects. Common external defects include berry cracking, discoloration and disease infections. Although external defects can be detected through visual inspection, this method requires substantial labor and is inefficient. This study aims to develop a model for identifying external defects in grapes using Visible Near Infrared (VNIR) hyperspectral imaging and artificial intelligence. The grape samples were harvested in Yeongwol-gun, Gangwon-do, Republic of Korea, comprising a total of 158 Campbell Early grape bunches, including 43 sound, 65 berry cracking, and 50 discoloration. VNIR Hyperspecral imaging system with VNIR hyperspectral camera (microHSI™ 410 Vis-NIR Hyperspectral Sensor, Corning®, USA) was employed to acquire hyperspectral image data of grapes. Hyperspectral images of the grapes were measured from four different sides of each bunch. The spectral characteristics of normal and defective areas in the grapes were analyzed. Based on these spectral features, a deep learning architecture utilizing Convolutional Neural Networks (CNN) was designed, and external defect detection models for grapes were developed. The optimal external defect detection model achieved a classification accuracy of 95%. The GradCAM visualization technique was applied to validate the model by ensuring that the deep learning model's weights focused on defective areas during computation. The results of this study are anticipated to contribute to the development of a VNIR hyperspectral imaging-based system for grape quality assessment.



ACKNOLEDGEMENTS: This research was supported by the Korea Institute of Planning and Evaluation for Technology in Food, Agriculture, Forestry and Fisheries (Project no. 3220525-5).

Keywords
VNIR Hyperspectral Imaging,Grape Quality Assessment,External Defect Detection,Convolution Neural Networks,Campbell Early Grape

P02.01.04

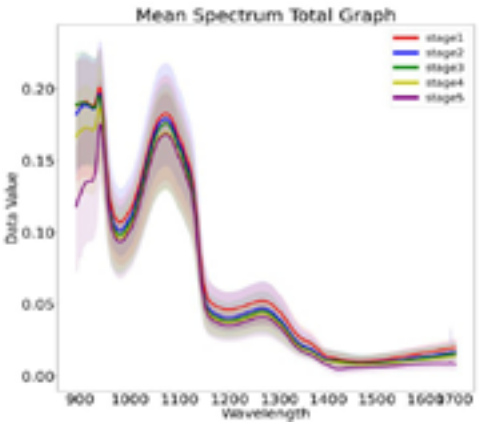
INTEGRATING NIR HYPERSPECTRAL IMAGING AND DEEP LEARNING FOR SHINE MUSCAT GRAPE RIPENESS ASSESSMENT

Jeongwoo Han [1], Jeong-Eun Lee [1], Su Ho Tae [1], Hong-Gu Lee [1], Woo-hyeong Yu [1], In-Geun Hwang [4], Cheon Soon Jeong [1,2], Changyeun Mo [1,3]

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Shine Muscat (SM) is a variety of table grape with high sugar and acidity levels, consumed primarily in Asian countries. The ripeness of SM varies in sugar and acidity and plays a crucial role in consumer purchasing decisions. Currently, the quality of Shine Muscat is assessed based on visual inspection, weight, and sugar content. However, the reliance on these methods individually poses significant challenges in accurately determining ripeness. Therefore, developing a reliable technology for determining the ripeness of SM grape is essential. This study aimed to develop a non-destructive, high-accuracy ripeness discrimination model using near-infrared (NIR) hyperspectral imaging (HSI) and deep learning techniques. The SM grape samples consisted of 70 grapes harvested at five different ripeness levels. NIR hyperspectral image data for SM grape, encompassing both bunch and berry samples, were acquired by NIR HSI system with a line-scan hyperspectral camera (Pika IR+, Resonon Inc., Bozeman, MT, USA in the range of 900 nm to 1700 nm. Hyperspectral images of the bunches were captured from four different sides, while for the berries, 15 berries were selected from each bunch, and hyperspectral images of each berry were taken from three different sides. The acquired hyperspectral images of Shine Muscat were calibrated using a correction equation to extract the spectral data of the region of interest. The extracted spectral data were preprocessed using various methods (Range, Maximum, Mean Normalization, SNV, MSC, Savitzky-Golay 1st- and 2nd- order derivative, Smoothing) to enhance wavelength characteristics and improve model performance. Based on the preprocessed data, ripeness discrimination models were developed using machine learning and deep learning. The SM grape ripeness discrimination model achieved more than 90% accuracy, as evaluated by metrics such as accuracy and F1 score. These results demonstrate the potential of utilizing NIR hyperspectral imaging and deep learning to develop the SM grape ripeness discrimination system.







ACKNOLEDGEMENTS: This research was supported by the Korea Institute of Planning and Evaluation for Technology in Food, Agriculture, Forestry and Fisheries (Project no. 322052-5)

Keywords
NIR Hyperspectral imaging,Shine Muscat graph,Ripeness discrimination,Machine learning,Grape quality assessment

P02.01.05

ASSESSMENT OF PURSLANE (PORTULACA OLERACEA L.) TOTAL OXALATE CONTENT, ASCORBIC ACID, AND TOTAL ORGANIC ACIDS USING NEAR-INFRARED SPECTROSCOPY

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Trakia University, Agricultural Faculty, Stara Zagora, BULGARIA

Research centered on underexploited foods has increased recently. Purslane (Portulaca oleracea L.) is one of the crops that has received the most attention. The plant is a source of nutrients and biologically active compounds including omega-3 fatty acids, alkaloids, flavonoids, cardiac glycosides, protein, carotene, vitamins E and C, etc. The plant may be eaten as a leaf vegetable. On the other hand, purslane has elevated levels of oxalic acid, a component that interferes with the bioaccessibility of minerals such as calcium and iron. The concentrations of nutraceutical compounds vary widely, and precise analyses are required to determine the metabolites present in crops. Developing reliable, rapid, low-cost methods of analysis is essential.

The main objective of this study was to evaluate the possibilities of near-infrared spectroscopy (NIRS) for the estimation of total organic acids, vitamin C, and total oxalate content non-destructively in purslane plants.

The tested Purslane samples were collected from the territory of four Bulgarian floristic regions from June to August 2024. Ascorbic acid in fresh samples was determined by Tilman's reaction with 2,6-Dichlorophenolindophenol titration and total organic acids by titration with 0.1N NaOH. Collected purslane samples were divided into three parts for total oxalate content analysis. One part was measured fresh, the second was blanched and the third was marinated for 30 days. The content of total oxalates was determined spectrophotometrically after their extraction from dry plant material in dilute hydrochloric acid at 100oC. The NIR spectra of tested samples were measured by NIR Quest 512 spectrometer (Ocean Optics, Inc.) in the region 900-1700 nm using a reflection fiber-optics probe. The probe was fixed in the reflection holder to position it perpendicular to the measured surface and at a constant distance to ensure uniform measurement conditions. Several measurements at different parts of the samples were made and averaged to minimize any possible effects of variation in the samples. A Pirouette 4.5 (Infometrix, Inc.) was used for performing spectral data processing. PLS regression was used for the quantitative determination. The calibration equations for each parameter were developed using second derivative spectral data transformation and validated with leave-one-out cross-validation.

Total oxalate content was the biggest in fresh samples and their content decreased as a result of the blanching and marinating. NIRS allowed an accurate determination of total oxalate content in all three types of samples. For each of them, the cross-validated correlation coefficient was bigger than 0.98, and SECV was between 0.20 and 0.42 g/100g DM. Similar accuracy was found for total organic acids and vitamin C determination.

The results of this study demonstrated that NIRS applied with chemometric methods could be used as an alternative to traditional analytical methods for the determination of bioactive compounds directly in the plant matrix without complicated sample preparation and using toxic reagents.

ACKNOLEDGEMENTS: The authors gratefully acknowledge receiving funding from the EU and the Bulgarian Ministry of Education and Science through projects: BG-RRP-2.004-0006-C03 "Development of scientific research and innovation at Trakia University in the service of health and sustainable well-being".

Keywords
Purslane (Portulaca oleracea),total oxalate content,organic acids,near-infrared spectroscopy

P02.01.06

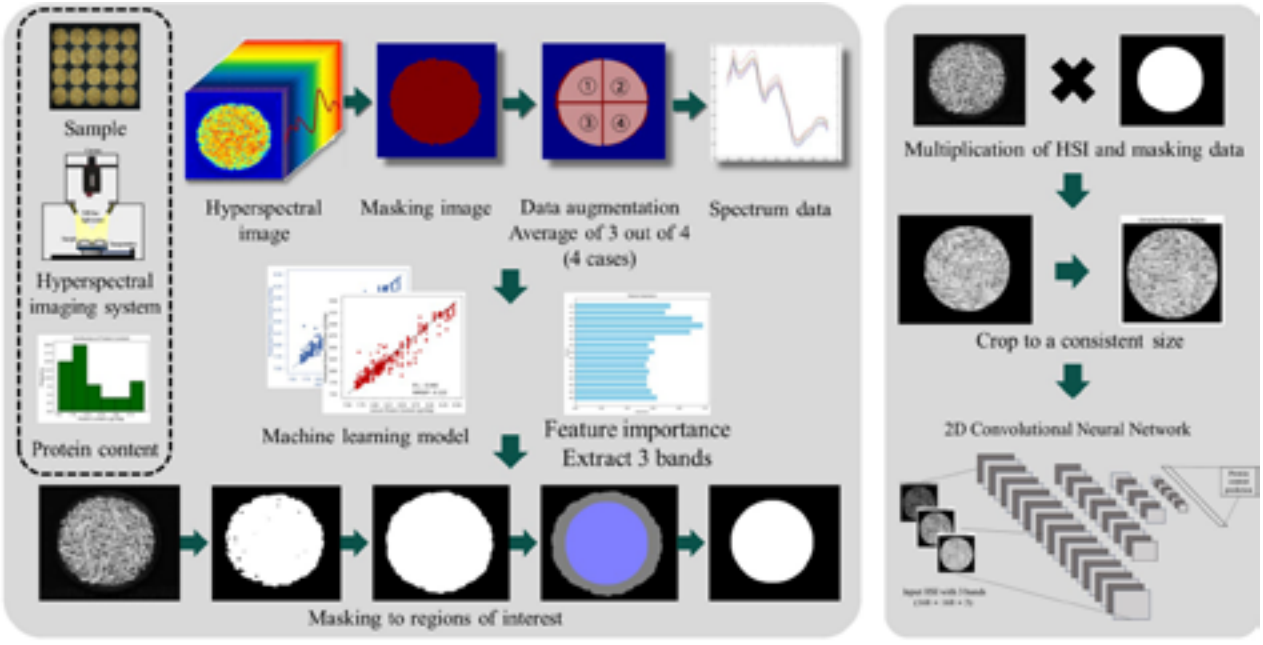
DEVELOPMENT OF NIR HYPERSPECTRAL IMAGING AND 2D-CNN MODEL FOR NON-DESTRUCTIVE PROTEIN CONTENT ANALYSIS OF PADDY RICE

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Rice (Oryza sativa L. subsp. Japonica) is a major food crop consumed by over 50% of the world's population. The protein content of rice, which determines the taste and flavor of rice, is an important indicator for nitrogen fertilizer management and quality. Currently, protein content analysis relies on physicochemical methods, which are time-consuming and economically costly. In addition, the analysis requires the removal of the husk, making it challenging to perform real-time analysis on freshly harvested paddy rice. Therefore, the development of a technology that can analyze the protein content of paddy rice non-destructively and in real time is required. This study aims to develop a model to measure the protein content of rice before milling using near-infrared hyperspectral imaging and a deep learning model.

The samples used in the experiment were paddy rice of the 'Jeonnam 10, Kangdaechan' variety harvested at optimal ripening periods from 14 regions in 2024. The collected paddy rice was sampled with 30 samples from each field, and a total of 420 samples were used for hyperspectral imaging measurements. Hyperspectral images of each sample were acquired using a near-infrared hyperspectral system with hyperspectral camera (PikaNIR-320, RESONON, USA) that can measure in the range of 895–1,720 nm. The protein content was analyzed using the Kjeldahl Method. Hyperspectral images were divided into four regions for each sample and data was extracted. Hyperspectral image correction and spectral preprocessing were applied to remove noise and bias. The preprocessing method used was the first and second differentiation of the Savitzky-Golay Derivatives filter, three types of normalization (maximum, mean, range), Standard Normal Variate, and Multiplicative Scatter Correction. Machine learning model for protein content measurement, including Partial Least Square Regression (PLSR) and Extra Trees Regressor (ETR) were developed using the preprocessed hyperspectral data. In addition, the optimal effective wavelength for rice protein measurement was identified. Based on the selected effective wavelength, a dataset and a two-dimensional convolutional neural network (2D-CNN) model were designed, and a product rice protein measurement model with a performance of R^2 of more than 0.894 was developed. This study demonstrates the potential of combining near-infrared hyperspectral imaging with effective wavelength selection and deep learning techniques to enable rapid and non-destructive measurement of protein content in paddy rice. The results of the study are expected to be important foundational data for the future development of a product rice quality assessment system using hyperspectral images after rice harvest.



ACKNOLEDGEMENTS: This work was partly supported by Innovative Human Resource Development for Local Intellectualization program through the Institute of Information & Communications Technology Planning & Evaluation (IITP) grant funded by the Korea government(MSIT) ((IITP-2025-RS-2023-00260267), 100%).

Keywords
Paddy Rice Protein Content,NIR Hyperspectral Imaging,2D-CNN,Machine Learning,Effective wavelength selection

P02.01.07

NEAR-INFRARED REFLECTANCE SPECTROSCOPY CALIBRATION FOR TRYPSIN INHIBITOR IN SOYBEAN SEED AND MEAL

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Trypsin inhibitors (TI) are naturally occurring antinutritional factors found in soybean seeds [Glycine max. (L.)], that decrease the growth rate of livestock, causing malnutrition and digestion troubles. To mitigate these effects, the feed industry heats the raw soybean and processes it into a meal to denature the TI and provide a nutritious, high-protein feed. The current accurate method to quantify TI levels in soybean seeds or meals is by High Performance Liquid Chromatography (HPLC); however, it is time-consuming, creating bottlenecks in industrial processing. Establishing a near-infrared reflectance spectroscopy (NIR) model for estimating TI in seeds and meals would provide a more efficient and cost-effective method for breeding programs and feed producers. In this study, 300 soybean lines, both seeds and meals, were analyzed for TI content using HPLC, and calibration models were created based on spectral data collected from a Perten DA 7250 NIR instrument. The resulting models demonstrated robust validation, achieving accuracy rates of 97% for seed total TI, 97% for seed Kunitz TI, and 89% for meal total TI. These models can be used by breeding programs to efficiently assess their lines and by industry to quickly evaluate their soybean meal quality.

ACKNOLEDGEMENTS: Funding for this study was provided by the United Soybean Board.

Keywords
Soybean,Trypsin Inhibitor,NIR Calibration

P02.01.08

CNN-BASED METHODS VS. KNN-WEIGHTED PLSR: A COMPARATIVE ANALYSIS FOR PROTEIN AND ADF PREDICTION ACROSS MULTI-PRODUCT FEED

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Accurate prediction of protein content in animal feed is essential for optimizing livestock nutrition and improving feed formulation efficiency. While traditional chemical analysis methods are precise, they are time-consuming and costly, necessitating rapid, non-invasive alternatives such as Near-Infrared Spectroscopy (NIRS).

Deep learning models, particularly Convolutional Neural Networks (CNNs), have demonstrated strong predictive capabilities by effectively extracting spectral features from large datasets. However, their performance compared to local and global calibration methods in multi-product feed analysis remains underexplored. This study evaluates the predictive accuracy of three CNN-based models—CNN, CNN + Partial Least Squares (CNN + PLS), and CNN + XGBoost—against local calibration approaches, including kNN-Weighted PLSR (kNN-LWPLSR) and PLS methods with Global Calibration Models (GCM).

Using a dataset of 3,143 samples from seven different feed products, models were developed using the entire dataset encompassing all sample types, rather than a single feed type. The results in validation (sample n=number) showed that for Protein prediction, kNN-LWPLSR achieved the lowest RMSEP (0.57) and highest RSQ (0.99), while CNN-based models (CNN: 0.62, CNN + PLS: 0.60, CNN + XGBoost: 0.60 with same RSQ: 0.98) demonstrated similar accuracy, outperforming global PLS calibration methods such as GCM (RMSEP: 0.73).

For ADF prediction, kNN-LWPLSR achieved the lowest RMSEP (1.03), while CNN-based models (CNN: 1.38, CNN + PLS: 1.39, CNN + XGBoost: 1.62) exhibited higher errors. The global calibration model GCM (1.44) performed similarly to CNN-based approaches but was less effective than KNN, reaffirming the continued relevance of local calibration techniques for fiber estimation.

As this study was conducted on the entire dataset covering all feed types not for the single type, the results confirm the generalizability of CNN-based approaches for protein prediction in multi-product feed, while reinforcing the effectiveness of local calibration for fiber analysis.

ACKNOLEDGEMENTS: The author gratefully acknowledges the Grant (2021YFD1300300) funded by the National Key Research and Development Program of China.

Keywords
Near-Infrared Spectroscopy (NI,kNN-LWPLSR,Convolutional Neural Networks,Local Calibration,Forage

P02.01.09

EVALUATION OF THE PERFORMANCE OF NEAR INFRARED SPECTROSCOPY PREDICTIVE MODELS FOR FERMENTATION PARAMETERS OF ALFALFA SILAGE BASED ON DRY AND WET MATTER

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BACKGROUND

Alfalfa silage is rich in protein and vitamins, making it an excellent feed resource. However, during market circulation, factors such as weather conditions often make it difficult to maintain fine quality [1]. Therefore, it is necessary to develop rapid and accurate methods for monitoring its nutritional value. Near infrared spectroscopy (NIRS), known for its rapid, non-invasive, and cost-effective nature, has been widely used to predict the nutritional composition of forage, including grass and maize [2,3]. However, research on the differences between fresh and dry samples in NIRS model development remains limited. This study aims to develop NIRS models for various chemical components using both fresh and dry alfalfa silage to evaluate the impact of sample physical properties on model performance.

METHOD

In this study, 88 alfalfa silage samples were collected, representing six varieties, three harvest times, and nne fermentation durations. Silage fermentation indicators - including dry matter (DM), crude protein (CP), water soluble carbohydrate (WSC), neutral detergent fiber (NDF), acidic detergent fiber (ADF), in vitro dry matter digestibility (IVDMD), pH, and organic acids values - were determined through laboratory analysis and used as reference values for fitting against two NIR spectral datasets from dry and wet samples. Each spectrum was obtained by scanning the samples at 1 nm intervals, across wavelengths ranging from 680 to 2500 nm. Spectral data were preprocessed using detrend and standard normal variate (SNV) methods. Calibration and validation of the NIRS models were performed using UCal™ version 4.0 software (Unity Scientific, Brookfield, CT, USA) with partial least squares (PLS) regression.

RESULT

The spectra of dry samples exhibit a more concentrated pattern, while those of wet samples showed greater dispersion. As shown in Table 2, for dry samples, the best model was observed for WSC (R2CrV= 0.958; SECrV = 0.464; RPD = 4.88), followed by NDF (R2CrV= 0.839; SECrV = 1.848; RPD = 2.50). The R^2 value for CP showed a significant decrease after cross-validation. For wet samples, the DM model significantly outperformed other indicators (R2CrV= 0.981; SECrV = 1.671; RPD = 7.21), with strong models also observed for pH (R2CrV= 0.885; SECrV = 0.240; RPD = 2.99) and lactic acid (R2CrV= 0.878; SECrV = 1.710; RPD = 2.84) (Table 3). In comparison, models developed using dried samples generally outperformed those based on fresh samples. Predictions for IVDMD and CP were less satisfactory in both groups.

CONCLUSION

Due to the high moisture content and larger size of fresh samples, which negatively impact NIRS model accuracy, this study suggests using dried samples to predict chemical components (DM, NDF, ADF, WSC) and fresh samples to predict fermentation parameters (pH, lactic acid, acetic acid).

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Table 2. Calibration and cross-validation statistics for NIRS analysis of chemical composition of alfalfa silage (n = 88) based on dry matter basis.

Statistics	CP	WSC	NDF	ADF	IVDMD
N[1]	81	87	82	81	85
Range	6.064	8.59	17.68	7.31	15.07
Mean	20.131	2.753	46.907	21.263	75.683
SD	1.166	2.265	4.621	1.568	3.457
Mathematical treatment[2]	2,8,8	2,16,16	1,8,8	4,16,16	2,16,16
PLS factors[3]	21	15	6	7	21
Calibration					
R^2_C[4]	0.955	0.985	0.889	0.828	0.909
SE_C[5]	0.246	0.278	1.540	0.650	1.040
Cross-validation					
R^2_{CV}[6]	0.315	0.958	0.839	0.752	0.580
SE_{CV}[7]	1.208	0.464	1.848	0.814	2.318
RPD[8]	0.97	4.88	2.50	1.93	1.49
RER[9]	5.02	18.51	9.57	8.98	6.50

[1] Number of samples, exclusive of outliers, used in building the calibration dataset. [2] In mathematical treatments, the first, second, third, and fourth numbers indicate derivative order, gap (nm) over which the derivatives are computed, and derivative smooth, respectively. [3] Number of factors in partial least square equation, which was selected according to the minimum SE_{CV}. [4] R^2_C = coefficient of determination of calibration. [5] SE_C = standard error of calibration. [6] R^2_{CV} = coefficient of determination of cross-validation. [7] SE_{CV} = standard error of cross-validation. [8] RPD = ratio percentage deviation (standard deviation/SE_{CV}). [9] RER = range error ratio (range/SECrV).

Table 3. Calibration and cross-validation statistics for NIRS analysis of chemical composition of alfalfa silage (n = 88) based on fresh matter basis

Statistics	DM	CP	WSC	NDF	ADF	IVDMD	PH	LA	AA
N[1]	83	81	80	84	85	82	84	69	66
Range	36.56	5.58	7.89	19.16	9.14	14.72	2.11	20.3	3.84
Mean	35.463	20.090	2.444	47.143	21.158	75.63	5.106	8.965	1.245
SD	12.054	1.10	1.97	4.563	1.834	3.40	0.718	4.857	0.883
Mathematical treatment[2]	2,16,16	2,8,8	2,8,8	2,16,16	1,16,16	1,16,16	3,16,16	3,16,16	1,8,8
PLS factors[3]	18	1	4	10	10	6	7	9	7
Calibration									
R^2_C[4]	0.997	0.014	0.920	0.901	0.840	0.580	0.926	0.935	0.853
SE_C[5]	0.705	1.093	0.557	1.433	0.733	2.204	0.195	1.239	0.339
Cross-validation									
R^2_{CV}[6]	0.981	0.026	0.870	0.812	0.668	0.374	0.885	0.878	0.757
SE_{CV}[7]	1.671	1.192	0.751	2.009	1.112	2.745	0.240	1.710	0.469
RPD[8]	7.21	0.92	2.62	2.27	1.65	1.24	2.99	2.84	1.88
RER[9]	21.88	4.68	10.51	9.54	8.22	5.36	8.79	11.87	8.18

ACKNOLEDGEMENTS: This study was financially supported by Cooperative Research Program for Agriculture Science and Technology Development (Project No. RS-2024-00398233) Rural Development Administration, Public of Korea.

Keywords
Alfalfa Silage,Fermentation Parameters,Near Infrared Spectroscopy,Predictive Model

P02.01.10

NON-DESTRUCTIVE QUANTIFICATION OF XYLO-OLIGOSACCHARIDES IN FEED ADDITIVES USING NEAR-INFRARED SPECTROSCOPY: ENABLING PRECISION QUALITY CONTROL FOR SUSTAINABLE ANIMAL NUTRITION

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In modern livestock production, oligosaccharides like xylo-oligosaccharides (XOS) are pivotal for enhancing gut health and feed efficiency. Their efficacy hinges on precise formulation: short-chain XOS fractions (xylobiose, xylotriose, and xylotetraose (X2-X4)) exhibit superior efficacy compared to longer chains, necessitating rigorous quality control to ensure consistency and regulatory compliance.

While accurate methods like High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection (HPAEC-PAD) exist, they are impractical for rapid screening in feed additive production. Although near-infrared (NIR) spectroscopy offers a potential solution, current NIR models focus on other oligosaccharides, such as fructo-/galacto-oligosaccharides (FOS/GOS). Despite the advantages of XOS—stability during processing, effectiveness at low doses, and sustainable sourcing from agricultural waste—rapid analytical tools specifically for XOS quantification are lacking. This study addresses this gap by developing a novel NIR model for real-time XOS quantification, enabling scalable, sustainable, and precision-driven quality control.

This study developed NIR calibrations to predict X2-X4 and xylose concentrations in feed additive blends. Approximately 300 sample blends were scanned in duplicate using a benchtop NIR monochromator spectrometer (FOSS DS3, spectral range 850–2500 nm, 0.5 nm resolution). The majority of samples were provided by the manufacturing site of the feed additive blends in the USA. Additional samples were blended at AB Vista Laboratories to ensure a broad range of X2-X4 values and sample matrix diversity. These blends contained XOS at concentrations ranging from 0.1% to 30%, with a mean of 13.1% and a standard deviation of 7.1%. Within one week, these samples were analyzed at AB Vista Laboratory Services using HPAEC-PAD to determine xylobiose, xylotriose, and xylotetraose content, providing the summed X2-X4 and xylose content.

Calibration development was performed using Partial Least Squares (PLS) regression implemented in WinISI software. Spectral pre-processing included Standard Normal Variate (SNV) and Detrend transformations, followed by first-derivative calculation. Outlier detection was based on spectral diagnostics (GH and NH) and subsequent T-value analysis. Calibration performance was evaluated using the standard error of cross validation (SECV) and the coefficient of determination (R^2).

The SECV for the NIR calibrations was 1.45% for X2-X4 and 0.50% for xylose. Independent validation was performed with 25 industrial XOS blends from diverse production batches. Validation results showed strong performance, with a standard error of prediction (SEP) of 1.58% and a very strong correlation with HPAEC-PAD (R^2=0.95) for X2-X4. For xylose, the SEP was 0.63% with a strong correlation with HPAEC-PAD (R^2=0.80).

These results validate NIR spectroscopy as a rapid, non-destructive solution for XOS quantification in feed additives. The method's precision and scalability empower manufacturers to standardize formulations, reduce reliance on costly chromatography, and meet the growing demand for sustainable prebiotics. Future work will expand the sample database, refining the model for global supply chain adoption.

ACKNOLEDGEMENTS: Virginie Rivera, AB Vista

Paul Steen, AB Vista

Xaviere Rousseau, AB Vista

Elias Bungenstab, AB Vista

Nichola Marshall, AB Vista

Keywords
Oligosaccharides,Xylo-oligosaccharides (XOS),Feed additives,Animal Nutrition,Rapid analysis

P02.01.11

ATOMIC SCALE MODELLING OF INFRARED SPECTRA FOR IDENTIFICATION OF VALUABLE MOLECULES IN OLIVE LEAF RESIDUALS

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The biorefinery industry is emerging as a sustainable alternative to fossil-based chemicals, leveraging nature's vast array of substances. However, many of these valuable compounds are neglected and disposed of as agricultural waste. Olive industry residuals, such as olive leaves, are a prime example. Annually, 4.5 million tons of olive leaves are produced worldwide, posing a disposal challenge for farmers and the olive oil industry. This biomass is often underutilized, being burnt in fields, fed to cattle, or combusted inefficiently for energy, leading to unnecessary emissions. The chemical composition of olive leaves varies based on species, horticultural practices, and climate, affecting their suitability for profitable biorefinery conversion. Developing reliable, low-cost, and rapid analytical methods for determining chemical composition directly in the field or sorting line is essential for intelligent, selective, and environmentally friendly assessment of bio-based residuals. NIR spectroscopy offers a promising solution, though understanding prediction success based solely on chemometric models is limited.

This research employs anharmonic modelling of NIR spectra to create detailed chemical maps of characteristic NIR bands in agricultural residuals, identifying their 'NIR fingerprint' in olive leaf biomass. The pilot analysis focuses on oleacein and oleuropein, phenolic compounds abundant in green biomass, including olive leaves. Theoretical studies were compared with experimental spectra from representative olive tree samples with varying oleuropein quantities. Leaf composition was determined through ethanol extraction and HPLC analysis. The results of numerical simulations closely matched the chemometric model's loadings used to predict oleacein and oleuropein contents from FT-IR and FT-NIR spectra. Additionally, a strong similarity between simulated and NIR-derived spectra was confirmed.

ACKNOLEDGEMENTS: Part of the work has been developed within project "Infrared spectroscopy and atomic scale modelling for identification of valuable molecules in agricultural residuals" (acronym AGRO-SPEC), co-financed by the Slovenian Research Agency (ARRS) BI-AT/23-24-024 and Austrian WTZ Project No. SI 26/2023. The authors acknowledge the support through the OLEAF4VALUE project. This project had received funding from the Bio-Based Industries Joint Undertaking under the European Union's Horizon 2020 research and innovation programme under grant agreement no. 101023256.

Keywords
olive leaves,atomic scale modelling,oleacein,oleuropein,biorefinery

P02.01.12

ON-FIELD NIR SPECTROSCOPY USING A COMBINE HARVESTER FOR QUALITY ANALYSIS IN WHEAT BREEDING

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Wheat is a staple crop crucial to global food security. Addressing the challenges of population growth and climate change requires higher-yielding, well-adapted wheat varieties. In breeding trials, assessing multiple quality parameters is essential, with near-infrared (NIR) spectroscopy (NIRS) commonly used to analyze moisture, protein, starch, and fat content [1]. Recent advancements in NIRS have led to smaller, portable devices [2], yet its application in wheat breeding still relies primarily on benchtop systems in laboratories, with limited in-field applications.

This study integrates an NIR spectrometer into a plot combine harvester, enabling real-time, automated grain analysis during breeding trials. Data were collected in-field from 20 common and durum wheat varieties from three Austrian breeders. Initial results reveal compositional differences across varieties and between common and durum wheat. On-field protein models demonstrate good predictive performance (RPD 4.06), while starch (RPD 2.15) and moisture (RPD 2.01) models require further refinement.

During spectra acquisition, 100 spectra per trial plot were recorded and averaged into a single representative spectrum. However, spectral outliers caused by erroneous sample presentation are not removed and thus also included in the averaged spectrum. The influence of these outliers as well as different strategies for automatically identifying and managing them were also investigated during this study.

Future research will focus on validating and refining the existing models using data from multiple years as well as adding new models for additional parameters, such as Fusarium infestation.

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ACKNOLEDGEMENTS: Funded as part of the RTI-Strategy Lower Austria 2027

Keywords
wheat,portable spectroscopy,chemometrics,protein,outlier detection

P02.01.13

A TWO-STEP PROCEDURE FOR COMPARING CLASSIFICATION MODELS BASED ON NEAR-INFRARED SPECTRA

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The growing demand for biofuel and sugar has driven the selection of sugarcane cultivars with higher fiber or sucrose levels. In the first phase of a breeding program, the researcher may be interested in classifying an individual as selected or if it presents a property value above or below a specific threshold. Seeking the most appropriate classification model combined with near-infrared (NIR) spectroscopy is crucial to facilitate the desired selection. It is not uncommon for researchers to consider a unique set of spectra pretreatments before evaluating competitive classification models, looking for the one with the best prediction ability. We hypothesize that this naive procedure would be unfair for model comparisons.

This work details a two-step procedure for a fair comparison of classification models under evaluation. We applied our approach to evaluate three frequently used classification methods, named Partial Least Squares Discriminant Analysis (PLS-DA), Support Vector Machines (SVM), and Random Forests (RF), regarding their performance in predicting the classes of two sugarcane properties from NIR data. Our NIR dataset was derived from 460 sugarcane stalk samples. Initially, a descriptive analysis was carried out to separate the data set into two classes according to fiber (%FIB) and apparent sucrose (%PC) content. Values above the average of the characteristic of interest were defined as class 1 and otherwise as class 2. We have chosen this way of classifying the data for simplicity. However, the researcher could choose to compare the classification models without specifying a threshold value, as in [1], or one could choose another more convenient threshold.

In the first step, the data set was stratified by the Kennard-Stone algorithm [2] into two sets: the training set, containing 368 samples (80%), and the test set, containing 92 samples (20%). The separation was performed individually for %PC and %FIB and made in the same class proportion of the complete set for each characteristic. This first step aims to try different pretreatments on the NIR spectra and choose the most appropriate one for each classification model. The pretreatment selected for each model was used in the second step of our procedure.

In the second step, we separate the 460 samples again into training (89%) and test (20%) sets, but we do it randomly, ten times. By doing so, we could better compare the classification models under an analysis of variance approach [3], followed by a pairwise mean comparison (Figure 1). Table 1 shows the results of each pretreatment applied and the respective cross-validation metrics obtained for the PLS-DA, SVM, and RF models. Each classification model has a different best set of pretreatments applied to the same dataset partition in the first step, which may compromise the model comparison in the second step. For the dataset used in this research, the PLS-DA and SVM were shown to be statistically equivalent and better than the RF method. The crucial result is that choosing the best set of pretreatments for a particular dataset depends on the classification model. The two-step procedure makes it easier for the researcher to infer the many classification models under comparison.

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Table 1. Cross-validation error rates (CV Error) for different pretreatments for classification of apparent sucrose percentage (%PC) and fiber percentage (%FIB) using the Partial Least Squares Discriminant Analysis (PLS-DA), Support Vector Machines (SVM), and Random Forest (RF) models.

		PLS-DA	SVM	RF
	Pretreatment	**CV Error**	**CV Error**	**CV Error**
% PC	None	0.3160	0.3262	0.3851
	MC	0.2912	0.3097	0.3831
	1D + MC	0.2939	0.3110	0.3306
	2D + MC	0.2883	0.3426	0.3397
	MSC + MC	0.3027	**0.2961**	0.3614
	MSC + 1D + MC	0.3033	0.3290	0.3337
	MSC + 2D + MC	**0.2783**	0.3318	0.3367
	SNV + MC	0.3047	0.3290	**0.3257**
	SNV + 1D + MC	0.3048	0.3452	0.3348
	SNV + 2D + MC	0.2858	0.3071	0.3380
% FIB	None	0.2856	**0.2904**	0.3823
	MC	0.2824	0.3110	0.3750
	1D + MC	0.2530	0.3259	0.3415
	2D + MC	0.3043	0.3588	**0.3364**
	MSC + MC	0.2667	0.2964	0.3617
	MSC + 1D + MC	**0.2476**	0.3211	0.3516
	MSC + 2D + MC	0.3104	0.3648	0.3555
	SNV + MC	0.2773	0.2961	0.3515
	SNV + 1D + MC	0.2558	0.3567	0.3564
	SNV + 2D + MC	0.3017	0. 3644	0.3530

Note: The lowest values for each classification method are highlighted in bold. MC, Mean Centering; MSC, Multiplicative Scatter Correction, 1D: First Derivative; 2D, Second Derivative; SNV, Standard Normal Variate.

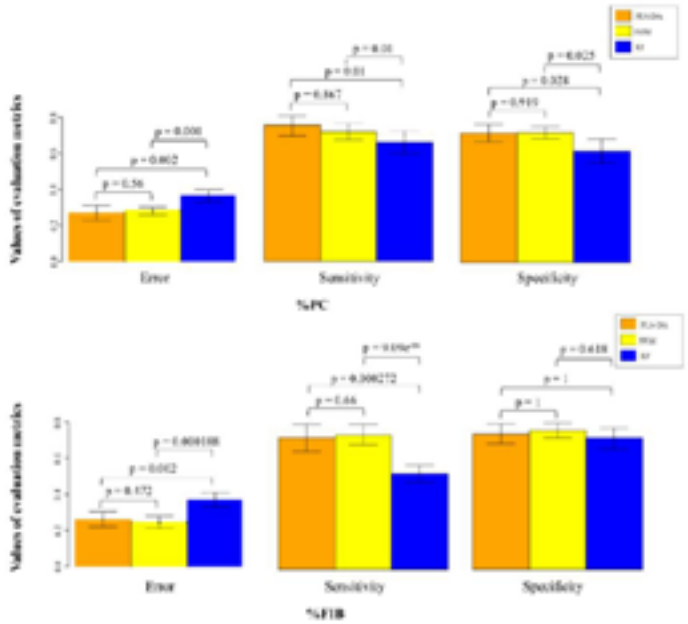


Figure 1. Means and 95% confidence intervals of each model for the evaluation metrics for the %PC and %FIB properties; and the p-value for each pairwise Student's t-test. PLS-DA, Partial Least Squares Discriminant Analysis; SVM, Support Vector Machines; RF, Random Forest.

ACKNOLEDGEMENTS: RIDESA, CNPq, CAPES, FAPEMIG, FINEP.

Keywords
Plant Breeding,Machine learning,sugarcane,fiber prediction,sucrose prediction

P02.01.14

THE NEED FOR VALIDATION OF NIR-PREDICTED FORAGE NUTRITIVE VALUE REVISITED

Miguel Castillo

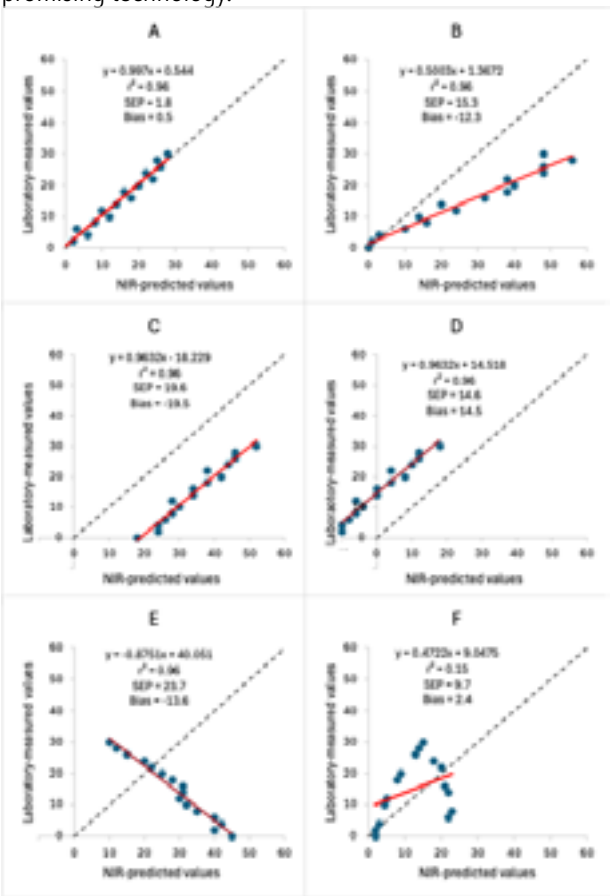
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The adoption of NIR spectroscopy applications is growing rapidly among researchers and non-researchers, and this technological tool is particularly being portrayed as a "turnkey" solution for agricultural products. However, it is essential that all users, especially scientists, remain cognizant of the potential limitations of erroneous NIR-predicted estimates when reporting response variables and treatment effects from experimentation, particularly when relying on third-party or pre-loaded NIR calibrations. Conducting an independent validation—comparing laboratory-measured to NIR-predicted values—is the ultimate standard for assessing the accuracy and reliability of NIR solutions. Using simulated data (Figure 1), we demonstrate how a visual assessment of the relationship between laboratory-measured and NIR-predicted values can reveal inconsistencies often overlooked if relying only on a few fit statistics. Notably, Panels B, C, D, and E in Figure 1 share identical r^2 values; however, the NIR-predicted values differ substantially from the laboratory-measured values with bias ranging from -19.5 to 14.5. Although Panel F has a lower SEP value compared to Panel B, C, D, and E, the relationship is not linear. Panel A represents a desirable outcome.

When reporting NIR spectroscopy as the methodology for forage nutritive value, the following items should be included:

1. A description of the reference analytical methods used to develop NIR models. This information should always accompany the description of the NIR-predicted response variables as it is critical to contextualize the results.
2. Fit statistics of the independent validation exercise, including:
a. The number of samples (n) used in the validation exercise. Ideally, equal or greater than 15-20 samples that represent the population and cover the range of NIR-predicted values.
b. The standard error of prediction (SEP), and bias.
c. The coefficient of determination (r2) and slope and intercept values of the fitted linear regression from laboratory-measured values (y-axis) and NIR-predicted values.

Adherence to these practices ensures robust NIR data quality, paving the way for valid experimental analyses and broader adoption of this promising technology.



Keywords
validation,NIR,handheld,accuracy,turnkey

P02.01.15

PASTURE MONITORING USING PORTABLE NIR SPECTROSCOPY

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Pasture-based livestock farming is a traditional system that facilitates the production high-quality dairy products. Nevertheless, alpine pastures, which are essential for their environmental, cultural, and productive value, are facing the prospect of abandonment (estimated at 11% by 2030) due to the depopulation of mountain areas and the lack of generational turnover. This study focuses on three alpine pastures in Alta Valchiavenna, where the regulations to produce Bitto DOP are followed. These regulations require that cows should primarily be fed on pasture, with a maximum supplementation of 3 kg of dry matter per day. The utilization of rapid and portable technologies, such as NIR spectroscopy, for the monitoring of pasture during the season can offer a substantial advantage in ensuring animal welfare maintaining product quality. Given the high soil and vegetation biodiversity in pastures under study, 10 exclusion enclosures of 16 m^2 each were set up. On each sampling date, biomass was collected from 3 m^2 within these enclosures, with five samplings carried out between late June and late September. The samples were then dried, ground, and analysed using a portable NIR device (MicroNIR VIAVI, equipped with a linear variable filter (LVF) monochromator and a cup accessory to standardize powder analysis). The 2024 samples were integrated with data calibrated on samples collected from the same pastures in 2023, for a total of 94 samples. Calibration and validation sets were defined using the Kennard-Stone DUPLEX algorithm. Predictive models were developed to assess the pasture's ability to meet the nutritional needs of the cows and ensure the maintenance of production. The following parameters were analysed: Crude Protein (CP): RMSEP=0.97, R^2=0.88, RPD=2.91; Neutral Detergent Fiber (aNDF): RMSEP=4.25, R^2=0.91, RPD=3.11; Acid Detergent Fiber (ADF): RMSEP=1.81, R^2=0.89, RPD=2.51; Starch: RMSEP=0.49, R^2=0.50, RPD=1.38; Fat: RMSEP=0.27, R^2=0.81, RPD=2.05. Models were also developed for evaluating nutrient digestibility, including Neutral Detergent Fiber digestibility at 240 hours (aNDFD240): RMSEP=3.09, R^2=0.86, RPD=2.54; Undigested Fiber at 240 hours (aNDFom240): RMSEP=1.39, R^2=0.89, RPD=2.68; Rumen protein Degradability (RPD_NRC01%SS): RMSEP=0.93, R^2=0.86, RPD=2.61. These results demonstrate how the use of portable NIR tools represents a promising solution for monitoring pasture quality during the summer season, enabling precise and accurate vegetation analysis to support animal welfare and management.

Keywords
NIR spectroscopy,Pasture monitoring,Handheld instruments

P02.01.16

DIRECT DETERMINATION OF TEXTURE IN SOIL USING BENCHTOP AND PORTABLE NEAR INFRARED SPECTROSCOPY USING PLS1 AND PLS2 REGRESSION MODELS

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For improving land use management fast and accurate information on soil quality has been required. Therefore, the main objective of this study was to evaluate a regionalized approach for soil texture and organic matter determination using benchtop (1,100–2,500 nm spectral range) and portable (1,100–1,700 nm spectral range) near-infrared spectrophotometers associated with chemometrics methods. Partial least squares (PLS) models were developed with a calibration sample set and applied to a test set collected from same region. These models were used for individual (PLS1) and simultaneous (PLS2) determination of clay, sand and silt. To improve model performance, synergy interval partial least square (siPLS) algorithms were also tested, selecting the most relevant variables from PLS1 models. With the PLS1 model using the benchtop spectrophotometer the root means square error of prediction (RMSEP) of 2.1% was obtained, while the portable spectrophotometer achieved an RMSEP of 2.0%, resulting in ratio of performance to deviation (RPD) of 2.0 and 2.1, respectively, indicating very good predictive performance for clay quantification. For sand quantification, the performance of the PLS1 and siPLS1 models was lower than when the analytes were quantified simultaneously (PLS2), with RPD values of 1.3 and 0.8 for the benchtop and portable methods, respectively. For silt was obtained low predictive performance of the models (RPD < 1.0). Benchtop and portable near-infrared methods have proven to be satisfactory for the direct determination of soil texture. In comparing the calibration models, PLS1 models allow for the development of more specific models and siPLS models offer additional refinement through variable selection. PLS2 models for simultaneous determination showed equivalent performance. Both benchtop and portable methodologies are considered white analytical methods due analytical performance, functionality and sustainable.



ACKNOLEDGEMENTS: To National Council for Scientific and Technological Development (CNPQ) - protocol and 302816/2023-2, Coordination for the Improvement of Higher Education Personnel (CAPES) - code 001, 88881.710390/2022-1 and Foundation for Research of the State of Rio Grande do Sul (FAPERGS).

Keywords
soil,texture,clay,sand,silt

P02.01.17

NON-DESTRUCTIVE POME AND STONE FRUIT PHENOTYPING IN A EUROPEAN PROJECT AIMING AT BREEDING SPECIFIC FRUIT CULTIVARS BETTER SUITABLE FOR ORGANIC AGRICULTURE

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The InnOBreed Project (2022-2026) aims to promote the breeding of organic crops in fruit sector and improve the fruit performance. The objective of one specific task of this project is to deliver high-throughput, efficient and robust tools to evaluate the fruit organoleptic and nutritional quality. As fruit quality evaluation is time consuming, NIR spectroscopy has been chosen as it allows a non-destructive and rapid fruit phenotyping and would contribute to improve the practices in breeding and selection programs of pome and stone fruit trees.

Experiments were carried out in 2023 and 2024 by partners involved in this project, mainly on apple, pear, apricot and peach. According to the partners' expertise, a panel of representative varieties of each species was characterized at harvest and after a period of post-harvest (few days) or storage (apple). Different NIR spectrometers were used, including 4 benchtop and 9 handheld ones. Among quality traits defined as reference data, firmness, color (L*a*b), Soluble Solids Content (SSC, °Brix), titratable acidity (TA) and dry matter content were evaluated for organoleptic traits and content of fibers and polyphenols for nutritional quality.

Results obtained with a handheld spectrometer (F-750 Felix Instruments) on apricot were promising as SSC was predicted with R2 of 0.9, RMSE of 0.86 °Brix and RPD of 3.1. As expected, the prediction was not so good for TA (R2 of 0.77). Overall, the results obtained with this Handheld spectrometer were very close to those obtained with a benchtop spectrometer (Bureau et al., 2009).

The challenge now will be to pool all data in a common database in order to build robust predictive models based on a large variability for each species at a European scale in order to improve fruit phenotyping in breeding programs.

ACKNOLEDGEMENTS: Horizon-CL6-2021-BIODIV-01-12, convention no. 101061028

Keywords
Pome fruit trees,stone fruit trees,near infrared spectroscopy,fruit quality

P02.01.18

THE DAY OF MEASUREMENTS AFTER SLAUGTHERING INFLUENCES THE INFRARED SPECTRUM OF BOVINE MUSCLE

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Introduction

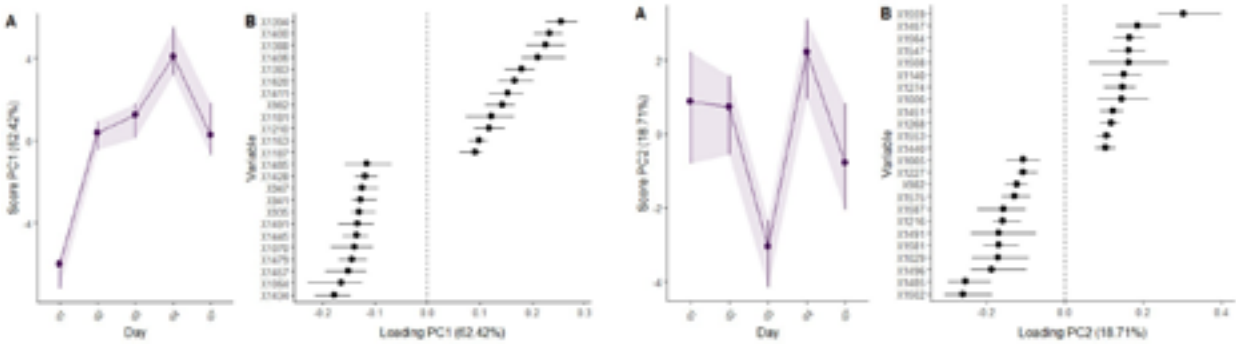
After slaughter, the bovine carcasses are stored at 4°C during 8 to 21 days where they undergo significant biochemical and physical changes, particularly in muscle and fat tissues [1]. These changes during ageing of carcasses could affect the spectra obtained by near infrared (NIR) spectroscopy and lead to the generation of inaccurate results when a prediction or grading model is applied to these spectra. The aim of this study is to determine whether the changes undergone by the carcass during ageing affect the spectra obtained by NIR spectroscopy.

Materials and methods

Near infrared measurements (NIR) were taken from Rectus Abdominis (RA) muscle of carcasses of eleven bovines. After slaughtering, spectra were taken in the same muscle on days 1, 2, 3, 4 and 7. For that, 10 reflectance spectra were measured directly on each sample once they had been wrapped in a plastic food-grade film using a Flame-NIR spectrometer. The instrument scans were run between 935 and 1665 nm every 6 nm. Spectral data underwent a second derivative treatment according to the methodology of [1]. NIR pre-treated spectra were submitted to an Analysis of Variance Simultaneous Component Analysis (ASCA) according to the following model: $Xijk = \mu + \alpha i + Sj + \varepsilon ijk$ where Xijk is the measured response, μ is an overall offset, αi represents the effect of day of measurement, Sj is the effect of the animal and εijk is the error. α was considered as a fixed repeated measured effect and S was considered as a random effect.

Results and discussion

The Figure 1 shows the results of the ASCA model related with the effect of the day of the measurement on the NIR spectra of RA muscle. This model was represented by the first (PC1) and the second (PC2) principal components, which explained 62.5% and 18.7% of the variability in the data set, respectively. Figure 1 illustrates the existence of notable discrepancies between the second derivative of the spectra obtained during the initial stages of the RA ageing process. These discrepancies are particularly pronounced between the spectra acquired 24 hours post-slaughter and those obtained subsequently. The wavelengths identified by the ASCA in the PC1 and PC2 were mainly related to C-H bonds (regions 1160-1200 nm,1227, 1383-1400 nm, 1428-1452 nm) O-H bonds (935-947 nm, 1046-1411 nm) and N-H bonds (1479-1491nm, 1502 nm, 1575-1581nm). The presented bonds illustrate the alterations in proteins, lipids and water that have been documented in the scientific literature [1].



a) First principal component (PC1) b) Second principal component (PC2)

Figure 1. Effect of the number of days after slaughtering on the 2nd derivative near infrared spectra of Rectus Abdominis muscle by ASCA (A, scores; B, loadings). The figure a) corresponds to the results obtained on the first principal component and the figure b) to the results obtained on the second principal component.

Conclusion

The results of this study shows that alterations in the chemical composition of the RA muscle during the initial seven days of the ageing process are reflected in the NIR spectrum. In order to create spectral bases of muscles obtained on different days after the slaughter of animals, it it would therefore be necessary to undergo a spectral standardisation process.

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ACKNOLEDGEMENTS: Funded by the EU. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the EU or the REA. Neither the EU nor the granting authority can be held responsible for them. This work was conducted as part of the Watson project, funded by the EU's Horizon Europe research and innovation programme, under grant agreement No. 101084265.

Keywords
NIR,cattle,meat,ASCA

AQUAPHOTOMICS

P02.02.19

BASED ON AQUAPHMTOMICS STUDY TO MONITOR THE MONITOR THE DRYING PROCESS OF HONEY PILL

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Background

As one of the factors affecting the quality of honey pills (HP), there are few researches on its quality control and solutions. But water can directly affect the appearance, stability and potency of the HP.

Method

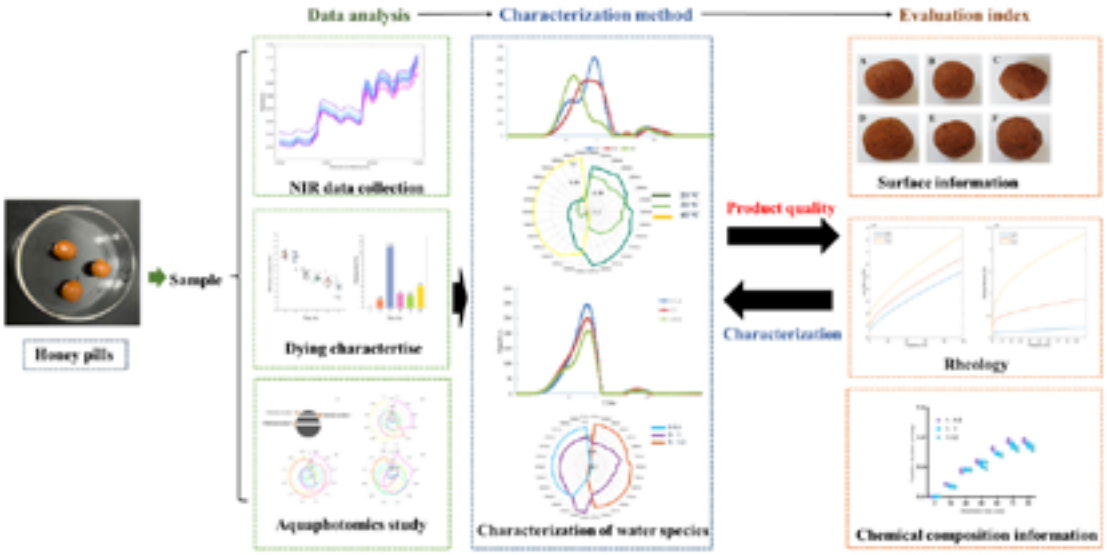
In this study, near-infrared spectroscopy was used to monitor the moisture content of HP with different drying conditions, and multiple linear regression analysis was carried out to establish the moisture content prediction model. The visualization of HP drying process were carried out by aquaphotomics method characterizes the status of the water molecular network at different drying stages. At the same time, low-field nuclear magnetic resonance (LF-NMR) information was carried out to analyze the water distribution in the drying process. We adopted the techniques such as rheology to comprehensively characterize the state of HP, and to evaluate the quality of them by using the dissolution test.

Results

The moisture content prediction model, and the R2 of the model prediction is 0.9532. LF-NMR information further verified the water solvent shell action in the water balance process of aquaphotomics analysis. We used aquaphotomics to demonstrate the process of dispersing water on the surface of the HP into the air and the water inside to the outside. We used in vitro dissolution experiments to verify the true quality of the HP.

Conclusion

The quality state of the pills was analyzed by the aquaphotomics analysis of water species. The above research system provided the scientific basis for revealing the drying process of HP from the viewpoint of aquaphotomics, as well as showed potential for expansion and application to other Chinese traditional medicine products.



ACKNOLEDGEMENTS: Shandong Province natural surface project No. ZR2022MB026. Shandong Province Key Research and Development Program (Science and Technology Demonstration Project) Project No.2022SFGC0104. Key technology research project of Shandong Province NO.2022CXGC020515.

Keywords
Near infrared spectroscopy,Aquaphotomics,Drying process

P02.02.20

AQUAPHOTOMICS STUDY OF NEAR-INFRARED SPECTRA FOR RAPID SKIN EVALUATION

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Objective

The skin is a cosmetically important organ and a key barrier between the body's internal and external environments [1]. Trans-epidermal water loss (TEWL, evaporation) and skin hydration (hydration) are indicators of this function [1]. As skin elasticity declines with age and contributes to wrinkles, maintaining it is essential for anti-aging [2]. This study used near-infrared (NIR) spectroscopy and Aquaphotomics [2,3] (NIR-Aquaphotomics) to compare skin with different barrier strengths and elasticity.

Methods

In this trial, 41 female subjects in their 20s to 50s who gave consent were asked to wash their face using a jell cleanser first, and a foaming cleanse. NIR spectra of both cheeks were measured at 5, 10, and 20 minutes post-wash using a MicroNIR ES 1700 (VIAVI). At each elapsed time, five consecutive NIR spectra were measured without moving the device from the measurement point on the cheek. Evaporation, hydration, and elasticity were measured at the same locations as the spectral measurements just before the 20-minute NIR measurement using a Tewameter, Corneometer, and Cutometer (Courage Khazaka). The experimental environment was monitored using a Thermo Recorder TR-73U (T&D). The acquired NIR spectra were analyzed using Pirouette 4.5 (Infometrix).

Results

Subjects were divided into 4 groups by evaporation and hydration median values (Table 1). Independently, based on elasticity values, subjects were also classified into 3 groups (Table 2).

Aquagrams (Figure 1), showing the absorbance spectral patterns of water [4], revealed that water solvation shells, trapped water, and proton hydrates (1366, 1373, 1385, 1397, and 1565 nm) [3,5] were abundant in both the Hyd L - Evp H and Elas Low groups, while proton hydrates, free water, hydration water, water solvation shells, and water molecular conformations with two to four hydrogen bonds (1342, 1410, 1422, 1435, 1441, 1453, 1466, 1472, 1497 nm) [3] were abundant in both the Hyd H - Evp H and Elas High groups.

Conclusion

Aquagrams showed that when subjects were grouped by evaporation and hydration or by elasticity, their spectral patterns already differed at 5 minutes post-wash. This suggests that, while conventional methods require at least 20 minutes after washing to measure evaporation and hydration, Aquaphotomics analysis of NIR spectra allows that as early as 5 minutes post-wash together with elasticity evaluation, as well.

Future research will clarify skin water structure-function relationships, enhance NIR-based skin prediction, and further develop Aquaphotomics for skin evaluation.

Table 1. Moisture related grouping. The subjects were divided into four groups based on whether their hydration (Hyd) and evaporation (Evp) values were higher (H) or lower (L) when compared with the respective medians (46.60 and 15.73, respectively).

	Group name	Hydration (Hyd)	Evaporation (Evp)
1	Hyd H - Evp L	> 46.60	≦ 15.73
2	Hyd H - Evp H	> 46.60	> 15.73
3	Hyd L - Evp L	≦ 46.60	≦ 15.73
4	Hyd L - Evp H	≦ 46.60	> 15.73

Table 2. Elasticity related grouping. (a) Medians of time – delayed recovery and net elasticity, (b) groups according to their respective elasticity (Elas) values.

(a)

Parameters	High	Low	Median (%)
Time-delayed recovery	> 69.35	≦ 69.30	69.35
Net elasticity	> 68.08	≦ 68.08	68.08

(b)

	Group name	Grouping Method
1	Elas High	Both parameter values are High
2	Elas Mid	Combination of High and Low
3	Elas Low	Both parameter values are Low

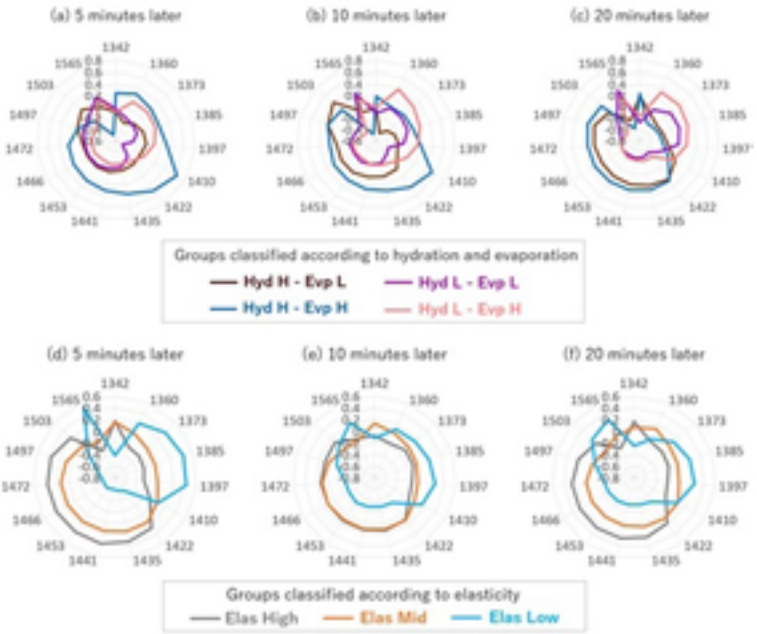


Fig. 1. Aquagrams comparing groups by evaporation, hydration, and elasticity over time. (a)-(c) show four groups by evaporation and hydration medians, and (d)-(f) show three groups by elasticity values.

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Keywords
Aquaphotomics,skin,Aquagrams,near-infrared spectroscopy

P02.02.21

ENHANCING DONATED BREAST MILK ANALYSIS THROUGH AQUAPHOTOMICS

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This study aims to assess the implementation of the Aquaphotomics technique to monitor quality in pasteurized donated breast milk. Aquaphotomics is based on understanding the structure and functions of water and its relationship to vital processes [1]. Applying aquaphotomics to breast milk spectra could be the first step toward creating a database that could serve as a complementary tool for developing feedback systems, where the water's fingerprint acts as a diagnostics tool to provide necessary information for monitoring and diagnosing the biological status of both the mother and the baby.

More than 50 samples of pasteurised donated breast milk from the milk bank of the Hospital Universitario Central de Asturias in northern Spain were used to carry out the study. The measurements were carried out with a portable device (MicroNIR 1700) in the wavelength range 908 – 1720nm. Previous work has shown that it is possible to quantify macronutrients using near infrared spectroscopy [2]. As a first approximation, the samples were graded according to their fat, protein, sugar and energy content. The relationship between breast milk as a system and milk functionality has been investigated using Aquaphotomics. For more exhaustive characterisation of the samples, Specific Water Matrics Coordinates (WAMACs) were defined using subtracted spectra, PCA loadings , SIMCA 's discriminating power and PLS regression vectors. Differences in the respective aquagrams of milk with high, medium and low content of each parameter have been analysed leading towards understanding the relationship between the milk spectral pattern, its water molecular structure and functionality.

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ACKNOLEDGEMENTS: The authors would like to acknowledge the Ministry of Science and Innovation for the project Ref. PID2022-142323NB-I00 and PID2023-149221-B-I00RB-I00 funded by MCIN/AEI/10.13039/501100011033/ and by "ERDF A way of making Europe; Project Ref. IDE/2024/000752, funded by Government of the Principality of Asturias through the Asturias Science, Business Competitiveness and Innovation Agency, SEKUENS. Candela Melendreras acknowledges her PhD grant (BP22-115) from the Asturias Regional Government (Spain).

Keywords
Breast milk,Aquaphotomics

P02.02.22

STUDY ON HYALURONIC ACID AQUAPHOTOMICS-FROM ONE DIMENSION TO TWO DIMENSION ANALYSIS

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1 - Introduction

Hyaluronic acid (HA) is widely used in cosmetics owing to its unique hygroscopic properties. Depending on the molecular weight, it can pass through different layers of the skin by interacting with water. However, there is still no research, especially on non-invasive methods, on the interaction between HA and water structures in the skin. Near-infrared (NIR) spectroscopy has the advantages of being fast, non-destructive, and capable of real-time monitoring. Aquaphotomics based on NIR was introduced to obtain water information, using water as a label-free probe to reflect the interaction between water and solutes. In this study, we developed a fast and nondestructive method for visualizing skin water species based on aquaphotomics to elucidate the mechanisms by which HA affects water species.

2 - Materials and methods

Nine HA samples with different relative molecular weights were selected. The back skin of the Bama pig was used to build the skin model. Active water absorption bands on the skin were identified using chemometric methods. Fig. 1 illustrated the comprehensive experimental framework for the visualization of water species in the skin model.

3 - Results and discussion

To compare the intensity difference of water species within the same group and between different groups in a 2D view, the HSI was processed with the characteristic wavelength assigned in Fig.2. The intensity differences of five water species, S0 (1415 nm), S1 (1446 nm), S2 (1464 nm), S3 (1476 nm), and S4 (1486 nm), on the skin model after the application of different HA solutions.

Fig. 3 illustrated the absorption intensity of five water species on the skin model. The application of different HA solutions caused changes in the distribution of the water species on the skin. In contrast to other water species studies, this research achieved an in-situ analysis without damaging skin tissue. NIR-HSI combined with chemometric analysis could directly extract the distribution and relative intensity of water species in the skin. A1 and A9 showed the strongest signals for the water species. A1 and A9 showed the strongest signals for the water species. This might be because HA1 had the smallest Mr and the best permeability, which led to HA1 entering different layers of the skin to exert hydration during the experiment. However, the Mr of HA9 was high, and after the sugar chain increased to a certain length, the molecule formed a rigid spatial structure in the aqueous solution. At that time, the solvation of water was reduced, effectively reducing the loss of these water species. We could conclude that, in future design endeavors for HA products, it is possible to consider the combination of HA tetraosaccharides and HA at 600~700kDa to enhance the abundance of water species in skin and reduce free water loss. With the help of water species distribution, it could help cosmetics formulators optimize their HA formulations to realize their best hygroscopic properties.

4 - Conclusions

In summary, we developed a visual analysis method to characterize the mechanism by which HA affects water species. In addition, the change rule of water species in the skin was visualized by converting 1D aquaphotomics to 2D aquaphotomics, which could help us quickly and nondestructively understand the interactions between biomacromolecules and water in biological systems.

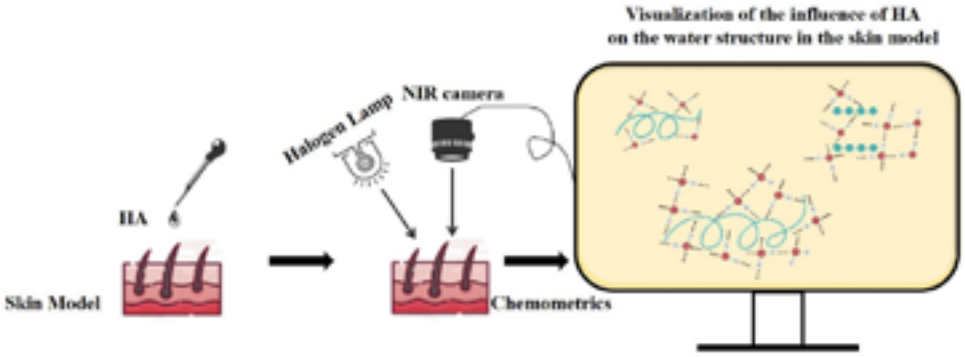


Figure 1 Experimental flow of Aquaphotomics analysis of skin model

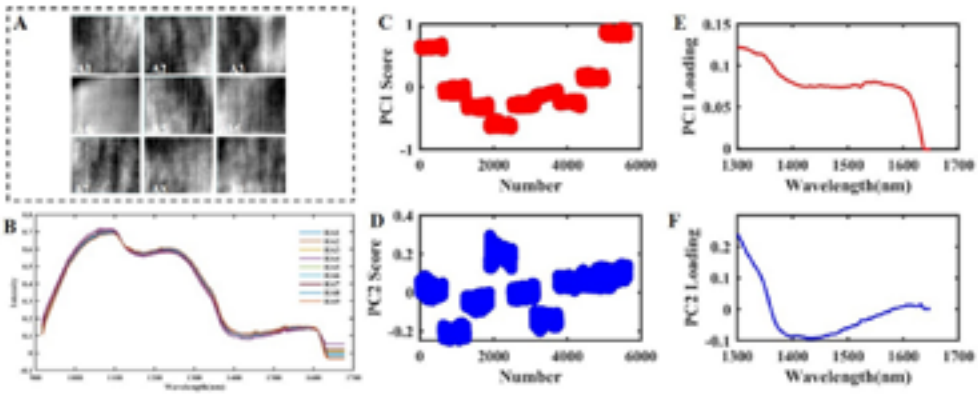


Figure 2 Aquaphotomics analysis of skin model

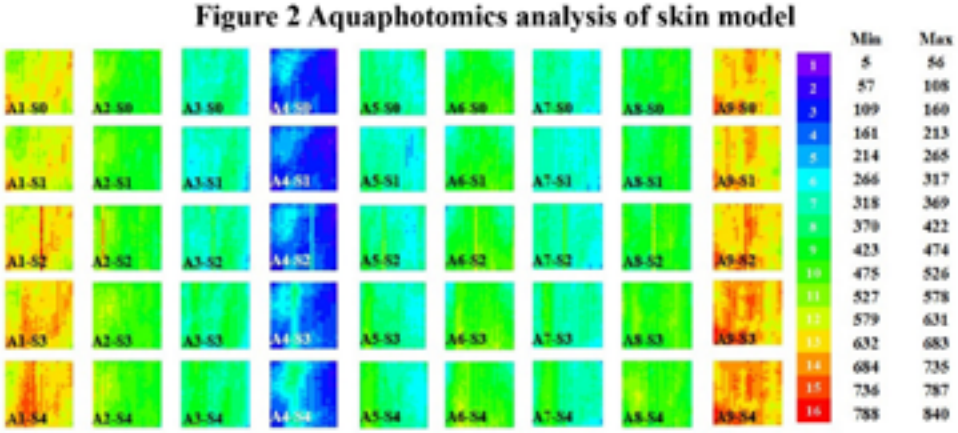


Figure 3 The distribution of five water species on the skin after application of HA1-HA9 aqueous solution

ACKNOLEDGEMENTS: This work was supported by the Key R&D Program (Science and Technology Demonstration Project) program of Shandong Province, China [No. 2022SFGC0104] and Shandong Provincial Natural Science Foundation, China [No. ZR2022MB026].

Keywords
Hyaluronic acid,Aquaphotomics,Hydration,Analysis of images,NIR

CHEMOMETRICS

P02.03.23

DATA AUGMENTATION FOR DEEP LEARNING BASED ON FACTORIAL CHEMOMETRICS METHODS

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Near-infrared (NIR) spectra are often distorted by numerous scattering effects and instrumental artefacts that can obscure useful information. These distortions are even more numerous due to the wide variety of devices and samples. The standard approach in chemometrics to deal with these effects consists in attenuating optical distortions with appropriate preprocessing of spectra. However, models calibrated on tailored processed spectra can include a residual space that is specific to the dataset, thereby compromising their applicability to related but independent datasets. Meanwhile, deep learning (DL) has emerged as a state-of-the-art tool for data analysis, without requiring the design of data-specific preprocessing. A dedicated strategy, data augmentation, has proven to be effective. It consists in enriching the dataset by various techniques, like adding distortion effects and oversampling. To be beneficial to supervised tasks, the augmentations should preserve the coherence between the augmented data and its associated labels [1]. Data augmentation helps to prevent models from overfitting by encouraging them to learn features that are robust or invariant to the induced perturbations. While developing augmentations is relatively intuitive for images, due to their psycho-sensory nature, developing spectral augmentations requires incorporating domain knowledge from human expertise. Reported studies have proposed spectral data augmentation to train neural networks, with the most notable being [2], [3], [4]. For example, the augmentation in [2] is based on Extended Multiplicative Signal Correction (EMSC [5]) which aims to correct spectra from baselines and multiplicative effects. The authors of [2] propose estimating Gaussian distributions for the parameters of the EMSC and then sampling from these distributions to obtain slightly different spectra.

This work investigates various spectral augmentations for training neural networks based on factorial methods that are consistent and well-established in chemometrics. These include decompositions such as EMSC (augmentation proposed in [2]), Principal Component Analysis (PCA), Independent Component Analysis (ICA), Multivariate Curve Resolution - Alternating Least Squares (MCR-ALS). These augmentations are applied to regression (of dry matter in Mangos and soil chemical properties) and classification problems (cultivars of wheat kernel and pharmaceutical tablets). A comparison is made with conventional chemometrics approaches: preprocessing and linear models Partial Least Squares (PLS) and Locally Weighted Partial Least Squares (LWPLS). This work aims to prove that incorporating chemometrics knowledge into data augmentations can improve the performances of DL models, significantly reduce overfitting and be competitive with classical chemometrics methods, particularly with small datasets. It encourages the community to invest in developing DL methods based on domain knowledge and enrich DL thanks to the established approaches in chemometrics.

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Keywords
chemometrics,deep learning,data augmentation

P02.03.24

NEOSPECTRA MODELER: AN AUTOMATED PIPELINE FOR OPTIMIZED CHEMOMETRIC MODEL DEVELOPMENT

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Büchi (Switzerland)

Building robust chemometric models for near-infrared (NIR) spectroscopy is a labor-intensive process, often requiring expertise in spectroscopic analysis, extensive experimentation with outlier detection, variable selection, preprocessing techniques, and dimensionality reduction methods. With the widespread use of handheld spectrometers, NIR analysis has expanded into various domains and industries, increasing the demand for a broader range of models. This growth highlights the need for faster, automated model optimization methods to keep pace with the evolving requirements of NIR spectral analysis.

To address this challenge, we present Neospectra Modeler, an automated pipeline designed to optimize model development and streamline the analytical workflow. The pipeline optimizes key performance metrics, including RMSE, R^2, SEP, and Bias, while offering two levels of optimization: a quick mode capable of generating a robust model in under two minutes and an advanced mode for comprehensive exploration. Additionally, the pipeline provides intuitive visual outputs that allow for identifying outliers, visualizing dataset consistency, interpreting spectral band contributions, assessing parameter optimization, and evaluating model performance across sensors.

The objective of this study is to assess the performance of models developed for three different materials using the Neospectra Modeler. Dry grass samples, measured using the powder cup accessory, were modeled for crude protein (CP) and in vitro organic matter digestibility (IVOMD). Small grain silage, analyzed using a scanner equipped with a rotator, was modeled for fat and protein. Milk samples, analyzed in diffuse reflectance mode, were modeled for fat and protein. The automated pipeline achieved RMSEcv values within ±10% of those obtained from manually developed models, highlighting the tool's efficiency and reliability across diverse materials and measurement setups.

Validation across diverse datasets confirmed the ability of Neospectra Modeler to deliver comparable performance to manually tailored models. By automating model development, the tool not only accelerates the process but also provides a standardized framework for exploring modeling options and generating optimal models. The Neospectra Modeler empowers users to deploy robust, interpretable models while enhancing the scalability of NIR spectroscopy, making it more effective and accessible for real-world applications.

ACKNOLEDGEMENTS: Special thanks are extended to Cesar Diaz, a key member of the Applications Team, for his exceptional support and collaboration. Your insights and commitment have significantly enhanced the outcomes of our efforts.

Keywords
Automatic Model Building,PLS regression,Data handling,NIR

P02.03.25

IMPROVING CLASSIFICATION PERFORMANCE OF WASTE PLASTICS USING A SPACE FILLING DESIGN

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Near Infrared (NIR) spectroscopy is the reference technique for material identification of waste in the recycling industry [1]. It is primarily used to differentiate materials between main polymers used in household packaging, such as polyethylene terephthalate (PET), polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC). Additionally, it can identify fibrous materials like paper and cardboard, or other plastics found in non-household waste streams, such as epoxy in electronic waste.

Classification algorithms process the NIR spectra to identify the waste category. Particular care must be taken in constituting the training dataset to achieve the best classification accuracy. Usually, experts create the training dataset by selecting representative samples, capturing their spectra, and adding these spectra to the dataset. This strategy works well with the K-nearest neighbours (KNN) algorithm [2], widely used in classification problems. However, it can lead to an oversized training dataset, increasing computation time for training and inference without adding relevant information and decreased accuracy.

To address this issue, we propose to select the minimal dataset needed for classification algorithms using space-filling design (SFD), specifically the Wootton, Sergent, Phan-Tan-Luu (WSP) algorithm [3]. We compared this approach on three typical classification algorithms: KNN, linear discriminant analysis (LDA) [4], and partial least squares discriminant analysis (PLSDA) [5]. We preprocessed the data using a second derivative followed by L2-normalization. The data was acquired with a custom NIR spectrometer operating between 1000 nm and 2000 nm consisting of 14 bands at fixed wavelengths chosen for waste analysis and sorting.

To demonstrate the usefulness of our approach, we addressed a complex classification task for the waste recycling industry: distinguishing PET materials between clear transparent bottles (CTB), mono-material trays (MoT), mixed-material trays (MiT), opaque bottles (OB), and opaque bottles with a carbon layer (OBCL). The training dataset comprised 31 spectra manually selected by experts (5 CTB, 8 MoT, 5 MiT, 7 OB, 6 OBCL), while the test dataset consisted of 5190 spectra (1646 CTB, 534 MoT, 500 MiT, 492 OB, 2199 OBCL).

For all classification algorithms, WSP successfully reduced the training dataset size while improving accuracy. For KNN, WSP selected 26 spectra (instead of 31), increasing accuracy by 3%. For LDA, WSP selected 12 spectra, increasing accuracy by 11%. For PLSDA, WSP selected 15 spectra, increasing accuracy by 7%. These results demonstrate that algorithms for creating SFD like WSP can enhance classification algorithm performance by selecting the most relevant spectra for the training dataset. Combined with active learning approaches, this strategy could help to identify the best samples to add to the training dataset without prior label information.

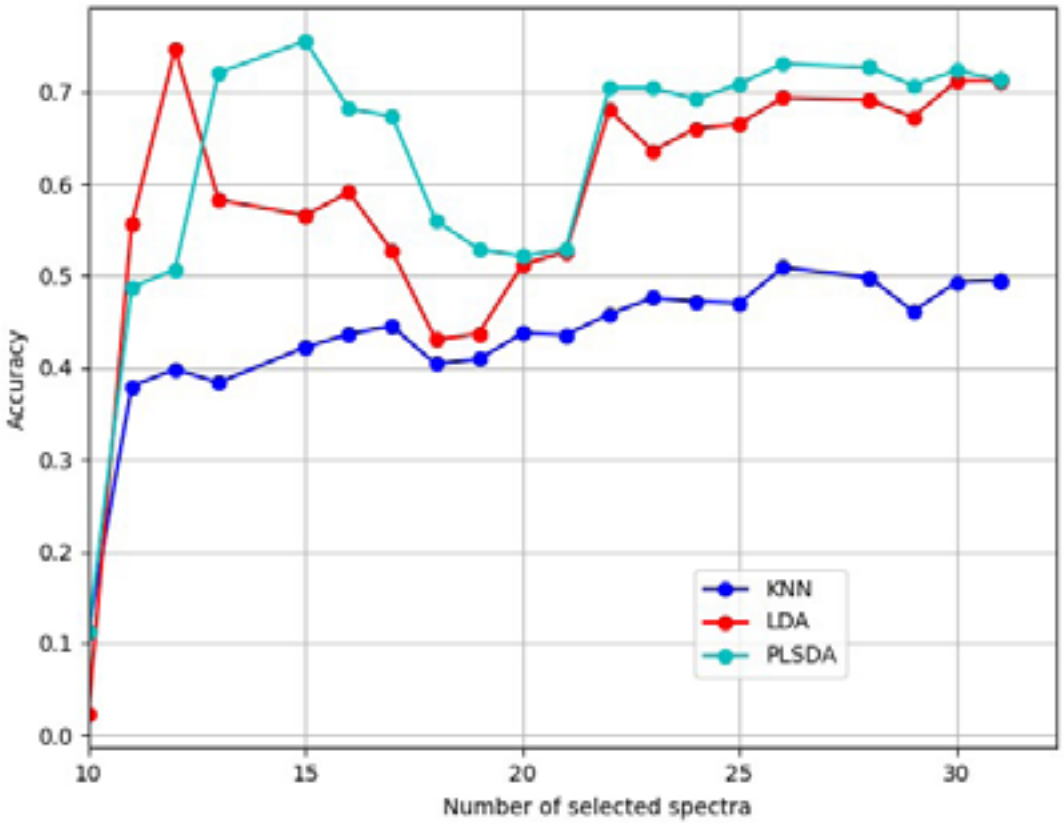
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Keywords
Plastics,Optical Sorting,Classification,Space-Filling Design

P02.03.26

QUALISPECTRA: ADVANCED MACHINE LEARNING ALGORITHMS FOR HYPERSPECTRAL IMAGE ANALYSIS APPLIED TO THE QUALITY CONTROL OF AGRONOMICAL AND FOOD PRODUCTS

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Qualispectra is a new project aiming at leveraging advanced Machine Learning and Deep Learning algorithms to address critical challenges in hyperspectral image analysis for food safety and industrial process optimization. By focusing on performance and computational efficiency, the project aims at delivering real-time, non-destructive solutions for safety insurance and quality control in the agronomical and food sectors. Two key applications are at the core of this research.

The first application involves detecting Fusarium Head Blight in common wheat, a fungal disease that impacts yield, grain quality and mycotoxin risk. Traditional assessment methods are labor-intensive, operator-dependent and time-consuming, highlighting the need for innovative solutions. Hyperspectral imaging offers a non-destructive alternative capable of detecting infection-related changes in the chemical composition of wheat ears. In this study, hyperspectral images of wheat ears were collected using the Specim FX17 camera (1000–1700 nm) in laboratory and field configurations. These images provide both spectral and spatial data enabling accurate identification of Fusarium infection. By leveraging tailored machine learning and deep learning models, this approach could enhance the efficiency of field trials, support the development of disease-resistant cereal varieties, and offer faster, more reliable disease assessments [1].

The second application concerns the homogeneity of flour blends, a critical parameter in the food industry to ensure consistent quality and uniformity. Traditional assessment methods are often destructive, slow and prone to sampling errors. Hyperspectral imaging provides a non-destructive alternative capable of revealing distribution patterns within mixtures. Using data from the Specim FX10 (400–1000 nm) and the Specim SWIR push broom (1000–2500 nm) systems, this study investigates the blending of common and durum wheat flours and of common wheat and spelt flours at various times. Taking full advantage of the spatial dimension of hyperspectral images, Machine Learning and Deep Learning techniques are applied to develop accurate, real-time monitoring solutions that improve blending efficiency and product uniformity.

For both applications, advanced Machine Learning and Deep Learning algorithms were designed to maximize the utility of spectral and spatial information while ensuring computational efficiency. The results demonstrate the potential of integrating hyperspectral imaging with artificial intelligence to address current food safety and quality challenges.

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ACKNOLEDGEMENTS: This project is funded by the Win4Collective – EXERCICES 2024 program of the Walloon Region and spans over 2 years (September 2024 - August 2026).

Keywords
Hyperspectral Imaging,Machine Learning,Fusarium Detection,Flour Homogeneity,Quality Control

P02.03.29

RELYON: A SOFTWARE FOR OPTIMIZING PREPROCESSING STRATEGIES AND VARIABLE SELECTION IN PARTIAL LEAST SQUARES REGRESSION MODELS

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Selecting the best preprocessing strategies for multivariate calibration in spectroscopy is challenging. Most people base their methods on past experiences or trial and error, which can be time-consuming and may not yield optimal results, as it often depends on the specific dataset. Selecting the informative variables is crucial for creating accurate and interpretable models. Recently, a new strategy called Combinatorial Order Preprocessing Search (COPS) has been developed for automatic preprocessing in data analysis [1]. COPS optimizes data preprocessing by efficiently searching for methodology combinations without a predetermined order. It includes prior hyperparameter optimization, which minimizes computational time and enhances accuracy. In 2019, an Ordered Predictors Selection (OPS) update was introduced [2]. The OPS identifies highly predictive variables using informative vectors. Relyon is a user-friendly, standalone tool developed in MATLAB with an intuitive interface for developing optimized multivariate models using COPS, OPS, and Partial Least Squares Regression (PLS). Users can optimize preprocessing by selecting from 21 options and using OPS for variable selection (Fig 1B-C). Preferences can be adjusted, and the best model can be saved for future use. Results can be exported to Excel for easy analysis and visualization (Fig 1B-C). Relyon software can be downloaded for free at www.aclab.ufv.br. To illustrate its capabilities, Relyon was used to develop a PLS model for predicting the sucrose content of ripe bananas using near-infrared spectroscopy. COPS and OPS significantly improved model parameters. In the raw dataset without preprocessing, the model showed a high root mean squared error of calibration (RMSEC) of 24.2, cross-validation (RMSECV) of 37.6, prediction (RMSEP) of 24.4, with correlation coefficients (R) of 0.80 for calibration, 0.54 for cross-validation, and 0.69 for prediction. Implementing COPS alone significantly enhances model performance, reducing RMSEC, RMSECV, and RMSEP to 17.5, 24.0, and 19.3, respectively, while increasing R values. Incorporating OPS enhances the model by selecting key predictive variables, leading to improved accuracy and robustness. This combination achieves the lowest RMSEC (11.0), RMSECV (15.0), and RMSEP (12.0), with the R values of 0.96, 0.93, and 0.93 for calibration, cross-validation, and prediction, respectively, as shown in Figure 1A. These results highlight the importance of preprocessing optimization and variable selection for accurate and reliable predictive models. Relyon helps researchers and professionals to create robust models efficiently, simplifying workflows in spectroscopy or other data-intensive fields.

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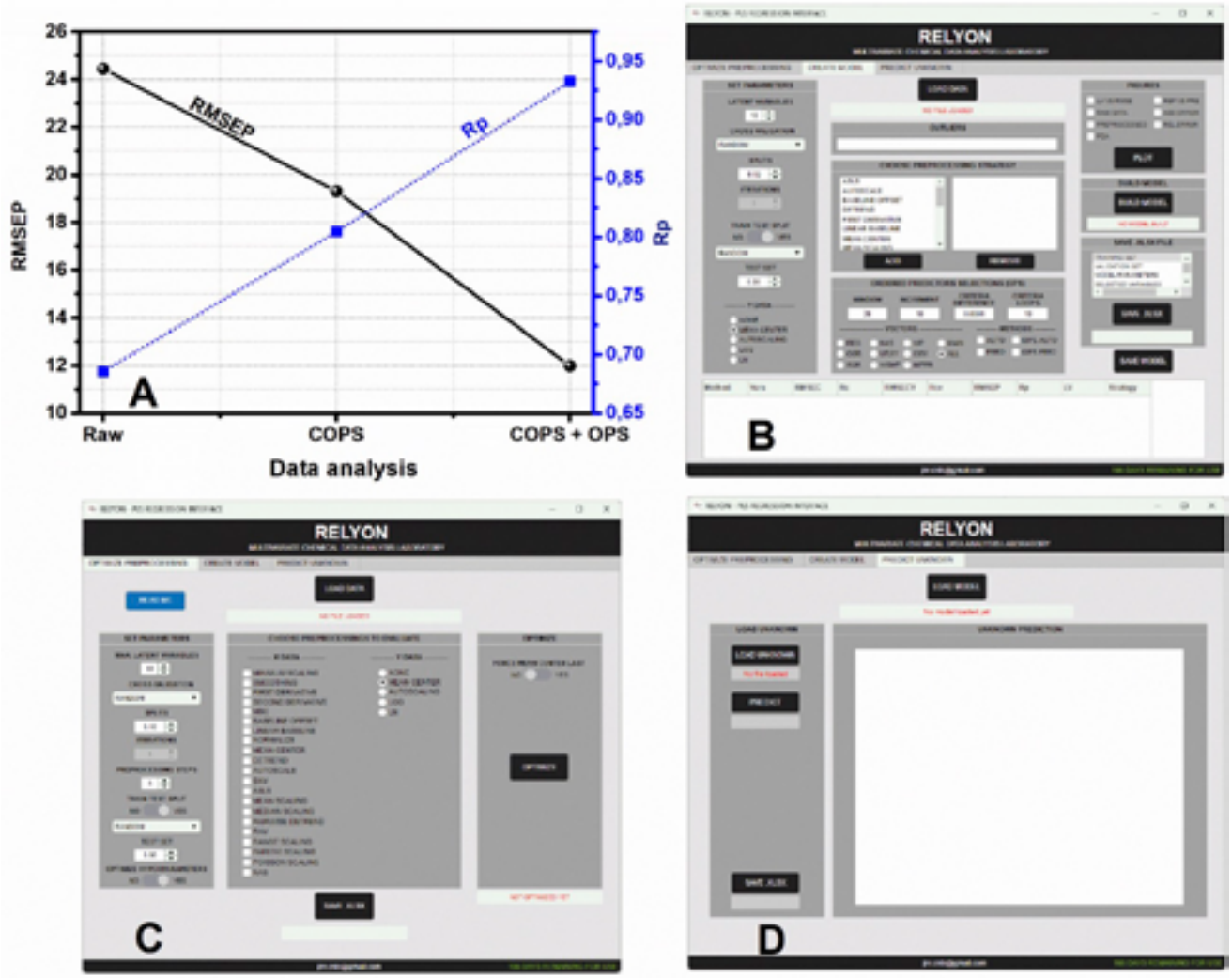


Figure 1. A: Results of the regression model for sucrose in banana. **B:** Interface for defining parameters for building models and loading data. **C:** Interface for defining COPS. **D:** Interface for predicting unknown samples.

ACKNOLEDGEMENTS: This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001, Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG) (Project: CEX - APQ-02254-15), and the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) (Process: 308387/2022-8).

Keywords
Preprocessing Optimization,Variable Selection,Multivariate Calibration,Software Relyon,Banana

P02.03.30

THE USE OF HYPERSPECTRAL IMAGING TO ASSESS PULP REDNESS LEVEL OF BLOOD ORANGES (CV. TAROCCO SANT'ALFIO)

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The global fruit industry continually faces technological challenges to meet the increasing consumer demand for high-quality produce. For hiqh quality blood oranges it's very critical the insurance of intensity and uniformity of the pulp red color, not discernible from the external surface. Nowadays color can be assessed only cutting the fruit and on a limited number of randomly selected fruit, but non-destructive techniques have proved to be effective in predicting internal quality and composition of fruit and vegetables. This study aimed to investigate the potential of VIS-NIR (400-1000 nm) and NIR (900-1800 nm) hyperspectral imaging combined with Partial Least Squares Discriminant Analysis (PLS-DA) and machine learning algorithms including artificial neural networks (ANN), and k-nearest neighbors (kNN) to classify Tarocco Sant'Alfio oranges into two classes of redness, namely Red and Non-Red. A total of 400 oranges were scanned using a hyperspectral imaging system. Following image acquisition, the fruits were longitudinally cut and RGB image of the internal section was taken to assess redness index. Among the algorithms used, the best performance was achieved by PLS-DA. Fruit classification was obtained using the preprocessing of the first derivative combined with the mean center, for VIS-NIR spectra, resulting in classification accuracies of 80% and 80% for calibration and prediction, respectively. Similar performance was achieved in the NIR region reaching 83 and 77% in calibration and prediction, respectively. These results demonstrate the potential of hyperspectral imaging as an effective, non-destructive method for assessing internal color variations in Tarocco Sant'Alfio oranges which can be implemented on selection line for fruit classification.

Keywords
Hyperspectral images,Oranges,VIS-NIR,PLS-DA,Machine Learning

P02.03.31

GOING BEYOND PLS AND IMPLEMENTING SMART WORKFLOWS FOR SMOOTH MODELLING

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Spectroscopy, and especially Near-Infrared (NIR) Spectroscopy, is a well-established and essential tool for quality control. However, the maintaining of existing models and methods and the development of new applications remain time-consuming. After years of adoption in all sectors, companies have accumulated extensive calibration datasets. However, utilizing this data requires expertise and effort and can become too demanding, especially for companies with a network of instruments and only a few experts in a center of excellence to operate them. Meanwhile, standard users often lack the time and expertise to effectively perform complex modeling and validation tasks. These challenges highlight the need for more automated and user-friendly solutions.

The focus of this study is to provide more flexible data handling and simpler procedures with (semi-) automated steps. Here the automated handling of missing reference values is handy and works just as well in the Kennard-Stone data splitting module. Besides the training set for calibration optimization, validation sets can now be defined for a simultaneous check of model performance and robustness.

To effectively handle larger and heterogeneous datasets where traditional methods as PLS show limitations, two new algorithms have been developed. The implementation of these algorithms has been kept user-friendly and as automated as possible, enabling users to easily integrate several types of samples or products into a single model. These algorithms are

- Support Vector Regression (SVR) based on PLS scores [1] for full control for the underlying PLS model and boosting with SVR grid search for finding optimal models.

- A new local regression algorithm based on wavelet compressed spectra [2] is implemented allowing fast and smooth library setup including validation of it.

Details of the modelling flow and the algorithms are presented.

These procedures and algorithms have been implemented in a new commercial calibration software for advanced PLS users, but with more user-friendly capabilities for rapid and better model development.

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[2] A. Deryck, A. Niemöller, V. Baeten, J. A. Fernández Pierna, Another pipeline in local Partial Least Squares Regression (LPLS) methods: Assessing the impact of wavelet transform integration, 2024, Microchemical Journal, 207, 112144, https://doi.org/10.1016/j.microc.2024.112144

Keywords
Multivariate Calibration,PLS,SVR,Local Regression (LR),Kennard-Stone

P02.03.32

DETECTION OF SINGLE-COMPONENT REGION FROM ANY BLIND DATASET WITHOUT PURE COMPONENT SPECTRA

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Finding wavelengths in the near-infrared spectra that are related to a single component is important for enhancing model interpretation and understanding variable causality. For many cases, priori knowledge of sample composition requires extra investigations and chemical usage, and it is difficult to obtain complete spectral profiles of single components in the sample. In our study, a causality analysis that uses functional causal inference was developed as an alternative approach to explore single-component regions in the spectra. Based on the principle of information theory, the method identifies important wavelengths from the difference in mutual dependence of two variable-residual pairs that result from the regression of absorbance on the objective variable and vice versa [1]. Mutual dependence between variables was approximated by distance correlation. The variable causality is robust under different settings of sample collection and can bypass the influence of spurious correlation from mixed sources, allowing the new method to identify important single-component regions for prediction models without the need for extra pure component spectra or test sets. The method was tested on both simulated and experimental datasets. Seven simulated datasets were constructed from theoretical formula of spectral baseline, signal, and noise while experimental datasets were obtained by measuring near-infrared spectra of mixtures containing common solvents (dimethyl sulfoxide, ethylene glycol, and glycerol) at different concentrations. Using index generated from the causality analysis, robust datasets and important wavelengths that contain no spurious correlation could be identified. The method was well responsive in causality-related regions and stable with other spectral treatments such as dimension reduction and derivation. Causality analysis also successfully identified the single-component region while common variable selection indexes, such as variance importance in projection and selectivity ratio, failed to produce comparable results. Complex spectra could be refined into a subset of important wavelengths to build prediction models with high reproducibility and low computational cost. This novel approach suggests that single-component region is potentially detected for any blind datasets without priori knowledge of composition.

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ACKNOLEDGEMENTS: We acknowledge financial support from the Japanese Government (Monbukagakusho: MEXT) Scholarship Program and JSPS Kakenhi [21H02311].

Keywords
Wavelength selection,Causality analysis,Spectral analysis,Single component detection

P02.03.33

SYNERGISTIC FUSION OF INTERFEROGRAM AND SPECTRAL DOMAINS FOR ENHANCED FT-NIR PREDICTIONS

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Büchi (Switzerland)

Fourier Transform Near-Infrared Spectroscopy (FT-NIRS) [1], combined with chemometric methods, has revolutionized analytical and predictive modeling across diverse industries, particularly with the introduction of Micro-Electro-Mechanical Systems Fourier Transform Infrared (MEMS FTIR) technology [2]. MEMS FTIR offers a significant advantage over traditional techniques such as dispersive elements and arrays of detectors by providing both interferogram and spectral data simultaneously. Partial Least Squares (PLS) regression has particularly emerged as a preferred technique due to its proven ability to extract meaningful insights from spectral data for accurate chemical composition predictions. While spectral data has proven effective for analysis, the inclusion of interferogram data offers additional insights that can significantly enhance predictive performance [3].

In this study, we propose a weighted fusion framework, illustrated in Figure 1, that combines the dual acquisition capability of MEMS FTIR of interferogram and spectral data to enhance the predictive performance of FT-NIRS data by integrating multiple modeling approaches. The spectral model, a well-established approach, combines Partial Least Squares regression with optimization of preprocessing techniques and latent variables, forming a strong baseline for comparison. The interferogram model, a novel strategy, leverages advanced interferogram data variants such as raw data, envelopes, normalized data, and smoothed signals, along with data cropping optimizations combined with ridge regression. Additionally, we investigate a spectral features model, a less conventional approach in NIRS, which utilizes spectral feature extraction combined with mutual information-based feature selection thresholds to refine predictions by prioritizing key spectral attributes. By optimizing the respective weights of these individual models, the weighted fusion framework generates an ensemble prediction that harnesses the complementary strengths of each approach.

Experimental results, derived from a dataset of 18 powder mixture samples with four different targets acquired using three sensors, demonstrate notable improvements in prediction metrics. As shown in Table 1, the weighted fusion model achieves a 19.5% improvement in RMSECV, a 4% increase in R^2CV, and a reduction in Bias-to-Error ratio by 50% compared to the best standalone model. These results highlight the advantages of leveraging multiple data sources to enhance NIRS-based predictive modeling, surpassing the performance of conventional single-data approaches.

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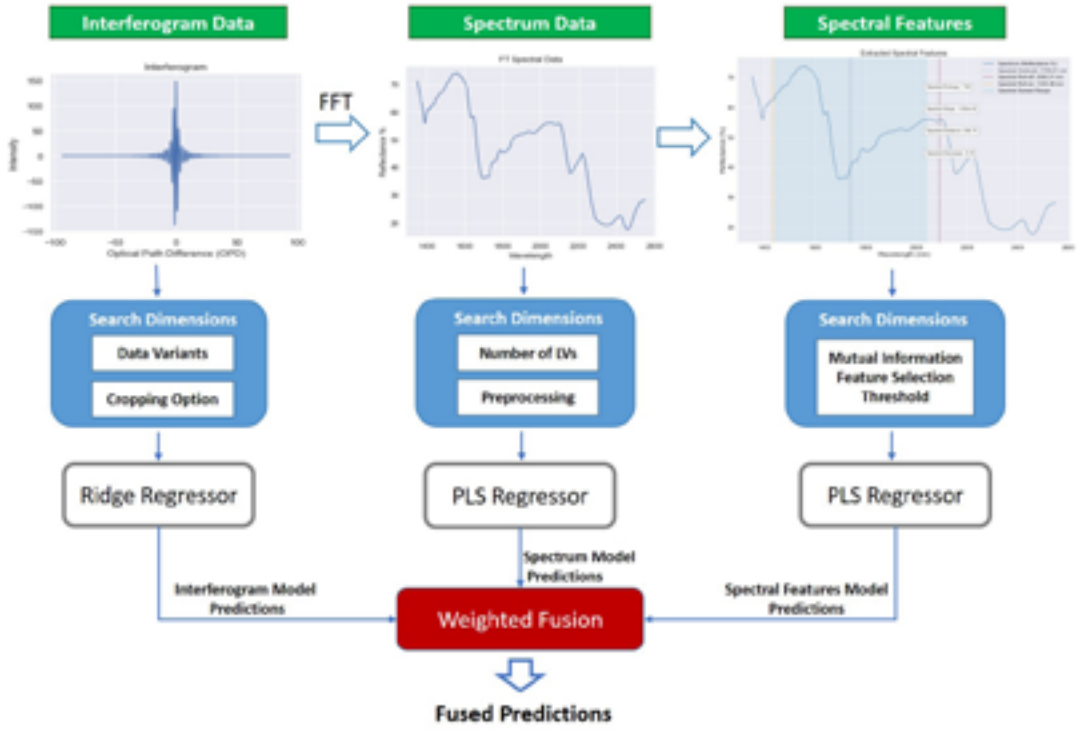


Figure 1: Block diagram illustrating the weighted fusion framework integrating the three base models: Spectrum, Interferogram, and Spectral Features.

Table 1: Comparative analysis for different modeling techniques (Spectrum, Interferogram, Spectral Features, and Weighted Fusion) across four targets (T1 to T4) and their average (AVG). The metrics illustrate the performance of each technique in terms of error ($RMSE_{CV}$), coefficient of determination (R^2_{CV}), and bias-to-error ratio ($Bias_{CV}/RMSE_{CV}$).

Modeling Technique	$RMSE_{CV}$					R^2_{CV}					$Bias_{CV}/RMSE_{CV}$				
	T1	T2	T3	T4	AVG	T1	T2	T3	T4	AVG	T1	T2	T3	T4	AVG
Spectrum	5.31	4.89	1.74	7.97	4.97	0.86	0.92	0.98	0.73	0.87	**0.026**	0.23	0.117	0.11	0.12
Interferogram	5.09	4.45	2.84	9.24	5.4	0.86	0.93	0.95	0.68	0.85	0.1	**0.04**	0.11	0.15	0.1
Spectral Features	5.58	4.83	2.1	**5.57**	4.52	0.85	0.92	0.97	**0.86**	0.9	0.05	0.09	**0.06**	0.11	0.077
Weighted Fusion	**4.49**	**3.55**	**1.85**	6.11	**4.00**	**0.89**	**0.95**	**0.98**	0.82	**0.91**	0.07	0.07	**0.06**	**0.04**	**0.06**

Keywords
Modeling Optimization,Weighted Fusion Framework,Partial Least Squares,Fourier Transform NIR,MEMS

P02.03.34

NEAR-INFRARED SPECTROSCOPY-BASED FINGERPRINTING OF NATURAL POLYPHENOLS AND THEIR ORIGIN USING MACHINE-LEARNING

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Natural polyphenols are playing an increasingly important role as renewable biomass in the substitution of fossil-based resources. Applications ranging from the materials sector to the biomedical and phytosanitary contexts lead to an increased demand and an increased need of a detained understanding of structural key features. The first aspect leads to an increase in production of tannins and lignins, the main representatives of natural polyphenols, and the presence of various technical lignins and tannins of questionable quality in terms of compositional and structural purity. The second aspect becomes important when applications and procedures face approval by the competent authorities. Obviously, both contexts are connected in terms of quality control aspects and the certification of origins and applied extraction processes.

In order to facilitate both structural characterization, otherwise cumbersome, and to introduce a quality control means, an analytical method is developed to perform a characterization (fingerprinting) of natural polyphenols using near-infrared spectroscopy (NIR). Preliminary tests performed on models of various complexity were pursued to determine the optimal conditions for the instrumental acquisition of the spectroscopic signals, in order to minimize the signal noise and maximize the reproducibility of the measurements. Samples of natural polyphenols in the form of both technical lignins and tannins, as well as mixtures of them, also in the presence of common impurities and process remains were analyzed for the subsequent fingerprinting-phase based on the correlation between the spectroscopic signals and structural properties of the polyphenols that were determined independently in house.

Differentiating between classes of polyphenols is possible as well as the identification of mixtures. It was further possible to identify the blending of natural polyphenols with artificial polyphenol-based antioxidants.

ACKNOLEDGEMENTS: NBFC, National Biodiversity Future Center, a project funded under the National Recovery and Resilience Plan (NRRP), Mission 4, Component 2, Investment 1.4, Call for tender No. 3138 of 16 December 2021, rectified by Decree n.3175 of 18 December 2021 of Italian Ministry of University and Research funded by the European Union – NextGenerationEU.

Keywords
natural polyphenols,near-infrared,machine-learning,fingerprinting,quality control

P02.03.35

SUITABILITY OF SELECTED NEAR-INFRARED SPECTROSCOPY INSTRUMENTS FOR DETERMINING CHEMICAL, PHYSICAL, AND MECHANICAL CHARACTERISTICS OF WOOD

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Wood, a material of biological origin, has become a key solution for mitigating the negative impacts of climate change induced by extensive anthropogenic carbon emissions. The importance of this resource is critical, as new technologies enable the conversion of wood into high-performing construction materials, biobased chemical ingredients, innovative packaging solutions, and biofuel for energy generation, among many other applications. However, the available stock of trees is limited, which could lead to an anticipated imbalance between supply and demand in the near future. This necessitates the rational utilization of forest resources by ensuring the circularity of wood transformation and careful optimization of existing value chains. A crucial step towards this is the implementation of knowledge-based solutions, where only properly identified resources are directed towards optimal transformation streams.

The goal of this research was to systematically compare selected NIR-based solutions trained to predict a broad range of material properties relevant for stakeholders in identifying optimal transformation paths. A unique set of wood samples derived from 50-year-old Scots pine (Pinus sylvestris L.) trees was used for spectroscopic evaluation. Both solid wood disks cut from harvested trees and milled chips were presented to spectrometers to acquire spectra representing native and homogenized wood. A customized measurement protocol for these samples was developed using three NIR spectrometers: FT-NIR MPA II (Bruker Optics), portable FT-NIR NeoSpectra (Si-Ware), and portable MicroNIR OnSite-W (VIAVI Solutions). The resulting set of spectra was complemented by hyperspectral images acquired with the FX17 hyperspectral camera (Specim). Data pre-processing and chemometric analysis were performed using the PLS toolbox (Eigenvector) and EVINCE (Predictera).

Reference data for prediction included diverse characteristics of wood that may determine its suitability for downstream conversion. Chemical analyses were conducted to determine the variation of composition within samples, including the determination of extractive components, lignin, holocellulose, and cellulose contents. The determined physical-mechanical properties of the studied wood samples included wood density, modulus of elasticity, static bending strength, and compressive strength parallel to the fibres.

This pilot study compares the effectiveness of spectroscopic methods for the rapid, accurate, and non-destructive phenotyping and prediction of wood quality descriptors that can be used in the forestry/wood industry to optimize the use of Earth's precious natural resources.

ACKNOLEDGEMENTS: Part of the work has been developed within the project "Spectroscopic Methods for Rapid Phenotyping of Trees Reflecting their Ecological Resilience" (acronym DENDRO-SPEC), co-financed by the National Science Centre Poland (NCN) No. UMO-2021/43/I/NZ9/02809 and the Slovenian Research Agency (ARRS) N4-0274. Part of this research has received funding from the European Union's Horizon Europe research and innovation programme under grant agreement No 101082051 (project SINTETIC). Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or European Commission. Neither the European Union nor the granting authority can be held responsible for them.

Keywords
Pinus sylvestris,chemical composition of wood,physical-mechanical properties,wood phenotyping

P02.03.36

MULTIWAY FUSION OF NIR AND EEM DATA FOR THE DETERMINATION OF SYNTHETIC CARAMEL IN BALSAMIC VINEGAR OF MODENA

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Synthetic caramel is among the most widely used colourants in the food industry. The use of caramel in Balsamic Vinegar of Modena PGI is regulated by the protocol of the Consortium of Balsamic Vinegar of Modena [1]. The addition of caramel may be allowed for colour stabilization and must be declared on the product label. Among the various colouring agents, the most commonly used is the Class IV additive, E-150d, also known as sulphite ammonia caramel. However, caramel may also be "naturally" present in balsamic vinegar due to cooked must, used as raw material during vinegar production. Currently, no official analytical method exists to distinguish between sulphite ammonia caramel and "naturally" occurring caramel in Balsamic Vinegars of Modena PGI. To address this, an analytical approach, combining two spectroscopic techniques, Near Infrared Spectroscopy (NIR) and Fluorescence Spectroscopy was employed. Vinegar samples were analysed both in their original state and after the addition of synthetic caramel.

NIR spectra were acquired, pretreated in order to minimize the non-relevant information and submitted to explorative analysis by Principal Component Analysis.

Fluorescence spectroscopy generated three-dimensional Excitation-Emission Matrices (EEMs) (i.e., samples × emission wavelengths × excitation wavelengths), which required a proper data analysis. Specifically, Parallel Factor Analysis (PARAFAC) [2] was applied to resolve and quantify fluorophores associated with caramel. NIR and EEM data will be fused at both low and mid-level, to build a model to determine the presence of synthetic caramel in balsamic vinegar samples from Modena.

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[2] PARAFAC. Tutorial and applications. Bro, R. Chemometrics and Intelligent Laboratory Systems, 1997, https://doi.org/10.1016/S0169-7439(97)00032-4.

ACKNOLEDGEMENTS: We thank the 'Consorzio Tutela Aceto Balsamico di Modena' for providing samples for analysis.

Keywords
balsamic vinegar,caramel,chemometrics,NIR spectroscopy,fluorescence spectroscopy

P02.03.36b

BEYOND THE LAB: UNLOCKING PORTABLE NIR POTENTIAL FOR REAL-TIME QUALITY ASSESSMENT OF DURUM WHEAT KERNELS

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NIR spectroscopy using portable sensors is rapidly gaining popularity due to its aUordability, ease of use, and the increasing availability of instruments that are both cost-eUective and, in some cases, highly performant. This technique oUers the significant advantage of being deployable directly in the field, enabling real-time analysis without the need for extensive laboratory setups. Recent advancements in instrument design and technology have further enhanced its applicability, making it a valuable tool for a wide range of industries. Its on-site usability and potential to streamline workflows make portable NIR spectroscopy an attractive alternative to traditional analytical methods.

However, not all that glitters is gold. Portable NIR sensors come with several limitations, primarily due to their narrower wavelength range and the limited possibility to optimize instrumental parameters compared to benchtop instruments. Additionally, various studies have demonstrated that these sensors are significantly more aUected by external variability factors, such as environmental conditions and operational inconsistencies. As a result, portable NIR devices should not be considered merely as miniaturized versions of benchtop instruments but rather as a distinct category of sensors with their own unique advantages and limitations. Recognizing these diUerences is crucial for their proper application and for maximizing their potential in real-world scenarios. In light of these considerations, this study aimed to assess the feasibility of using portable NIR devices to develop reliable calibration models for quantifying protein and moisture content in wheat samples. A total of 103 wheat samples originating from various regions (Emilia-Romagna, Marche, Tuscany, Puglia, Lombardy, Veneto, Umbria, Lazio, Campania, Molise and Sicily) were analysed. NIR spectra were obtained in the 900-1700 nm range using a poliSPEC NIR (ITPhotonics S.r.l., Fara Vicentino, Italy) and in the 1250-2500 nm range using a NeoSpectra Scanner (Si-Ware, El Cairo, Egypt) portable device, with no sample pretreatment. Signal acquisition involved single-point and continuous modes for the first instrument, while for the second device, variability sources were considered, including operation under battery power versus external power and the impact of diUerent background recording modes. The NIR signal matrices were analysed using Principal Component Analysis to select eUective preprocessing methods for minimizing spectral artifacts and identify outliers. The data were then divided into calibration and validation sets, and Partial Least Squares regression was applied to model the relationship between wavelengths and protein/moisture content.

This study oUers promising applications in industry, enabling faster raw material monitoring and increased sample analysis throughput when integrated directly into processing environments.

CULTURAL HERITAGE & ARCHAEOLOGY

P02.04.37

NEAR-INFRARED SPECTROSCOPY AND HYPERSPECTRAL IMAGING FOR IDENTIFYING LOTUS FIBERS IN TEXTILES

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1. Purpose

Recent studies of cultural properties have identified Lotus fiber threads drawn from the petiole of lotus (Nelumbo nucifera Gaertn.) by microscopic observation and infrared spectroscopic analysis (FTIR) [1]. However, due to the large size of FTIR and concerns regarding risks to cultural properties, we examined whether lotus fibers could be quickly and safely identified by a small near-infrared spectrophotometer (NIRs). As lotus fibers may be interwoven with other fiber types, we also examined whether near-infrared Hyperspectral imaging (HSI) could identify warp and weft fibers microscopically.

2. Methods

(1) 130 natural fiber fabrics or yarns from the 19 to 21st century were prepared, of which 100 were used for modeling and 30 for test data. The diffuse reflection method obtained absorption spectra in 1350-1650 nm (2 nm intervals, average of 10 measurements) using compact NIRs (NIRONE S1.7 by Spectral Engines). For each sample, measurements were taken at three locations.

The spectra data were divided into four groups based on differences in composition: cotton, flax, and ramie (CO, LI, RA); hemp and ohyo (Manchurian elms [Ulmus laciniata]) (Hemp, Ohyo); lotus fiber and basho (Musa balbisiana) (LF, Basho); and silk (SE). Three groups with sufficient sample size were used as test data (CO, LI, RA: n = 16, LF, Basho: n = 5, SE: n = 9). Savitzky-Golay second derivative transformation and SNV (Standard. Normal Variate) were applied to obtain spectra and subject them to partial least squares discriminant analysis (PLS-DA).

(2) Using a line-scan type HSI camera (Hyspex SWIR640 Classic by NEO Corporation), HSI images of a lotus fiber weaving (by Keiko Yamato, Buzen Kokura-ori Research Association) made by imitating an actual cultural asset, were acquired. The wavelength range was 960-2500 nm (spatial resolution: approx. 0.03 mm/pixel). After the second derivative processing of the obtained HSI images, visualized images were created using the PLS-DA model constructed using HSI cubes of one sample from each group (Factor: 7).

3. Results and Discussion

(1) The model discriminating all four groups together (number of components 7, Model 1) had LF, Basho's coefficient of determination R^2 = 0.91, and root mean square error RMSE: = 0.09. After classification by a model that discriminates between SE and other plant fibers (Factor: 3), the discrimination model for only plant fibers (Factor: 7, Model 2) had R^2 = 0.94 and RMSE = 0.09, both discriminating approximately 90% of the samples (Table 1). Analysis of these loading and second derivative of the absorption spectra suggest that the fibers were classified according to the content of lignin and other polyphenols in the fiber.

(2) Fig. 1 shows pixel values in proportion to the possibilities attributed to each group, demonstrating that the pattern is made of lotus threads and the ground of silk. Consequently, we identified the pattern as lotus threads and the ground as silk. Possibly, the pattern's pictorial weft was twin threads of SE and LF (Fig. 1: arrow).

Table.1 PLS-DA results of test samples

Models	CO,LI,RA		LF,Basho		SE		Accuracy (%)
	Correct	Incorrect	Correct	Incorrect	Correct	Incorrect	
Model 1	45	3	13	2	20	7	86.7
Model 2	45	3	14	1	22	5	90.0

* Results with 95 % confidence Level

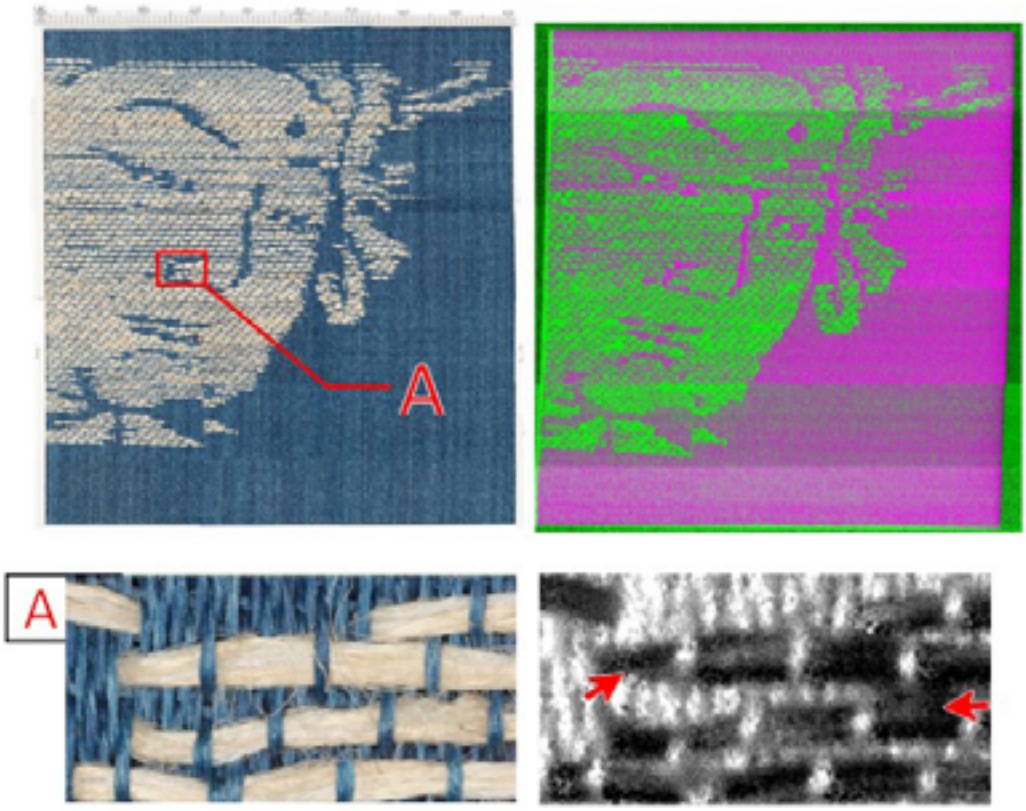


Fig.1 NIR-HSI of a replica made with silk (SE) and lotus fiber (LF); Left: visible image, Middle: enlarged view of A and PLS-DA image (SE: white, red arrow: threads twisted with SE and LF), Right: PLS-DA image (LF: green, SE: purple)

4. Conclusion

The PLS-DA model divided fiber types into four groups, allowing us to classify lotus yarns into LF and Basho groups. HSI could also visualize them. As lotus fibers and Basho can be easily distinguished by observing fiber width under a microscope at low magnification, the combination of near-infrared spectroscopy or HSI and microscopic observation was proven a non-destructive and low-risk method for identifying lotus fibers.

References

[1] Y. Ohashi et al, Bulletin of the Kitakyushu Museum of Natural History and Human History. Series B. History, 2019, 16, 1-14.

ACKNOLEDGEMENTS: This work was supported by JSPS KAKENHI Grant Numbers JP20K00206.

Keywords
near-infrared spectroscopy,Hyperspectral imaging,lotus fibers,textiles

ENVIRONMENT & FORESTRY

P02.05.38

SPECTRAL EVALUATION OF EUCALYPTUS SPP. UNDER WATER DEFICIT USING HSI (VIS-NIR) AND NEAR-INFRARED SPECTROSCOPY: AN ASCA-BASED APPROACH.

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Data analysis in complex biological matrices, such as plants, requires advanced statistical approaches to decompose and quantify the influence of multiple experimental factors. In this context, Analysis of Variance (ANOVA) Simultaneous Component Analysis (ASCA) is presented as a key tool for interpreting variability in spectroscopic studies, providing evidence of effects that may be associated with genetic variability, treatment conditions, genetic and instrumental replicates, among others (Freitag et al., 2024). This methodology allows the identification of response patterns that may go unnoticed in conventional exploratory analyses, facilitating the extraction of relevant information for the study of plant response to adverse environmental conditions.

In this study, the response of different genotypes of Eucalyptus spp. to water deficit was evaluated by hyperspectral analysis (HSI) in the range of 400 to 1000 nm and near infrared spectroscopy (NIR), by portable probe in the range of 1300 to 2500 nm, under controlled conditions. Two experimental trials were carried out (2023 and 2024), in which plants were subjected to periods of water deficit and subsequent recovery, collecting spectral data at different sampling points. The spectral information was analyzed by ASCA revealing that the genetic variability, among the 6 genotypes analyzed, treatment condition (irrigation or water deficit) and different sampling days have a significant effect ($p<0.001$). For HSI (VIS-NIR) data, spectral variability was dominated by the genotypic factor (22%), followed by treatment condition (12%) and sampling days (9%). For the portable NIR data, the main factor was treatment condition (16%), followed by genotypic variation (8%) and sampling days (5%).

These findings highlight the importance of considering the spectral range when interpreting plant responses to environmental stress. HSI proved to be more influenced by genetic variability, as it captures absorption bands related to photosynthetic pigments, while NIR spectroscopy primarily reflects variations in water-related bands, which are more closely associated with different sampling days and the corresponding stress conditions.

Moreover, the results obtained underline the potential of ANOVA-Simultaneous Component Analysis (ASCA) as a robust approach for spectral data interpretation. ASCA enables a quantifiable differentiation of genotypic responses to drought conditions, offering valuable insights for the selection and monitoring of forest species in adaptation programs.

Freitag, S., Anlanger, M., Lippl, M., Mechtler, K., Reiter, E., Grausgruber, H., & Krska, R. (2024). Simplifying Wheat Quality Assessment: Using Near-Infrared Spectroscopy and Analysis of Variance Simultaneous Component Analysis to Study Regional and Annual Effects. ACS Measurement Science Au. https://doi.org/10.1021/acsmeasuresciau.4c00044

HSI (VIS-NIR) – 400 to 1000 nm			
Model effect	**Effect (%)**	**Nums. PC**	**p-values**
Mean	0.0	0	
F1-Genotypes	21.9	5	0.0001
F2-Replicas	0.4	2	0.6699
F3-Sampling Days	8.6	5	0.0001
F4-Treatment	12.5	1	0.0001
Residuals	56.6	2	
Portable NIR – 1300 to 2500 nm			
Model effect	**Effect (%)**	**Nums. PC**	**p-values**
Mean	0.0	0	
F1-Genotypes	7.5	5	0.001
F2-Replicas	0.1	9	0.999
F3-Sampling Days	5.3	5	0.001
F4-Treatment	16.0	1	0.001
Residuals	71.0	2	

ACKNOLEDGEMENTS: Acknowledgment: The authors would like to thank the following projects: ANID national doctoral scholarship n° 21221771, Direccion de Postgrado Universidad de Concepción, Fondecyt 1251201 and EQM230094, ANID- Chile

Keywords
ASCA,Hyperspectral Imaging,Near-Infrared Spectroscopy,Water Deficit,Eucalyptus spp.

P02.05.39

EHNANCED METHOD DEVELOPMENT AND VALIDATION FOR THE ANALYZING BISPHENOL A IN ENVIRONMENTAL WATER USING MICRONIR TECHNOLOGY

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Bisphenol-A (BPA) is an organic compound widely used in the production of synthetic polymers, particularly epoxy resins, due to its good mechanical properties and thermal stability. BPA does not occur naturally and enters the environment through human activities, including manufacturing, wastewater treatment, and landfill leaching. [1,2] It is a toxic, teratogenic, carcinogenic and estrogenic substance, acting as an endocrine disrupting xenoestrogen. [3,4,5] Its presence in surface water is primarily attributed to industrial and wastewater sources, though research of its levels in seawater is limited.

The study aims to develop an innovative method for the identification, discrimination and quantification of BPA in environmental water matrices using near-infrared spectroscopic sensors. Spectra were acquired with a microNIR instrument, a PAT-L probe, operating between 900 and 1700 nm in transflectance, with a thermostatically controlled cell at 40°C, a flow of 2 mL/sec and optical path of 3 mm. Calibration solutions with increasing BPA concentration, were prepared in both distilled water and artificial seawater. Twenty spectra acquisitions were performed in both static and flow modes for each concentration.

The acquired spectra were analyzed using chemometric models. The spectra were first acquired in static mode. Principal Component Analysis (PCA) was employed to explore the data and determine BPA detection capability. The tool effectively distinguished between the presence and absence of BPA. A predictive model for BPA in water was developed using Partial Least Squares Discriminant Analysis (PLS-DA) with excellent classification accuracy in both distilled water and artificial seawater. For quantification, a PLS regression model was developed, demonstrating a good fit in both distilled water and artificial seawater. For the spectra in distilled water and artificial seawater acquired in flow mode, the same procedure was followed: exploratory data analysis was carried out through PCA to assess whether it was possible to determine the presence or absence of BPA in the samples, followed by a prediction model using PLS-DA. An exploratory data analysis was then conducted through PCA was also used to explore the potential for distinguishing samples based on BPA concentration, leading to the construction of a quantification model through PLS, which showed a good fit in both distilled water and artificial seawater.

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ACKNOLEDGEMENTS: The authors would like to thank the Italian National Institute of Health and La Sapienza University of Rome.

Keywords
Bisphenol-A,MicroNIR,Chemometrics,Environmental analysis,Aquaphotomic

P02.05.40

NEAR INFRARED SPECTROSCOPY METHOD SET UP AND VALIDATION FOR ANALYSIS OF ALKYLPHENOLS IN ENVIRONMENTAL WATER SAMPLES

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Alkylphenols are the degradation products of alkylphenols polyethoxylated, molecules which have been extensively utilised in industry due to their amphoteric properties as detergents. Since the 1980s, studies have demonstrated the capacity of AP to mimic the action of 17-beta-estradiol, thereby acting as endocrine disrupters [1, 2]. International legislation has been implemented to strictly regulate the use of these molecules in products and to establish environmental quality standards. Among AP, the two main representatives of the class are the 4-tert-octylphenol and the nonylphenol. 4-tert-isomer is synthesised from trimethylpentane and phenol, whereas NPs is synthesised from an isomeric mixture of nonane. As a result, NPs is an isomeric mixture of 221 constitutional isomers, and the estrogenic potency of NPs depends on its isomeric configuration. Despite the passage of several decades characterised by rigorous regulation within the EU, the United States and, most recently, China, the presence of AP remains a persistent concern in environmental waters. Aquaphotomics [3], a field of study that exploits the lower energy and intensity of the water absorbance in Near Infrared Spectroscopy to apply it in agriculture and food science [4], is a developing area of research. However, the analysis of pollutants in water using NIR is still in its infancy, with a paucity of published research. The present study aims to address the current lack of knowledge in this field and to develop an analytical method for the identification, discrimination and quantitation of 4-NPs and 4t-OP separately or in mixtures in environmental water matrices, using near infrared spectroscopy and chemometric analysis. The approach has the potential to be applied to highly challenging analytical situations, such as the analysis of seawater, where logistic problems are in place, extensive pre-treatment is required and low concentrations are present. The study proposes a rapid and portable technique with no pre-treatment required, capable of detecting even low concentrations (ng/L) as a valid option for analysis of alkylphenols in environmental water samples. Six-point calibration curves were prepared for both analytes separately and in mixture of increasing concentration, in both distilled water and artificial seawater. Spectra acquisition was performed with a microNIR flow instrument working between 900 and 1700 nm, with a thermostatically controlled cell at 40°C, a flow of 2 mL/sec and optical path of 3 mm. Several chemometric tools were used to build and evaluate the model: These included Principal Component Analysis, Partial Least Square – Discriminant Analysis and Partial Least Square Regression. The performance of the model was evaluated in calibration, validation and prediction. The results demonstrate the model's efficacy in discriminating blank samples from spiked ones and in predicting unknown very low concentrationsαααα, with limit of detection and quantification comparable with the traditional gas chromatography mass spectrometry techniques, making NIR a valid alternative for environmental analysis.

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Keywords
Alkylphenols,microNIR,Environmental analysis,Seawater,Aquaphotomics

P02.05.41

NIRS APPLIED TO SCHISTOSOMIASIS EPIDEMIOLOGY: DIFFERENTIATION OF SUSCEPTIBLE MOLLUSC SPECIES

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Schistosomiasis is caused by the trematode Schistosoma spp and is the second parasitic disease with the greatest socio-economic impact in the world, second only to Malaria. Present in 78 countries, it affects around 200 million people, while 700 million live in areas at risk of transmission. This disease is transmitted by a mollusc vector, which acts as an intermediate host. The use of near-infrared spectroscopy to differentiate species of Biomphalaria, the main intermediate hosts of Schistosoma mansoni, which causes schistosomiasis in Brazil and Africa, and in the parasitological diagnosis of molluscs in the laboratory and field has been proven. In this context, this study aims to develop a spectrophotometric differentiation model between specimens of B. glabrata (Barreiro/MG) resistant and susceptible to Schistosoma mansoni, under experimental conditions at the IHPE laboratory - Universitè de Perpignan Via Domitia. A total of 100 specimens of B. glabrata from Barreiro/MG - Brazil were analyzed, 50 of them belonging to a resistant strain and another 50 animals to a strain of molluscs susceptible to S. mansoni infection. These two groups of animals were infected with the S. mansoni strain from Venezuela, kept individually and numbered in the IHPE molluscarium, under the same environmental conditions. In addition to the analysis of live molluscs, 154 samples of 20 µl of hemolymph were also analyzed, 62 samples from control animals and 74 from animals infected with S. mansoni after the 3rd week of infection. The B. glabrata specimens were individually positioned in the equipment window, with the sinister side facing downwards, in order to obtain the spectrum. A slide with a drop of 20 µl of hemolymph was used for the hemolymph. The portable NIR (Spectral solutions MyNIR-R) with a spectral range of 900 to 1700 nm was used for collection. The chemometric analyses were carried out using the Unscrambler® software and pre-processing, PCA and LDA were carried out. The PCA results showed a tendency to separate the two strains of B. glabrata (susceptible and resistant) according to PC-2 with 21% variation and 89.51% separation according to the classification method applied before infection. After the molluscs were infected with the S. mansoni strain (Venezuela), new samples were collected weekly until the 3rd week of infection (pre-patent period). The same trend of separation between the groups was observed, especially between the 2nd and 3rd week of infection (pre-patent period). The results showed that it is possible to diagnose, using NIR, populations of molluscs resistant and susceptible to S.mansoni infection before the elimination of cercariae by the molluscs (gold standard test). The results of using NIR on the hemolymph of the two groups of infected molluscs showed a difference between them in PCA as a function of PC-2, with a variation of 17%. This result expands our methods of analysis, since there are no studies describing the use of NIR in mollusc hemolymph, and shows the biochemical changes that occur in the animal. This study confirms that NIR is an innovative tool for diagnosing the Biomphalaria/S,mansoni relationship, which could lead to be faster and more effective action to control the transmission of schistosomiasis as a public health problem.

ACKNOLEDGEMENTS: CNPq funding code 001

Fiotec- FIOCRUZ- IOC- 008-FIO-22-2-6

Embassy of france for the financing of doctoral student Vanessa Valladares to the University of Perpignan

Keywords
environmental health,diagnosis,health innovation

P02.05.42

RAPID DISCRIMININATION OF BALTIC HERRING STOCKS USING HANDHELD NIR SPECTROSCOPY AND CHEMOMETRIC ANALYSIS OF OTOLITHS

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Baltic herring (Clupea harengus) is a species of significant ecological and economic importance, contributing to both, marine ecosystem stability and commercial fisheries. During seasonal migrations, herring groups from distinct spawning areas aggregate in common feeding grounds, leading to stock mixing. These groups exhibit variations in biological characteristics and population dynamics, necessitating precise stock differentiation for accurate assessments and sustainable fisheries management.

Traditional methods for stock identification, such as otolith shape analysis and morphometric measurements, are well-established but often time-consuming and labor-intensive. The development of alternative, rapid, and efficient analytical techniques is therefore of great interest for improving stock discrimination.

This study investigates the application of handheld Near-Infrared (NIR) spectroscopy combined with chemometric methods, specifically Partial Least Squares Discriminant Analysis (PLS-DA), for the classification of herring stocks based on NIR spectroscopic data of otoliths. The proposed approach achieved discrimination accuracies exceeding 80%, demonstrating its potential as a rapid, non-destructive, and field-deployable screening tool. Integrating this technique into routine sampling protocols could enhance near-at-sea assessments of stock composition in mixed scientific and commercial catches.

A total of over 800 otolith samples were analyzed to examine the mixing dynamics of Western Baltic Spring Spawning (WBSS) and Central Baltic Herring (CBH) stocks using both biological and chemical markers. Given the ecological and economic relevance of Baltic herring, accurate stock identification is essential for effective resource management and the development of sustainable fisheries strategies.

ACKNOLEDGEMENTS: To the NMFRI research project PopHerr and the National Marine Fisheries Research Institute, Gdynia, Poland.

Keywords
handheld NIR spectroscopy,otolith,herring,PLS-DA,stock discrimination

FOOD & BEVERAGE

P02.06.46

MULTI-INFORMATIVE SPECTRAL APPLICATION AS AN IN-SITU TOOL FOR COMPREHENSIVE TRACEABILITY AND SUSTAINABLE MANAGEMENT IN THE SEAFOOD SECTOR

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A wide range of information is needed to maintain traceability in the seafood sector. Rapid and sustainable tools capable of verifying labeling claims and supporting management systems are essential for preventing seafood fraud and addressing economic concerns. Approximately 30-40 squid species hold commercial value, in the European market Illex illecebrosus, Illex coindetii, and Illex argentinus are prominent[1]. Sector challenges include management systems, traceability, store control, overfishing, and the requirements of different stocks. Size and morphometric traits were critical for planning vessel activities, such as programming the fishing time of day and preventing resource depletion. They could also be an important parameter for batch traceability and economic valorisation based on product specifications. This preliminary study describes a case of multi-informative application of portable tools to assess traceability throughout this supply chain, to explore various features and information with the Near-infrared spectroscopy (NIRS) classification. The analysis was based on labeling information and morphometric evaluation of 76 frozen-thawed squid from the three species: 57 samples of I. illecebrosus (19 batches), 11 I. coindetii (3 batches), and 8 I. argentinus (4 batches) were collected from a seafood supply chain hub during March–September 2024 across three fishing areas (n° 27, 37, and 41). Morphometric data, including eight-length measurements[1], were obtained using a ruler, while spectra were collected with a portable NIRS instrument (900–1680 nm, 2 nm resolution). Classification performance was compared between the two approaches, with the evaluation of species, fishing area, and life stage (juvenile/adult) conducted using cross-validation. Principal Coordinates Analysis was applied to morphometric data, while Random Forest was used for NIRS data, and the accuracies (Ac) were assessed for both methods. Morphometric analysis achieved correspondence rates of 57.80% for I. illecebrosus, 72.73% for I. coindetii, and 87.50% for I. argentinus. NIRS approach outperformed morphometric methods, with an overall species identification Ac of 75% compared to 63.16% for morphometric classification. Although NIRS showed strong agreement with the declared species on labeling, morphometric traits revealed high similarity among species. Interestingly, the prediction of fishing areas using NIRS spectra demonstrated a higher Ac (74%), while morphometric traits were less effective in determining the same sample origin (65% Ac). The size parameter value, defined by mantle length (ML), distinguishes the juvenile (<11 cm) from adult stage (>=11 cm) [2]. The NIRS classification of these 2 stages has an Ac of 73%. Morphometric traits can be detected in situ along the supply chain, aiding batch management and species identification. However, these techniques are time-consuming and may be influenced by factors like sex, age, environment, time of day, and requiring specimen integrity[1]. The NIRS portables offer an efficient, sustainable solution for real-time traceability and squid batch management, from vessels to distribution. These findings confirm the NIRS multi-informative potential to verify traceability rapidly, supporting sustainable seafood management.

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ACKNOLEDGEMENTS: Thanks to VRM S.r.l for sampling and data management.

Keywords
squid,labeling,classification,multi-informative application,traceability

P02.06.48

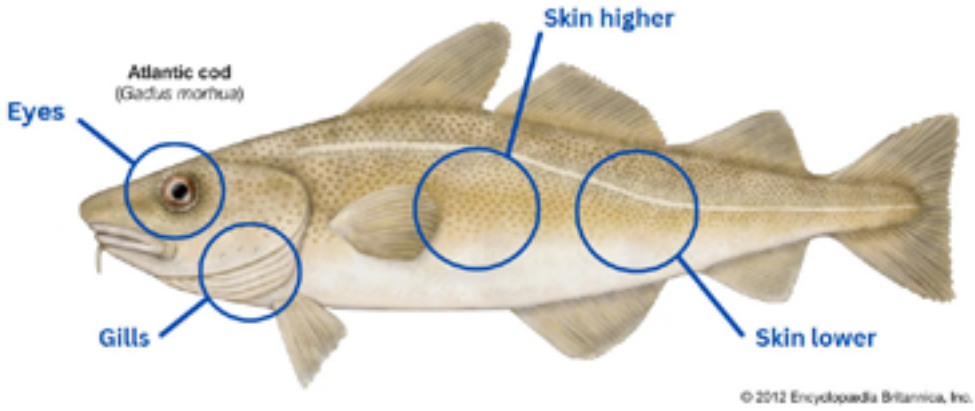
MULTISPECTRAL IMAGING AS A PREDICTIVE TOOL FOR FRESHNESS IN WHOLE ATLANTIC COD: WITH SENSORY, CHEMICAL, AND MICROBIOLOGICAL ANALYSIS

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This study investigated the potential of using multispectral imaging (MSI) techniques to predict the quality of whole gutted Atlantic cod throughout its shelf life during storage on ice. By capturing progressive changes in the optical properties, the aim was to determine whether spectral data can be used to predict sensory evaluation scores, with the Quality Index Method (QIM) method serving as the reference. Furthermore, we wanted to explore whether chemometric methods can enable the development of a non-invasive, real-time approach for assessing the freshness of whole cod. Lastly, we aimed to identify which area of the fish (eyes, skin, or gills) provides the most accurate predictions of sensory scores.

The MSI images were captured using the MSI system, VideometerLab 4 (Videometer A/S, Herlev, Denmark). The VideometerLab 4 combines 19 distinct wavelengths, spanning from 365 nm to 970 nm, into one high-resolution spectral image, where each pixel corresponds to a reflectance spectrum.



During the study, 110 whole gutted Atlantic cod (Gadus morhua) were stored on ice at 0-2 °C for 16 days. Images of the eyes, gills, and two skin regions (Figure 1) were captured throughout the storage period. Concurrent with the MSI imaging, eight reference sampling events were conducted. In these sessions, each cod specimen underwent sensory evaluations by eight trained panelists using the QIM method. This sensory evaluation method records and rates individual quality factors of the whole fish, which are then aggregated into an overall quality index, a metric linearly correlated with storage time on ice, thus providing an estimate of the shelf life and remaining freshness (Martinsdóttir et al., 2001). In addition to sensory evaluations, chemical and microbiological analyses were conducted, including measurements of total volatile basic nitrogen (TVB-N) in the muscle and total viable count (TVC) in the muscle and on the skin.

Segmentation models were developed for each component, gills, skin, and two models for the eyes, the iris, and the pupil, respectively. Normalized Canonical Discriminant Analysis (nCDA) with morphological filtering was used for segmenting the gills, iris and pupils, while a fixed mask was applied for the skin segmentation. Spectral information was extracted from these segmented areas, including mean values and standard deviations of the reflectance spectra. The spectral data was augmented using combinations of wavelengths derived through mathematical transformations.

Predictive models were made to predict the total QIM score (range 0-23). Partial least square regression (PLSR) and Artificial Neural Network (ANN) models were made for all four segmented image areas. The gills had the best-performing model for predicting the total QIM score. The PLSR model achieved a cross-validation coefficient R_CV^2 = 0.86 with a root-mean-square error (RMSECV) of 1.9, while the ANN model performed similarly, achieving R_CV^2 = 0.87 and an RMSECV of 1.2. In comparison, models based on the skin, iris, and pupil performed less effectively than those based on the gills. For the skin, the ANN model achieved R_CV^2 = 0.72 with an RMSECV of 2.9. For the iris, the ANN model had R_CV^2 = 0.72 and an RMSECV of 2.8, while the ANN model for the pupil achieved R_CV^2 = 0.71 and an RMSECV of 2.9. These results suggest that while the gills provide the most reliable predictions, ANN models are generally better suited for predicting relationships between measurements and QIM scores across all regions analyzed.

ACKNOLEDGEMENTS: This work was supported by the 'TraceMyFish' project funded by ERA-NET BlueBio Cofund and the Icelandic Technology Development Fund (project no. 2014156-0611). Special thanks to the team at Videometer A/S.

Keywords
Multispectral Imaging,Sensory evaluations,Fish freshness,Chemometrics

P02.06.49

PORTABLE NEAR INFRARED SENSOR FOR FOOD SAFETY MONITORING: PREDICTING ACRYLAMIDE LEVELS IN BAKED BREAD

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Acrylamide is a neurotoxin classified as probably carcinogenic to humans by the International Agency for Research on Cancer (IARC). It is naturally formed during the baking process of bread through the Maillard reaction between reducing sugars and free asparagine [1]. Therefore, determining acrylamide levels in bread loaves is essential for food safety.

Currently, the most commonly used methods for acrylamide detection rely on conventional chromatographic techniques. These methods are known for their sensitivity and accuracy but are also complex and time-consuming, requiring a sample processing stage to obtain the acrylamide extracts for analysis [2]. Consequently, near-infrared spectroscopy (NIRS) has emerged as an alternative non-destructive analysis method. This technique has been increasingly utilized in recent years for quality control although recent research is beginning to explore its application for safety purposes [3]. Moreover, advancements in technology have reduced the size and cost of NIRS devices, making them practical for on-site determinations.

This study aims to utilize a portable NIRS device to predict acrylamide in bread. While some studies have employed NIRS to predict acrylamide in food products, they have predominantly focused on potato chips [1], where the acrylamide content and the recommended consumption limits are higher, and they usually rely on large laboratory equipment, limiting their applicability in real industrial environments [2]. To the authors' knowledge, this is a pioneering work using a handheld NIRS device to predict acrylamide levels in bread.

In this study, 72 loaves of bread were baked at four distinct temperatures to produce samples with varying acrylamide levels, ranging from low to very high. After baking and cooling, measurements were taken using a portable NIRS device (MicroNIR OnSite, Viavi) operating from 900 to 1650 nm. Specifically, 12 measurements were taken from the top and 12 from the bottom of each loaf, covering all the entire surface, resulting in a total of 24 spectra per loaf. Subsequently, the samples were minced, and extracts were obtained for acrylamide analysis using liquid chromatography–mass spectrometry (LC-MS).

The obtained data was divided into two sets, ensuring that all spectra corresponding to the same loaf were in the same set of samples: 80% of the data was used for calibration, while 20% was reserved for external validation. Before creating the model, NIR signals were pre-processed using various techniques, including SNV with and without detrend, Savitzky-Golay derivatives, mean centering, autoscaling, and different combinations.

A predictive classification model was developed using the partial least squares discriminant analysis (PLS-DA) algorithm. For this purpose, the samples were categorized into three groups based on the EU benchmark level for this product (50 ppb): loaves with low acrylamide content (less than 30 ppb), medium content (from 30 to 80 ppb) and high content (more than 80 ppb). The model demonstrated effective discrimination between these groups, as illustrated in Figure 1.

CALIBRATION

CLASS	SENSITIVITY	SPECIFICITY	PRECISSION
CLASS 1	0.95	0.99	0.99
CLASS 2	0.96	0.97	0.91
CLASS 3	1.00	0.99	0.96

ERROR RATE: 0.03
NON-ERROR RATE: 0.97
ACCURACY: 0.96

CROSS-VALIDATION

CLASS	SENSITIVITY	SPECIFICITY	PRECISSION
CLASS 1	0.95	0.99	0.99
CLASS 2	0.96	0.97	0.92
CLASS 3	1.00	0.99	0.96

ERROR RATE: 0.03
NON-ERROR RATE: 0.97
ACCURACY: 0.96

EXTERNAL VALIDATION

CLASS	SENSITIVITY	SPECIFICITY	PRECISSION
CLASS 1	0.97	0.97	0.97
CLASS 2	0.93	0.99	0.97
CLASS 3	1.00	0.98	0.96

ERROR RATE: 0.03
NON-ERROR RATE: 0.97
ACCURACY: 0.97

This study highlights the potential of NIRS for detecting acrylamide in bread, opening new opportunities for this technology in food safety applications.

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ACKNOLEDGEMENTS: Authors acknowledge the Basque Government–Department of Economic Development, Sustainability and Environment. –Vice. Dept. of Agriculture, Fishing and Food Policy, Directorate of Quality and Food Industries for the funding of the project.

Keywords
Acrylamide,Bread,Near infrared spectroscopy,Portable,Food safety

P02.06.50

BLACK SPOT DETECTION IN POTATOES WITH SPECTRAL IMAGING IN THE VISIBLE AND NEAR-INFRARED RANGE

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When potatoes collide with each other or with harvesting machinery they can develop black spot. Detection of these affected potatoes is necessary to guard the quality. However, detection from the outside is impossible with the naked eye since it manifests itself in the peripheral region of the tuber cortex. Therefore, spectral imaging in the 400-1000 nm range has been proposed to detect the black spot in a non-destructive manner. Three different sample presentation modes were explored: transmittance, interactance and reflectance. Black spot was induced in six different cultivars with different incubation times. Partial least squares discriminant analysis and support vector machine methods were used for building pixel classifiers to predict whether the tissues are black spot-affected. This research expanded the knowledge about the detection of black spot in potatoes already present in literature [1] since it researched the three sample presentation modes and used six different cultivars.

The resulting models are compared to each other with the help of sensitivity, specificity and accuracy metrics. The classifiers fitted on the data, reveal that interactance mode is most suitable for detecting black spot. The models were not able to distinguish fresh tissue from black spot-affected tissue when the measurements were taken in transmittance mode. If they were able, it was suspected that overfitting took place. Besides this, the partial least squares classifiers did not perform as well as the support vector machines, which indicates a non-linear relationship between the presence of black spots and the resulting spectra of the potatoes. Cultivars where black spots form close beneath their peel were found to have better detection accuracy than cultivars in which the black spot occurred deeper in the tissue. It is also found that the structural changes due to the bruising are important for detection, which indicates that early detection might be possible. Since black spot is the result of an enzymatic reaction and thus, some time is required for the colour of the spots to form. However, the structural chances are a direct result of the bruising and can thus be detected more quickly. These structural changes are seen in the spectra due to lower scattering at the longer wavelengths and higher scattering at the shorter wavelengths. These structural changes are clearer in the spectra of the interactance measurements, which is why the classifiers made on this data have higher accuracy. With this work we contribute to further improve the state-of-the-art of non-destructive black spot detection in potato tubers

It can be concluded that the preferred sample presentation mode is interactance for the detection of black spot in potatoes since in this mode black spot can be detected when it is close to the surface as well as when it manifested deeper in the tissue. It can also be concluded that if black spot is present closer to the surface the detection is more accurate.

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Keywords
Quality control,Potato,Black spot,Vis-NIR,Spectral imaging

P02.06.51

THE USE OF NEAR INFRARED SPECTROSCOPY FOR THE ANALYSIS OF FUMONISINS

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Fumonisins are mycotoxins produced by Fusarium fungi, which regularly contaminate maize, a staple food in Africa. Their dietary intake has resulted in nephrotoxic, hepatotoxic, immunosuppressive, and carcinogenic effects in both humans and animals. Mycotoxin surveillance is critical in limiting consumer exposure and requires rapid, accurate, and cost-effective analysis techniques. Near-infrared (NIR) spectroscopy has, therefore, gained considerable interest. However, its potential to analyse fumonisins has been associated with detecting spectral changes caused by indirect factors such as the mycotoxigenic fungi and their effects on maize, which may reduce sensitivity to the toxin. This study explores the feasibility of using NIR spectroscopy (1000 – 2500 nm) for the direct analysis of fumonisins.

To confirm the potential of NIR spectroscopy for the direct analysis of FB1, whole and ground maize inoculated with Fusarium verticillioides were analysed. Whole maize again yielded performances unsuitable for fumonisins analysis. However, the PLS model of ground maize achieved good and reliable results (R2p = 0.77). Furthermore, PLS-DA could classify the samples with 100% accuracy at the 2 mg.kg-1 maximum levels legislated in South Africa. Wavelengths similar to those previously determined as potential FB1 absorbance bands were identified, and the performance of the model reconstructed on them (R2p = 0.80) indicated that they contain valuable information for FB1 prediction. However, it was determined that the mycotoxigenic fungi also influenced these bands. Additionally, NIR spectroscopy obtained better results in analysing maize contaminated with Fusarium compared to artificially FB1-contaminated samples. These results confirmed the value of NIR spectroscopy as an indirect fumonisin analysis method.

This study demonstrates NIR spectroscopy's effectiveness as a fumonisins screening method in maize, providing valuable insights for its implementation in the maize industry.

ACKNOLEDGEMENTS: Stellenbosch University, for the financial support provided through the Post Graduate Scholarship bursary

National Research Foundation of South Africa for project funding

The Central Analytical Facility (CAF) Mass Spectrometry Unit at Stellenbosch University for performing fumonisin quantification with ultra-performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS)

Keywords
NIR spectroscopy,fumonisins,maize,mycotoxins

P02.06.52

COMPARING MEASUREMENT APPROACHES IN NEAR INFRARED SPECTROSCOPY FOR DETECTING JUICE ENRICHMENT WITH PLANT EXTRACTS

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In order to maintain good health, it is essential to take in the right amount and quality of health-promoting compounds. There is a growing demand for nutrient-rich products, and the food industry needs to adapt to it. Fortifying certain products with plant extracts is an effective way to ensure adequate nutrient intake. By using plant extracts, more useful compounds are taken in at the same time as they contain significant amounts of valuable antioxidants, vitamins, minerals, and more [1]. The use of fruit juices as a base for fortification is a good choice due to their favourable sensory and consistency properties. In addition, being nutrient rich and offering diverse flavours, juices also contribute to meeting daily fluid intake [2]. Qualification and authentication of different fortified products is a major challenge when focusing only on traits (e.g., colour, soluble solids, acidity, etc.) that are used empirically, for example in juice quality control. It is often more effective to perform profiling based on specific compound groups instead of, or in addition to, targeted analyses. Particularly in the case of fruit products, differences in sugar, organic acid and polyphenol composition are telltale. However, such techniques do not necessarily ensure fast, non-destructive and chemical-free operation. To address such issues, the authors explored the performance of NIR instruments with different measurement arrangements in detecting plant extracts added to fruit juices. In the study, fruit extracts (cranberry, grapeseed, pomegranate) were added in simple, binary or ternary combination to naturally dark-coloured juices at concentrations between 0 and 2.5% w/w. The spectra of these samples were recorded using both desktop and handheld NIR spectrometers in transmission and transflection modes, respectively. The data analysis focused on the wavelength range of 1100 to 1650 nm. Principal component analysis combined with linear discriminant analysis was utilized to assess the effects of extract addition and concentration, while partial least squares regression was used to predict the degree of extract enrichment. All models were validated using sample replicates. The results show that chemometric modelling based on data recorded with the benchtop instrument resulted in more accurate classification and extract concentration prediction. During the testing of the models, the coefficient of determination could be above 0.9, and the mean square error could be below 0.2 g/100 mL. These could be attributed, on the one hand, to the instrument's resolution, and on the other hand, to the measurement setup, highlighting that the transmission measurement approach is better suited for measuring transparent liquids like juices. The outcomes raise the need for further tuning of the spectral data obtained, also suggesting calibration transfer between the different devices enabling in situ investigations.

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ACKNOLEDGEMENTS: The study was supported by the Doctoral School of Food Science of the Hungarian University of Agriculture and Life Sciences; the Cooperative Doctoral Programme (KDP-2023), the University Research Scholarship Programme (EKÖP-MATE/2024/25/K), the 2023-1.2.4-TET-2023-00068 Program of the Ministry for Innovation and Technology (Hungary) from the source of the National Research, Development and Innovation Fund, and the CEEPUS mobility grant.

Keywords
fruit juice,fortification,quality control,near infrared spectroscopy,chemometrics

P02.06.53

NON-DESTRUCTIVE QUALITY ASSESSMENT OF ORGANIC AND PROTECTED GEOGRAPHICAL INDICATION (PGI) SWEET ORANGE CV. BIONDO DEL GARGANO USING NIR SPECTROSCOPY

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The citrus industry has grown exponentially due to increasing demand for its consumption. However, it is a non-climacteric fruit that must be harvested only at an optimal edible ripening stage. Hence, to determine its internal quality and ripening stage, producers and packinghouses typically collect fruit sets throughout ripening and use them to predict quality attributes using traditional and, in many cases, destructive and time-consuming methods. This strategy leads to an increase in fruit waste because it does not conform to the expected quality and doesn't allow repeated measurements of the same fruit over time [1], which has resulted in a growing demand to develop rapid, accurate, economical and non-destructive technologies for determining food-produce quality, such as Near Infrared spectroscopy (NIR) [2,3].

This study was carried out in the framework of the European project InnOBreed (2022-2026), which aims to promote the breeding of organic crops in the fruit sector and improve the performance of the fruit. The objective of one specific task of this project is to deliver high-throughput, efficient, and robust tools to evaluate the fruit organoleptic and nutritional quality of fruits by NIR spectroscopy. In this context, the application of NIR spectroscopy was evaluated to monitor and eventually predict the quality evolution of an ancient orange variety Biondo del Gargano, certified organic and PGI, during ripening (~6 weeks) and post-harvest (~6 weeks) under two storage conditions, room temperature at 20°C (RT) and cold temperature at 5°C (CT), by Partial Least Squares (PLS) regression. In both cases, the full dataset was split into the calibration set (70% of the total number of samples), used to build the regression model, and a validation set (the remaining 30%), used to test the model. In addition, for the post-harvest dataset, ANOVA-Simultaneous Component Analysis (ASCA) [4] was used to understand and highlight the importance of storage period and temperature on the fruit quality based on their NIR spectral signature. The model to predict pH showed promising prediction performance in post-harvest, with RMSEP of 0.10 and a relative error close to 3%. ASCA highlighted that both studied factors were significant, although time exerted the main effect on the NIR signature and fruit quality change during storage. NIR spectroscopy and ASCA have proven to be a powerful combination for understanding the impact of different sources of variability on fruit quality attributes (time and temperature). This valuable approach could be used for further studies considering the influence of other factors, such as variety, climate, or geographical position.

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ACKNOLEDGEMENTS: Horizon-CL6-2021-BIODIV-01-12, convention no. 101061028

Keywords
Biondo del Gargano,citrus,fruit quality,storage,ASCA

P02.06.54

EVALUATING THE IMPACT OF SAMPLE STATE ON BREAST MILK MACRONUTRIENT CALIBRATION MODELS: A COMPARATIVE STUDY OF FROZEN AND THAWED PRESENTATIONS

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Human milk banks play a vital role in providing donor breast milk for vulnerable infants, particularly preterm or critically ill newborns who cannot be fed their mother's milk. To ensure that donor milk meets the nutritional needs of these infants and adheres to safety standards, thorough analysis of its composition is essential.

Breast milk exhibits a high degree of variability, with fat content fluctuating significantly between foremilk and hindmilk. To ensure that the milk composition is suitable for the infant, a rapid and non-invasive analysis is required. To enhance efficiency and clinical applicability, it is crucial to develop calibration models that can be reliably transferred between frozen and liquid states. However, differences in sample temperature, viscosity, and particle size distribution (especially in frozen vs. liquid states) alter NIR spectra, making it challenging to directly transfer calibration models between different sample states.

In this work, 83 breast milk samples with reference values for fat, protein, lactose, and energy were scanned using a portable NIR instrument (MicroNIR VIAVI) in a wavelength range between 908–1676 nm, in different presentation modes: frozen and thawed (at 37°C). Individual calibration models for macronutrients were developed, withcorrelation coefficients of determination (R^2) of 0.693 and 0.899 for fat and 0,702 and 0.650 or protein when scanning breast milk frozen or thawed, respectively. Considering the obtained statistics, thawed samples were selected to develop the calibration models for macronutrients, and different standardization matrices were evaluated to predict frozen samples with models developed using thawed samples spectra.

ACKNOLEDGEMENTS: This research was funded by the Spanish Ministry of Science and Innovation (PID2023-149221OB-I00, PID2022-142323NB-I00, PID2020-117282RB-I00).

Keywords
Breast milk,sample presentation,macronutrients

P02.06.55

RAPID ASSESSMENT OF ITALIAN POLYFLORAL HONEY OVERALL QUALITY INDEX BASED ON NON-DESTRUCTIVE SPECTRAL SENSORS

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The quality of polyfloral honey is based on different physicochemical and sensorial characteristics such as sugars, pH, colour, texture, odour and flavour, appealing to varied consumer preferences. Traditional honey quality assessment methods, such as solvent extraction and laboratory analyses, are time-consuming, costly, and require chemical reagents. This study investigated the efficacy of a Near Infra-Red (NIR)-based device for rapid honey quality evaluation. A total of 215 Italian polyfloral honey samples collected from different beekeepers in 2022 were analyzed for parameters like moisture, water activity (aw), conductivity, pH, free acidity, diastase index, sugars (maltose, glucose, fructose), and rheological traits (colour, odour, taste and toughness). Per each trait, samples were assigned to quality classes according to a tertiles classification (1–3; low to high) based on standard thresholds [1]. An overall quality index (QI) was determined as the sum of the quality class scores for each individual trait. NIR spectra (850–2500 nm) were acquired in transmittance mode using a FOSS DS2500 spectroscopic instrument. The device operates within a scanning range of 850 to 2500 nm, with 0.5 nm intervals, and is equipped with a small rotating ring cup featuring a gold reflector with a depth of 0.5 mm. Using WinISI 4 V4.10.0.15326 (FOSS Analytical A/S, Hillerød, Denmark), a partial least squares discriminant analysis (PLS-DA) was applied to the collected spectral data. Finally, a confusion matrix was utilized to derive metrics, including sensitivity, specificity, accuracy, and the Matthews correlation coefficient (MCC), which collectively evaluated the PLS-DA model's effectiveness in discriminating honey quality. Based on the recorded reference analysis, the overall QI values were categorized into three tertiles: below 27 as low (LQI), between 27 and 31 as medium (MQI), and above 31 as high (HQI). Results showed sensitivity, specificity, accuracy, and MCC values of 0.71–0.71, 0.82–0.88, 0.79–0.82, and 0.50–0.61, respectively. MQI class was correctly assigned in 71% of samples, with minimal misclassification into nearest groups (16% to LQI, 13% to HQI). Similarly, LQI and HQI groups were correctly classified in 72% and 71% of cases, respectively, with most misclassifications occurring also in the nearest merit class. The findings demonstrate that NIR spectroscopy effectively discriminates among honey quality classes for Italian polyfloral honey. It provides a rapid, non-destructive method for segregating high-quality polyfloral honey in productive Italian ecological areas.

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ACKNOLEDGEMENTS: The authors are grateful to Lucia Piana and Giancarlo Naldi from the Italian National Honey Observatory for their collaboration in the collection and coordination of the sensorial assessment of honey samples. Research financial supported by iNEST from the European Union Next GenerationEU (PNRR – ECS00000043). This manuscript reflects only the authors' views and opinions; neither the European Union nor the European Commission can be considered responsible for them.

Keywords
polyfloral honey,quality assessment,NIR,PLS-DA,overall quality index

P02.06.56

QUALITATIVE AND QUANTITATIVE ASPECTS OF NIR-SPECTROSCOPY FOR THE ANALYSIS OF SUNFLOWER SEEDS AND THEIR CHEMICAL COMPONENTS

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NIR spectroscopy has been successfully used in the past to chemically analyze a range of crop products, including whole grain samples. However, especially with sunflower seeds, there have been problems with sufficient penetration depth of the NIR radiation, which can be attributed to the black color pigments in their hulls. A powerful measurement technique is diffuse reflection spectroscopy, which can be applied to powders or whole seeds, requiring little or even no sample preparation. Important aspects for routine work are multivariate calibrations with chemometric tools such as partial least squares, which require a population of samples that are representative for the forages to be analyzed. For reference analysis often laborious analytical methods are applied that can handle larger grain quantities for making sure to yield representative analytical results for the samples to be analyzed.

To explore the qualitative and quantitative aspects of NIR spectroscopy for the analysis of sunflower seeds, a literature review highlighted the chemical composition and structure of sunflower seeds. It was found that sunflower seed hulls predominantly consist of cellulose, xylose, and lignin. The black color of sunflower seeds is attributed to anthocyanins and phytomelanin. The composition of the kernel varies significantly by variety, with oil content mainly of oleic and linoleic acid esters ranging from 40 to 90 %. Other components include proteins and cellulose.

Spectroscopic measurements were performed using an IFS66 Bruker spectrometer equipped with a rotational ellipsoidal accessory and a fiber-optic probe for diffuse reflectance measurements, and a GrainSense spectrometer with an integrating sphere. The rotational ellipsoidal measurements demonstrated that the chemical components are reflected in the NIR spectra. Measurements of intact kernels showed the shell spectrum only, questioning any available information on the composition of the kernel at first glance. Samples were sorted based on oil content using the first overtone band of the C-H vibrational band at 5800 cm-1 within the kernel spectra. The fiber probe measurements revealed that Fresnel reflection occurs at the surface, further complicating whole kernel measurements. SW-NIR intra-cavity measurements using the GrainSense spectrometer showed two bands after baseline correction. By shaving off the melanin layer from the hull surface, the band at 10800 cm-1 was increased. However, sorting by oil content, as possible with the rotational ellipsoidal measurements, could not be replicated.

Principal Component Analysis (PCA) was conducted using spectra of the GrainSense spectrometer. Samples were differentiated by form (intact/shell/kernel/ disassembled) and by variety. Particularly in the PCA of the second derivative spectra, distinct clusters were observed. Additionally, a rough distinction by variety was evident across all sample forms in the third score, suggesting measurable differences between the seed varieties. In summary, NIR spectroscopic examination of sunflower seeds is challenging due to the thick hull and black pigment layer. However, the presented PCA has demonstrated that it is not impossible.

ACKNOLEDGEMENTS: I would like to express my gratitude to everyone who contributed to the successful completion of this research project.

Firstly, I extend my deepest appreciation to Alina Schulz for her exceptional work and dedication during her Bachelor thesis.

I am also immensely grateful to Professor Heise for his invaluable guidance and support throughout this project.

Keywords
Sunflower seeds,Diffuse Reflection,Principal Component Analysis,Photon penetration depths,Chemometric

P02.06.58

EXPLORING THE POTENTIAL OF HYPERSPECTRAL IMAGING TO DETECT MICROPLASTICS IN SEA SALT

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Microplastics (MP), plastic particles of sizes 0.1 up to 5,000 µm, have become an emerging environmental and health risk. MP are a matter of concern, as they have entered the food chain and therefore, are being consumed by humans, with the potential consequent effects on human health [1]. MP have been found in a wide range of food products such as honey [2], beer [3], seafood [4] and sea salt [5][6][7]. Although the ingestion of products such as honey, beer or seafood can be reduced or eluded, the consumption of food ingredients such as table salt seems unavoidable. MP have been detected in table salt from more than 100 brands worldwide [8]. However, the lack of a standardized and cost-effective analytical methodology still makes the understanding of occurrence and sources of MP limited. Optical non-destructive technologies, particularly Near Infrared and Hyperspectral imaging, have shown to have potential for the detection of MP in biological matrices such as sea salt [9]. This study seeks to assess the viability of analysing polyethylene terephthalate (PET), polystyrene (PS), polypropylene (PP) and low-density polyethylene (LDPE) microparticles in sea salt using Hyperspectral imaging. With this purpose, PET, PS, PP, and LDPE microparticles (2.4, 1, 0.8, 0.3, 0.2 and 0.1 mm) were obtained by grinding and sieving plastic pellets. Coarse salt (0.46-2.21 mm) and fine salt (135-480 µm) samples were artificially contaminated with the obtained MPs. A push-broom Specim FX17 camera (935-1720 nm) equipped with two different lenses (FOV38 and OLESMacro) was used to acquire spectral images. MIA Toolbox 9.1 (2024) running in MATLAB R2023B environment was used for analysis. The best combination of spectra preprocessing strategies were used to develop models (partial least square discriminant analysis, PLS-DA) for MP classification.

The PLS-DA classification model developed was able to classify PET, PS, PP and LDPE microparticles in fine and coarse salt samples with sensitivities and specificities of calibration and cross-validation of 1 for all four polymers. For coarse salt, particles of PET, PS, PP and LDPE were detected in the range 0.1-2.4 mm. For fine salt, particles of PET, PS, PP and LDPE were also detected in the range 0.1-2.4 mm. Smaller particles detected were in the range of the pixel size of the HSI equipment using OLESMacro (20 µm). It must be considered that the focusing of particles in matrices of bigger particle size (such as coarse salt) is an added difficulty.

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ACKNOLEDGEMENTS: This work was funded by the Spanish Ministry of Science and Innovation, and the European Regional Development Fund project number PID2021-127128OR-I00, and by the Consolidated Research Group (2021 SGR 00461 and SGR 00468) from Generalitat de Catalunya and by CERCA Programme.

Keywords
Microplastics,Polymer microparticles,Sea Salt,NIR spectral imaging,Chemometrics

P02.06.60

NIR SPECTROSCOPY FOR EVALUATING TEMPERING EFFECTS ON POLYMORPHIC BEHAVIOUR IN DARK CHOCOLATE

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Dark chocolate, a globally popular confectionery, consists of sugar and cocoa particles dispersed within a cocoa butter matrix [1]. The quality of dark chocolate is heavily influenced by its manufacturing processes, with tempering being one of the most critical steps. Tempering is the controlled melting and cooling of chocolate to promote the formation of the desired BV polymorphic form of cocoa butter. This crystalline structure is essential for imparting favourable sensory characteristics, such as a smooth mouthfeel, glossy appearance, satisfying snap, and resistance to fat bloom - a common defect that compromises texture and appearance. Differential Scanning Calorimetry (DSC) and X-ray Diffraction (XRD) have been used to determine the polymorphic forms of cocoa butter. While highly accurate, they require specialised equipment and expertise and are unsuitable for real-time monitoring during production. Near-infrared (NIR) spectroscopy offers an cost-effective alternative, suitable for industrial applications. This study investigates how different tempering treatments influence the polymorphic form of cocoa butter in dark chocolate using NIR spectroscopy. Identifying the desired tempering conditions that promote the formation of the BV polymorph, this research aims to provide chocolate manufacturers with actionable insights to improve product quality and consistency. Using NIR spectroscopy highlights the potential for real-time monitoring and quality control in chocolate production, offering a significant advancement over methods like DSC and XRD.

Dark chocolate samples were produced using four temper treatments (under-tempered, well-tempered, over-tempered and untempered). Using a Viavi handheld MicroNIR device (950-1650), spectra from molten chocolate samples were collected post-tempering. NIR spectroscopy combined with partial least squares discriminant analysis (PLS-DA) and soft independent modelling of class analogy (SIMCA), were used to discriminate dark chocolate samples produced by either of the four tempering treatments. PLS-DA and SIMCA models were built using spectra preprocessed with standard normal variate (SNV) + mean centering (MC). The most accurate PLS-DA model was built using 2 latent variables (LVs), accumulated variances of 93.97% in the X block and 35.68% in the Y block. The under-tempered SIMCA model was built using 4 PCs, which explained a total of 94.45% of the captured variance. The well-, over- and untempered chocolate SIMCA models were built using 3 PCs, each explaining 89.41, 86.43 and 98.86% of the total variance, respectively. SIMCA validation models achieved 88% and 75% overall classification accuracy for modelling chocolate samples subjected to different tempering treatments, using 'prediction most probable' and 'prediction strict' (threshold = 0.05) as classification rules, respectively. PLS-DA models achieved 84% and 68% overall classification accuracies, using the respective classification rules. Both SIMCA and PLS-DA models accurately (100%) classified untempered dark chocolate samples, whereas moderate classification accuracies were obtained for the sub-models, depending on the classification rule used. The classification results underscored the importance of developing models using both classification rules to better interpret the results of complex datasets. Overall, NIR spectroscopy could be a valuable analytical tool for monitoring the tempering process during chocolate manufacturing, contributing to the enhancement of chocolate quality.

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ACKNOLEDGEMENTS: Acknowledgements

Naomi Douglas acknowledges receiving funding from European Union's Horizon 2020 research and innovation programme under grant agreement (#101008228).

Keywords
chocolate,tempering,cocoa butter,polymorphic form,PLS-DA

P02.06.61

BEYOND FREEZING: NIR INSIGHTS INTO MEAT SUPERCHILLING AND RADIATION PENETRATION

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The cold chain plays a crucial role in preserving and transporting perishable food, ensuring quality and safety for consumers [1]. By optimising processes within the cold chain, we can not only reduce food waste but also promote sustainable resource management. In this context, superchilling has emerged as a promising strategy to extend the shelf life of fresh meat by 2-4 times compared to conventional refrigeration, while avoiding some of the disadvantages of freezing, such as quality degradation and higher energy consumption [2]. Despite its potential, the lack of fast and non-destructive tools for real-time monitoring of ice crystallisation during superchilling remains a significant challenge for both research and industrial applications [3]. Furthermore, understanding the penetration of NIR radiation into meat under different crystallisation conditions is crucial to optimise its application in food processes [4].

This study has two main objectives: 1) to monitor ice crystallization in real-time during superchilling and 2) to investigate the penetration depth of NIR radiation into pork loin samples treated under different thermal conditions. For the superchilling study, six pork loin cubes (3 × 3 × 3 cm) were exposed to airflow at -18°C and 1.3 m/s. The process was monitored using a MicroNIR spectrometer (Viavi Solutions, USA) and a temperature sensor (Elitech GSP-6, UK), with data recorded every 2 minutes. For the radiation penetration study, duplicate samples of three different sizes (3 × 3 × 3 cm, 2 × 2 × 2 cm, and 1 × 1 × 1 cm) were analyzed sequentially under three different thermal conditions: refrigerated, superchilled, and frozen.

Principal Component Analysis (PCA) conducted on the NIR spectra, collected during superchilling process, showed a distribution pattern consistent with the theoretical freezing curve, supported by loadings revealing the influence of water's physical state.

Multivariate control charts, based on PCA scores, were calibrated considering four freezing tests, and subsequently validated with two independent tests. The initial and final point of the transition phase were determined by δPC1 between two consecutives sampling.

Regarding the penetration study, the results showed different depths of NIR penetration, indicating the influence of thermal history on the interaction between NIR radiation and the flesh matrix.

These findings demonstrate the potential of NIR spectroscopy not only for optimizing the superchilling process but also for providing valuable insights into the interaction between radiation and food matrices. The results lay the foundation for more efficient, precise, and sustainable industrial protocols for food transformation and preservation.

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Keywords
ice crystallization dynamics,ray penetration depth,data analysis

P02.06.62

SIMULTANEOUS QUANTIFICATION OF TOTAL POLYPHENOL AND CAFFEINE IN TEA LEAVES OF NORTH EAST INDIA USING NIR SPECTROSCOPY

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Tea is the second most popular non-alcoholic beverage in the world. It is processed from tender shoots of the plant Camellia sinensis. Tea shoots are rich in polyphenolic substances and are responsible for most of the characteristics of processed tea. Total polyphenols and caffeine in tea leaves are the most critical constituents for quality attributes and health benefits. Polyphenols in tea are responsible for health-beneficial properties like antioxidant activity, preventing cardiovascular disease and boosting immunity, etc. [1,2]. Caffeine is another important ingredient that contributes to the brisk taste and stimulatory effect. This paper reports on the leaf quality measurement using near-infrared reflectance (NIR) spectroscopy for quick and in situ estimation of the total polyphenol and caffeine content of fresh tea leaves [3].

Fresh tea leaves were plucked from the experimental garden of Tocklai Tea Research Institute, Assam, India. Total polyphenols and caffeine content were estimated in 110 samples using the standard chemical methods of analysis (ISO 14502-1 & 23 (ISO, 2005). The NIR spectra were collected in the reflectance mode using a near infrared spectrometer (Ayudyog Pvt. Ltd., India). Each spectrum had an average spectrum of 32 scans at a resolution of 2.96 nm over the range of 900–1700 nm, thus a total of 256 reflectance values were produced. To increase the signal to noise ratio, the average of three spectra per sample was taken, and analysis were conducted by the SpectraFind software (Ayudyog Pvt. Ltd., India). The flat absorption peak at 1200 nm was attributed to the second overtone of the CH stretching vibration and the band at 1464 nm was assigned to the vibration of phenolic OH. These bands were closely related to various functional groups of caffeine and polyphenols in tea [4].

Several spectral preprocessing methods, including Savitzky-Golay (SG) smoothing, standard normal variate (SNV) and multiplicative scatter correction (MSC), were applied to eliminate noise interference, baseline drift, light scattering and so on caused by undesired factors [5]. Comparing among the pretreatment methods, SG proved to be the best pretreatment method for determination of caffeine, while MSC was more suitable for measuring total polyphenol, with higher R2, lower RMSE and higher RPD in calibration and prediction sets. Therefore, the following analysis were all carried out on these pretreated spectra with optimal pretreatments. The PLS regression resulted in the performance parameters with RMSEC, RMSEP, Rp2, Rc2, RPD of 0.52, 0.75, 0.83, 0.87, 3.91 for total polyphenol, whereas the SVM regression produced the best RMSEC, RMSEP, Rp2, Rc2, RPD of 0.63, 0.75, 0.85, 0.82, 3.08 for caffeine respectively. This analytical protocol made a foundation for the development of on-line quality control in tea.

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Keywords
Total polyphenol,Caffeine,SG,SVM,PLS

P02.06.63

OPTICAL TECHNIQUES FOR NON-DESTRUCTIVE PREDICTION OF INTERNAL CONSTITUENT OF TOMATOES

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This study aimed to assess the potential of non-destructive optical techniques including Hyperspectral images (HSI) (Liu, Pu and Sun, 2017) and Fourier Transform (FT)-NIR spectroscopy (van de Voort, 1992) in predicting internal quality constituents of tomatoes. To scale up the experiment, three distinct hydroponic growing techniques, varying in water and fertilizer use, were implemented across two cultivation cycles for two tomato varieties (cv 'Carminio' and cv 'Mose') (Fazayeli et al., 2024). Hyperspectral images (HSI) in the Vis-NIR and NIR range, along with reflectance spectra obtained through Fourier Transform (FT)-NIR spectroscopy, were acquired throughout the harvesting period, on approximately 250 tomatoes at different maturity stages. Internal composition of individual fruit in term of total soluble solid content (TSS), pH, total titratable acidity (TA), L-ascorbic acid (AA), and vitamin C (VC) was assessed. Predictive models for each quality attribute were developed using Partial Least Squares Regression Analysis (PLSR) (Geladi and Kowalski, 1986; Sijmen Jong, 1993) applied to HSI (Vis-NIR and NIR) and FT-NIR spectrometer spectra. Models constructed on FT-NIR data with selected ranges demonstrated superior accuracy and robustness in prediction (R2 of 0.96, 0.93, 0.89, 0.81, 0.83 for pH, TSS, TA, AA, VC, respectively) compared to model obtained using hyperspectral imaging. These findings demonstrate the potential FT-NIR spectroscopy, in predicting chemical components in intact tomatoes. Notably, our results for Vitamin C content exceeded reported literature values, while other components, such as TSS, showed similar or better trends, considering experimental conditions and inherent variability. These advanced technologies offer potential benefits in quality assessment and grading processes, leading to enhanced product selection and reduced waste.

Keywords
FT-NIR,Hyperspectral Imaging,Vitamin C,Soluble Solids,Acidity

P02.06.64

RAYLEIGH AND MIE SCATTERING-INDUCED MULTIPLICATIVE EFFECTS IN NIR SPECTRA OF COFFEE SAMPLES

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This study explores the scattering-based differentiation of coffee samples analyzed using NIR spectroscopy, with data acquired from two distinct spectrometers: a benchtop FT-NIR (Büchi NIRFlex N-500) and a handheld VIS-NIR device (ASD QualitySpec Trek). Three distinct sample groups were identified through t-SNE clustering, primarily differentiated by multiplicative scattering effects. Conventional preprocessing methods such as SNV, MSC, and EMSC did not significantly reduce these distinctions, indicating the persistence of underlying scattering phenomena of non-uniform nature. A notable improvement in classification of Arabica coffee cultivars was achieved by incorporating a dummy variable aligned with the observed clustering using Linear Discriminant Analysis (LDA). The joint use of t-SNE and PCA was essential; non-linear t-SNE grouping enabled mapping of these groups to PCA scores, while PCA X-loadings provided direct interpretation of the involved scattering effects, suggesting a robust underlying factor related to sample microstructure or optical properties.

Region-wise analysis of the power-law baseline slopes revealed a clear transition from absorption-dominated behavior in the 4000–7000 cm-1 range to Rayleigh-like scattering in the 10000–20408 cm-1 range, with the 7000–10000 cm-1 range serving as a transitional zone primarily dominated by Mie-like scattering due to micrometer-scale particles, though some Rayleigh contributions may persist. Differences between the two spectrometers highlighted the influence of optical design and sampling geometry on scattering sensitivity. The handheld device exhibited steeper slopes in the VIS region, suggesting greater sensitivity to surface scattering, while the benchtop device's shallower slopes in the same region implied enhanced diffuse collection.

Power-law fitting provided physical insights into the interplay of distinct scattering effects. Rayleigh-like scattering was attributed to surface porosity at the nanometer scale, while Mie-like scattering was linked to micrometer-scale powder particles. These scattering mechanisms introduced multiplicative effects that were elucidated through power-law analysis. The study underscores the importance of understanding scattering effects in NIR spectral analysis, particularly for heterogeneous samples such as coffee, and emphasizes the co-contribution of chemical and physical sample features to NIR spectral variations. Understanding these scattering effects is essential due to their common presence in agri-food samples and enhances the interpretability of NIR spectra for such complex materials.

Keywords
Rayleigh and Mie Scattering,Multiplicative Effects,Power-Law Analysis,t-SNE,Interpretability

FUTURE TRENDS

P02.08.66

TIME RESOLVED NIR SPECTROSCOPY OF THERMALLY ACTIVATED DELAYED FLUORESCENCE (TADF) EMITTERS

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For efficient operation, OLEDs – employed, for instance, in smartphone displays – ought to convert singlet and triplet excitons into light. These excitons are formed by electron-hole recombination and 75% of these recombinations result in triplet excitons. One approach to utilize the triplet excitons is thermally activated delayed fluorescence (TADF) [1]. In TADF, triplet excitons are converted into singlet ones by reverse intersystem crossing (rISC). Efficient emitters need to exhibit small singlet-triplet energy bandgap (δEST ~ 25 meV) and higher states (Sn>1 and Tn>1) that contribute to spin vibronic mixing to enable ISC and rISC [2]. These states can be localized energetically by time resolved near-infrared (NIR) spectroscopy [2, 3]. Such a localization was performed with our recently built femtosecond NIR set-up. A prototypical NIR emitter (TAA-DCN [4], see Figure 1) was excited with 400 nm pulses and the spectroscopic response was probed by NIR pulses. The respective NIR transitions (Figure 1, right) exhibited a dynamic shift on the same time scale as the stimulated emission. From the results, vertical and adiabatic energies of Sn>1 states can be extracted.

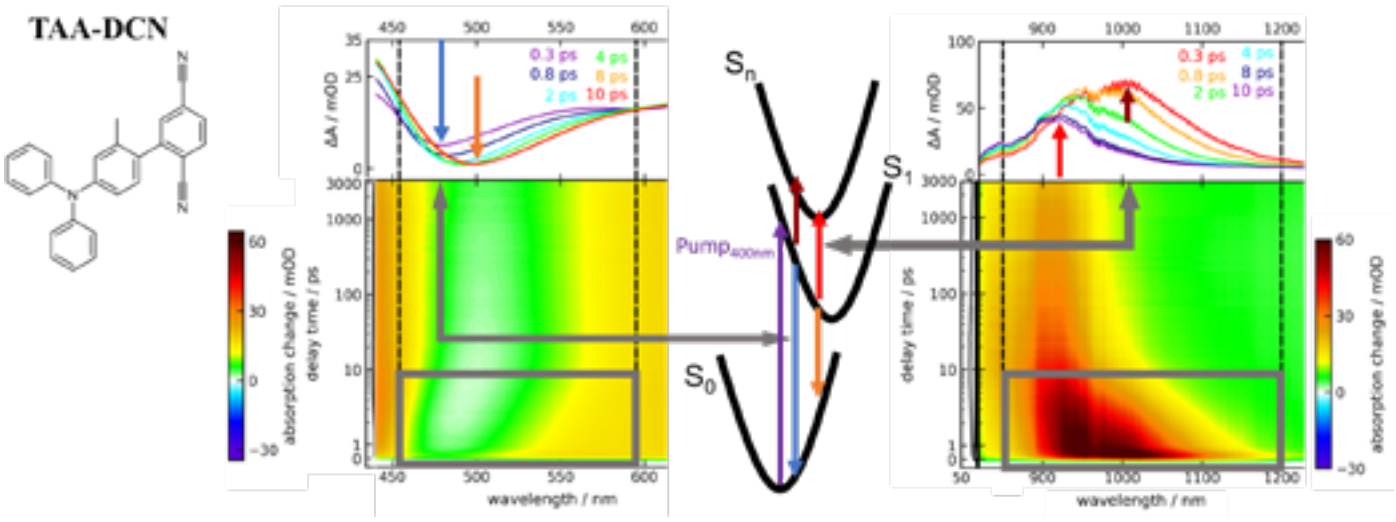


Figure 1: fs UV/Vis (contour plot in left) and NIR (contour plot in right) measurements on TAA-DCN dissolved in toluene. The observed dynamic shifts are rationalized with the diagram shown in the centre.

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ACKNOLEDGEMENTS: Financial support from the Deutsche Forschungsgemeinschaft (grant 396890929/GRK 2482, "ModISC") is gratefully acknowledged.

Keywords
time-resolved NIR spectroscopy,TADF,intersystem crossing,spin vibronic mixing

P02.08.67

COMPARATIVE EVALUATION OF AN ALTERNATIVE ONLINE AND FREE DATA ANALYSIS PROGRAM FOR ITS IMPLEMENTATION IN THE CLASSROOM

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Near infrarred spectroscopy is a well stablished analytical technique in a wide variety of high relevance fields including health and pharmaceutics, agronomy and food industry, textiles or petrochemical industry. The exponential improvements of technical aspects regarding instrumentation, decentralization and automation of the analysis result in the obtaining of large amounts of complex data [1,2]. In order to solve the commented issue, chemometric tools were developed and soon gained relevance [1]. Bibliography research showed an exponential increment of publications regarding the application of chemometrics to this analysis technique for the last 30 years. This background points the need to include advanced data analysis subjects for undergraduate students in chemistry-like degrees. Hence, new trends in chemistry education should cover a comprehensive and practical knowledge on this topic and the correct use of the analytical tools linked to it [1]. While the theory behind chemometrics should not be an issue attending to the vast amount of literature available, practical exercises may be challenging to be carried out due to the lack of updated, useful and accessible data analysis programs. In this aspect, commonly available softwares tend to be either pricey and require subscription plans, unintuitive or outdated. This work focuses on the evaluation of one new online, free-use program in current development for data analysis. For this task, the adulteration of extra virgin olive oils, a hot topic in agronomy and quality control[2], was taken as an example to compare analytical performances between one standard commercial software with the online, free-use alternative program. To replicate real life work with chemometric tools, spectra of adulterated olive oils were applied standard pretreatments such as smoothing and derivatives and were then evaluated with principal component analyses and partial least squares regressions. Other technical aspects of the program regarding user-friendly features were also evaluated, including interface clarity, intuitive display and responsive design among others. The evaluated program showed a performance equivalent to the standard software, making it suitable for its application in the classroom. Furthermore, it showed some interesting features regarding interconectivity, such as the capability of saving and transferring workspaces and downloading results with formats compatible with other softwares.

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ACKNOLEDGEMENTS: The authors would like to acknowledge the Ministry of Science and Innovation for the project Ref. PID2022-142323NB-I00 and PID2023-149221-B-I00RB-I00 funded by MCIN/AEI/10.13039/501100011033/ and by "ERDF A way of making Europe. Jesús Montero acknowledges her PhD contract from the Electrical, Electronic, Computer and Systems Engineering department in collaboration with GEAB research group, both from University of Oviedo. Candela Melendreras acknowledges her PhD grant (BP22-115) from the Asturias Regional Government (Spain).

Keywords
Chemometrics,Education,Olive Oil,Fraud detection

P02.08.68

APPLICATION OF PORTABLE NIRS IN ADVANCED AGRI-FOOD TRACK AND TRACING SYSTEMS COMBINING RFID, BLOCKCHAIN AND IOT: A SWEET POTATO USE CASE

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The rapid growth of the human population, rising consumer demand for healthy and authentic food, and insufficient coordination between agricultural and food industry production highlight the need for reliable tracking and tracing solutions for food commodities. With the increasing trend of miniaturization and portability for near infrared spectroscopy (NIRS), the relevance of applying the technique in such systems is on the rise. The present research aims to apply portable NIRS combined with chemometrics for the quality assessment of certified Hungarian sweet potato at multiple production steps, while integrating the technique with a self-developed track and tracing solution using Radio Frequency Identification (RFID), Internet of Things (IoT) and blockchain. Three different sweet potato types (n=60 each) were scanned before/after a storage and a following transport scenario using two handheld NIRS devices. Samples were sub-divided into three environmental temperature groups: 14 oC (optimal), 8 oC and 20 oC. Classification models to differentiate between types and handling circumstances were realized using principal component-based linear discriminant analysis. Models to predict starch, sugar and total polyphenols in samples were built using partial least squares and support vector regression. Models were leave-three-consecutives-out cross-validated, while a test set was also applied by using a representatively selected 20% of samples for prediction, repeated three times to further evaluate model robustness. Finalized models were containerized as a Dockerfile and deployed using a cloud computing solution. Independent samples (n=10/each type) were evaluated at the above-mentioned relevant stages of the production chain using the developed NIRS method. Measurement results, including the predicted sweet potato type and chemical parameters were automatically summarized and updated as a product certificate in a format compatible with the rest of the system, including the blockchain-based validation. Models built using both devices and algorithms could reach high predictive performance, evaluated by R2, root mean squared error values and observations on regression vectors – reaching reliable predictions even on the independent sample set. The present findings further verify that NIRS combined with chemometrics is a reliable, easy-to-integrate tool to monitor agri-food product quality "from farm to fork" as a part of modern digital systems, promoting transparency, authenticity and consequently, consumer health.

ACKNOLEDGEMENTS: This work was supported by the Doctoral School of Food Science of the Hungarian University of Agriculture and Life Sciences; the KDP-2023 Program of the Ministry for Innovation and Technology (Hungary) from the source of the National Research, Development and Innovation Fund and the Visegrad Fund supported "Food Quality in Digital Age" project.

Keywords
food quality control,machine learning,near infrared spectroscopy,short supply chain,track and trace

P02.08.69

REPOSITORY OF BIODIVERSITY SPECTRAL DATA FOR THE STUDY AND CONSERVATION OF SPECIES IN TIMES OF CLIMATE EMERGENCIES: FEASIBILITY STUDY

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Near-infrared spectroscopy (NIRS) has been used in the biological field for ecological, taxonomic and biological studies of cryptic species and for the eco-parasitological diagnosis of biological vectors. The raw spectra are collected, processed and analyzed by specialists, but are kept by them and are often not made available when articles are published. In the field of ecology and health, there is no repository of NIR spectra for external validation of data, nor is there a website with information on the use and analysis of NIR data for biodiversity studies. With climate change becoming more and more frequent, species' metabolic patterns tend to change and their behaviors as a result, which can lead to the extinction of a species. Epigenetic studies have already shown that the environment directly interferes with gene silencing, which can lead to significant metabolic changes for species. This database will enable NIR specialists to create robust chemometric models with significant quantitative representation of the target populations in their natural habitat and make them available as a chemical portrait of the organisms present at a given time in a given environment. These models will be able to outline classification, species prediction, evolution and infection or environmental contamination studies worldwide. The aim of this work is to present a feasibility study based on a systematic literature review and the creation of an educational platform and repository for NIR spectrophotometric data in the field of biodiversity and parasitology. This work was conducted in the form of a systematic literature review with meta-analysis in accordance with the PRISMA guidelines (Preferred Reporting Items for Systematic Reviews and Meta-analyses) whose objective was to gather and categorize articles that use NIR spectroscopy with a focus on biodiversity and parasitology. The following descriptors were used: "Biological Vectors" AND "Near infrared spectroscopy"; "Parasite" AND "Near infrared spectroscopy" and "Biodiversity" AND "Near infrared spectroscopy" in three databases: web of Science, scopus and pubmed for 21 years. The purpose of this review was to highlight the main researchers in the area (the platform's target audience) and the types of research carried out. In total, 277 articles were pre-selected according to the inclusion criteria during the search in the Scopus (72), Web of Science (149) and Pubmed (56) databases. After screening, 54 articles published by 327 researchers from 18 countries were identified. Repeated, out-of-scope and unavailable articles were excluded. The years 2021 and 2023 were the most productive with 10 and 09 articles, respectively. As for the nationality of the last authors and leaders of the research groups, the countries that stood out the most were the United States in first place, followed by Australia, Brazil and the United Kingdom. There were also contributions from groups in France, Spain and Austria. The platform is being built on FIOCRUZ's virtual campus and interested parties will be able to sign up soon. The copyright of the raw data published will be guaranteed. This platform will enable multicenter and networked studies on the chemical profile of global biodiversity influenced by climate emergencies, using NIR as the main analysis methodology.

ACKNOLEDGEMENTS: Financial support by CNPq code 001 and pibic program

Keywords
Biodiversity,Climate change,environmental health,epigenetic,chemical phenotype

P02.08.70

A NOVEL FEMTOSECOND NIR SPECTROGRAPH

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Commonly, the femtosecond characterization of photo-excited molecules involves UV/Vis probing [1]. Assignment of the observed signals is often challenging since transitions to highly excited states are involved (e.g. S1 to S7 or T1 to T8). Transitions like S1 to S2 or T1 to T2, which are easier to assign, are often located in the near infrared region (NIR). Lower excited states Sn>1 and Tn>1 are also of interest in emitters for OLEDs [2].

This motivated us to develop a femtosecond NIR setup to enable detailed investigations in this spectral region. It incorporates a diode array detector (Hamamatsu InGaAs linear image sensor, G11478-512WB) which is sensitive up to 2550 nm. Building on work by Bradler et al. [3] and Ishii et al. [4], continuum generation in the NIR was optimized. Several crystals (sapphire, YAG), crystal lengths (from 3 mm to 20 mm), and focusing conditions (75 mm, 250 mm, 400 mm, 500 mm) were tested. An 8 mm long sapphire crystal yielded the best results with the NIR continuum extending to ~1800 nm (see Figure 1).

Measurements on the photophysics of β-carotene in cyclohexane illustrate the performance of the setup. The sample was chosen as it exhibits several bands in NIR [5]. With our setup, the spectro-temporal characteristics of β-carotene are reproduced (see Figure 1). The spectral coverage is extended by ~200 nm to 1800 nm.

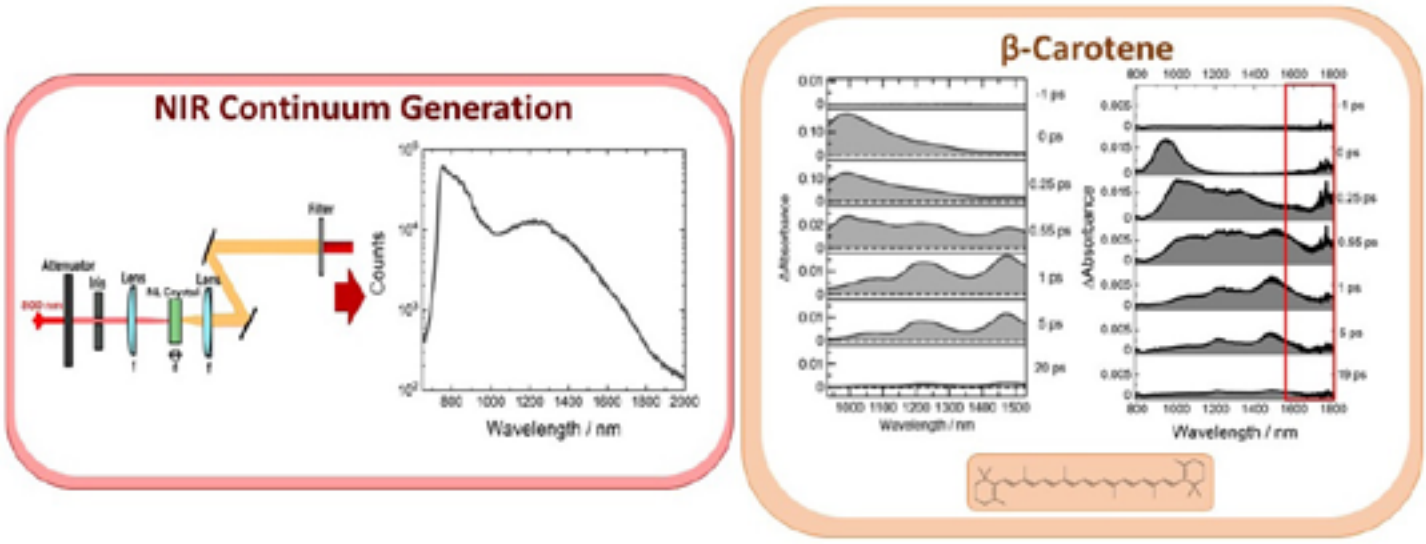


Figure 1: NIR continuum generation in the fs-NIR setup (left, red box) and an exemplary measurement (right, orange box), with data from Takaya et al. [5] (left) compared to our results (right). The red box in our results marks the increase of spectral coverage in the present setup.

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ACKNOLEDGEMENTS: Financial support from the Deutsche Forschungsgemeinschaft (grant 396890929/GRK 2482, "ModISC") is gratefully acknowledged.

Keywords
Femtosecond NIR Spectroscopy,NIR Continuum Generation

IMAGING & REMOTE SENSING

P02.09.71

APPLICATION OF NEAR-INFRARED HYPERSPECTRAL IMAGING FOR NON-DESTRUCTIVE FILM THICKNESS EVALUATION ON WOOD SURFACES

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The application of protective coatings on wood surfaces is critical for improving durability, aesthetics, and resistance to environmental factors. However, traditional methods for evaluating the thickness of these coatings often pose challenges due to their destructive nature, low efficiency, and limited precision. This study explores the potential of near-infrared (NIR) hyperspectral imaging (HSI) as a novel non-invasive technique for accurately assessing film thickness on wood.

The methodology involved the use of an NIR hyperspectral imaging system to capture spectral data across a range of wavelengths. Dry and wet wood samples with various coating thicknesses were analyzed. Preprocessing techniques, including Standard Normal Variate (SNV), were applied to enhance spectral quality and minimize variability caused by scatter effects. Multivariate regression models, such as Partial Least Squares Regression (PLSR), were developed to correlate spectral signatures with coating thickness. Additionally, Partial Least Squares Discriminant Analysis (PLS-DA) was employed to classify and differentiate coatings based on thickness and layering. For wet coatings, individual layers were spectrally resolved, even in the absence of water absorption bands, demonstrating the robustness of the approach.

Key findings demonstrate the ability of NIR hyperspectral imaging to differentiate dry film thickness with great precision and resolve multilayer wet coatings. Regression models delivered reliable quantitative predictions, while PLS-DA successfully classified samples based on their coating characteristics. These results emphasize the method's potential for real-time, non-destructive evaluations.

This study not only highlights the utility of NIR hyperspectral imaging for monitoring and quality control in wood coating processes but also suggests broader applications in industrial scenarios where non-destructive, real-time measurement is critical. The integration of this method could lead to significant advancements in manufacturing efficiency, reduced material waste, and improved product consistency.

Keywords
Hyperspectral,Wood,Paint,Coating,Thickness

P02.09.72

CALIBRATION OF HYPERSPECTRAL CAMERAS USING A MODULAR CALIBRATION BOARD

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Hyperspectral imaging (HSI) systems require accurate calibration to ensure reliable data interpretation, yet traditional processes are labor-intensive and prone to errors [1-3]. This study introduces a modular calibration board that automates and simplifies the calibration process by addressing intensity, spectral, and spatial corrections. The board features seven 3D-printed modules designed for specific tasks, including intensity correction using a high-reflectance Restan plate, wavelength calibration with LEDs and a narrow-band laser diode, and spatial resolution assessment via modulation transfer function (MTF) analysis. An artificial neural network (ANN) detects and identifies modules, enabling precise and automated calibration with reduced user intervention. Experimental results demonstrate effective elimination of intensity variations, successful wavelength calibration, and reliable MTF calculations for spatial resolution assessment. The modular design ensures flexibility and compatibility with various HSI systems, making advanced imaging techniques more accessible to general engineers and industrial users.

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ACKNOLEDGEMENTS: The authors thank the Innovation Fund Denmark, University of Southern Denmark, Newtec Engineering A/S, Aarhus University, Dansk Affaldsminimering, Vestforbrænding, and Plastix for financial support.

Keywords
Hyperspectral imaging,Calibration,Automated data processing,Object Detection,Wavelength Calibration

P02.09.74

RAPID AND NON-DESTRUCTIVE SEED VIABILITY PREDICTION USING NEAR-INFRARED HYPERSPECTRAL IMAGING COUPLED WITH A DEEP LEARNING APPROACH

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Seeds are fundamental to the agricultural food industry, and gaining a deeper understanding of seed viability before planting can improve both storage management and field performance. In this study, we addressed this challenge by employing cost-effective near-infrared hyperspectral imaging (NIR-HSI) alongside a convolutional neural network (CNN) deep learning approach. The NIR-HSI camera was chosen for its ability to detect both molecular vibration information (i.e., chemical composition differences) and the spatial distribution within each seed sample, providing more detailed insights than a standard RGB digital camera. The focus of this study was to develop a methodology for improving the interpretation of viable and non-viable seeds using principal component analysis (PCA) and clustering of the hypocotyl endosperm seed coat, by analyzing X-ray CT and NIR-HSI data. Subsequently, a CNN was trained to automatically classify seeds based on their viability by recognizing the differences between viable and non-viable seeds. Experimental results demonstrate that the proposed methodology achieves high classification accuracy for both a five-fold cross-validation set and a test set of tomato seeds. Therefore, this study introduces a novel and practical strategy for predicting seed viability.

ACKNOLEDGEMENTS: The authors gratefully thank the financial support provided by JSPS (KAKENHI, No. 24K09016).

Keywords
seed viability prediction,near-infrared hyperspectral im,convolutional neural network,X-ray CT,principal component analysis

P02.09.76

MACHINE LEARNING METHODS FOR SUGAR QUANTIFICATION IN GRAPES BASED ON NEAR-INFRARED HYPERSPECTRAL IMAGING

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Hyperspectral imaging [1] has applications in many fields, including agriculture, environment, medicine and industry. It can be used to classify objects according to their composition, or to quantify compounds present on the surface and show their spatial distribution. This last case is often challenging as the reference is not known for each pixel. Image processing is even more important in this case to extract the Region of Interest (ROI) to build the model but also to be able to apply it on new images.

This scientific study presents the methodology for analyzing hyperspectral images for grape bunch maturity prediction directly in vineyards. In this example, the parameter to be predicted is the sugar content (in °Brix) on each whole bunch of grapes, providing an average measurement of sugar content per image.

The database includes 131 hyperspectral images of 2 red grape varieties (Fer Servadou N. and Syrah N.) acquired with the SPECIM IQ (SPECIM - Konica Minolta) portable visible – near-infrared hyperspectral camera, with a spectral range of 400 to 1000 nm. Grape bunches are measured in the vineyard with the natural illumination of sunlight and without a uniform background. As a result, the image bank is quite heterogeneous. Extracting the object of interest (single bunch) is therefore a complex matter and a specific methodology and algorithm had to be created.

A methodology to automatically process the images has been developed. It consisted of different steps: 1) spectral preprocessing with row normalization, 2) extraction of the Region of Interest (ROI) by a Soft Independent Modeling of Class Analogy (SIMCA) [2] approach, combined with classical thresholding, and 3) average of the pixels.

The results of the quantitative models built on the average spectra shown that a Support Vector Machines (SVM) [3] regression model is significantly outperforming Partial Least Squares (PLS) regression [4].

The automatic tool for image processing has been successfully applied to predict the sugar content of the whole image ROI to visualize the pixel spatial distribution of the sugar in the bunch.

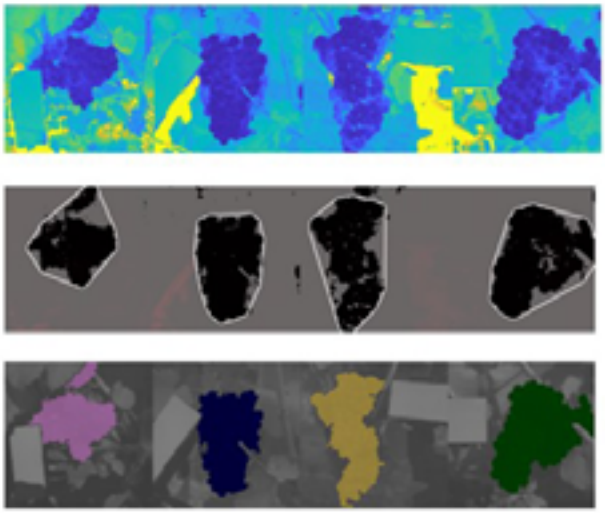
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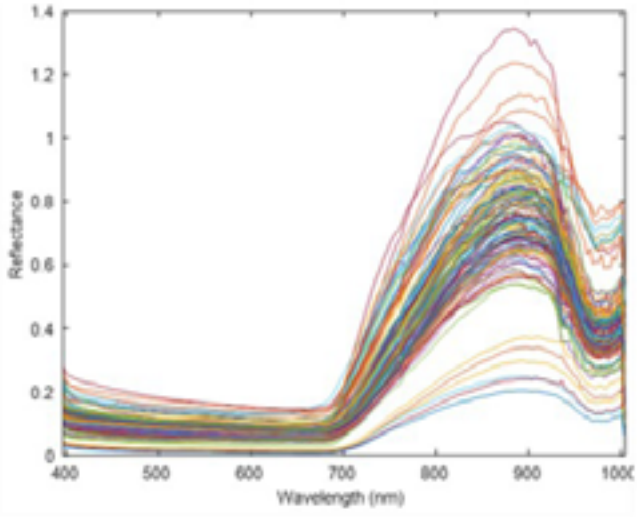
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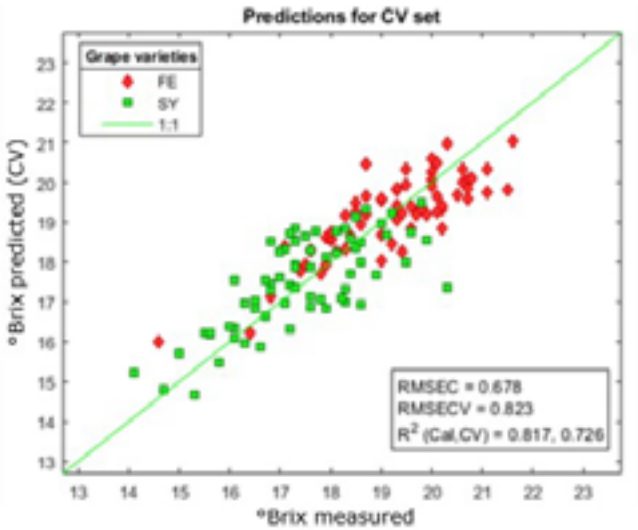
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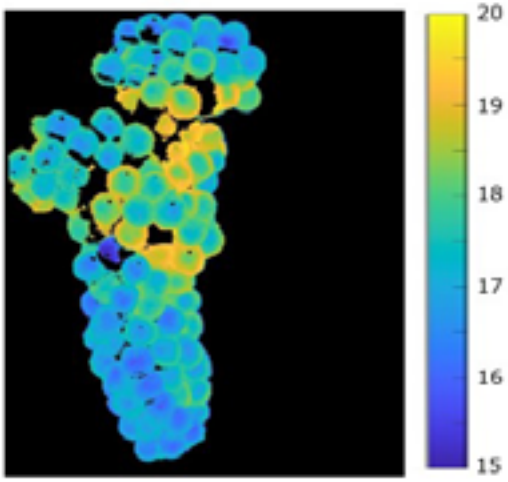
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Figure 1: Example of extraction of the ROI on 4 images (top-left), resulting raw averaged spectra for all the images of the whole database (top-right), predictions in cross-validation of the average sugar content for each image of the SVM model (bottom-left) and predictions for each pixel for one of the images (bottom-right).









ACKNOLEDGEMENTS: This HIS data analysis study was carried out by Ondalys in collaboration with INRAE (UMR ITAP, Team COMIC and UE Pech Rouge) and IFV (IFV South West) as part of the VINIoT project, an Interreg SUDOE project funded by the European Regional Council.

Keywords
Hyperspectral Imaging (HSI),Machine Learning,Quantification,Sugar,Grape bunch

P02.09.77

NEAR-INFRARED SPECTROSCOPY AS A TOOL TO CLASSIFY GUMMY CANDIES MADE FROM BOVINE, PORCINE, AVIAN AND PISCINE GELATIN

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Gummy candies are a popular confectionery product enjoyed worldwide, valued for their ability to satisfy hedonic hunger and their wide variety of flavours and textures. A key ingredient in gummy candies is gelatin, which provides the characteristic chewy texture. Gelatin is derived from various sources, such as bovine, porcine, avian, and piscine, each with distinct amino acid profiles that can influence the final product's properties. For consumers with dietary restrictions, religious beliefs, or ethical concerns, the source of gelatin is a critical factor, making accurate identification essential. Near-infrared (NIR) spectroscopy offers a promising alternative to the current methods, for distinguishing between gelatin types in finished products like gummy candies, which are often time-consuming, labour-intensive and destructive. This study aims to develop a NIR spectroscopy classification model to differentiate gummy candies produced with gelatin from four sources (bovine, porcine, avian, and piscine). The research has significant implications for quality control, regulatory compliance, and consumer trust, as it enables manufacturers to verify labelling claims and meet the growing demand for transparency in food production.

To enable effective classification model development, 16 batches of four types of gummy candies, each with a different gelatin source, were produced. From each batch, five gummy candies were randomly selected and scanned three times using the Viavi microNIR handheld spectrophotometer (908-1650 nm). Principal component analysis (PCA) was applied on the NIR data, pre-processed with Savitzky-Golay second derivative and mean centering for exploratory purposes. A PCA scores plot showed a differentiation between gummy candies produced with porcine gelatin and the other three types along PC1 (explaining 72.06%). However, no further clustering was observed.

Subsequently, the possibility of building a classification model for the identification of the gelatin source using partial least squares discriminant analysis (PLS-DA) was attempted. To this purpose, the available spectra were split into a training and test set through a suitably modified version of the Kennard-Stone algorithm, to ensure all the candies from a batch were kept in the same set. Cross-validation on the training data for the selection of the optimal model complexity and the best pre-processing was also performed through a leave-a-batch-out strategy. The best model, built on the spectra pre-treated with second derivative followed by mean centering, showed an average correct classification rate on the training data of 83.75% (85.0% bovine, 86.7% porcine, 76.7% avian, and 86.7% piscine). When applied to the test set, the model provided 72.5% overall accuracy with the highest correct classification rates observed for porcine (90.0%) and piscine (85.0%) gelatin. On the other hand, the prediction accuracies were lower for bovine (60.0%) and avian (55.0%) gelatin.

In a second stage, a model was built for the discrimination of porcine gelatin with respect to the others. This model, built on data pre-processed by first derivative and mean centering, was able to correctly classify 94.6% of training samples and, when applied to test individuals provided a 93.7% correct classification rate. Particularly, sensitivity was 100% for porcine test samples, a promising outcome in the light of religious dietary restrictions.

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ACKNOLEDGEMENTS: Ilke Olivier gratefully acknowledge receiving funding from European Union's Horizon 2020 research and innovation program under grant agreement (#101008228).

Keywords
NIR,Gummy candy,PLS-DA,Classification

P02.09.78

HYPERSPECTRAL IMAGING FOR NONDESTRUCTIVE DETECTION OF FUSARIUM WILT IN TOMATO PLANTS

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Fusarium wilt in tomato plants, caused by the soil-borne fungus Fusarium oxysporum f. sp. lycopersici, is challenging to manage once symptoms developed and often resembles the symptoms of drought stress. Since this disease causes significant economic losses for tomato farmers, it is crucial to develop rapid and non-destructive techniques for early detection. In this study, we aimed to develop a non-destructive diagnostic method for the early detection of Fusarium wilt and its differentiation from drought stress in tomato plants using hyperspectral imaging and deep learning. Spectral data for tomato plants were collected using a visible/near-infrared (Vis/NIR) and short-wave infrared (SWIR) hyperspectral imaging system under various Abiotic and Biotic stress conditions. Advanced deep learning analysis models were investigated and developed to analyze the hyperspectral data of tomato plants. The results demonstrated that Fusarium wilt and drought stress can be distinguished based on differences in several key wavelengths. This study highlighted that a hyperspectral imaging system can be effectively utilized for the early, non-destructive detection of Fusarium wilt in tomato plants.

ACKNOLEDGEMENTS: This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (No. RS-2024-00459812).

Keywords
Hyperspectral imaging,Fusarium wilt,Drought stress,Spectral analysis,Non-destructive detection

P02.09.80

FIELD ASSESSMENT OF FUSARIUM HEAD BLIGHT ON WHEAT USING NEAR INFRARED HYPERSPECTRAL IMAGING

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Recent reviews pointed out several gaps in current research on plant stress detection using hyperspectral imaging (HSI). Among these gaps is the limited number of HSI applications in real field conditions. In addition, the current research focuses on discriminating between healthy plants and those affected by the stress of interest, meaning that the models are not tested against other stresses. This study aims at contributing to filling both gaps by applying near infrared hyperspectral imaging (NIR-HSI) in the field to detect Fusarium Head Blight (FHB) and investigate the ability of this technique to differentiate FHB, which is caused by fungal pathogens on the ear, and Take-all infections, caused by a fungal root pathogen affecting the whole plant. The differentiation of both diseases is of great interest as they show similar symptoms on the ear, but their respective pathogen agents are well distinct.

A field trial was designed with six winter wheat varieties sown outdoors in a cultivation bed. The trial was inoculated at flowering with Fusarium graminearum (causing FHB) and the plants were scanned in the field at seven different dates using a NIR-HSI system mounted on a mobile gantry. During the trial's growing period, a Take-all infection occurred simultaneously with the FHB infection. This co-occurrence of two biotic stresses provided an opportunity to test the method's ability in classifying both stresses affecting the ears.

A dichotomous classification method was developed using PLS-DA models. The method was validated at pixel level and showed that pixels of healthy and diseased ears could be accurately classified (accuracy > 90%). However, the classification between FHB and Take-all did not provide satisfactory results. At the plot level, an evaluation of the models was achieved by comparing the predicted disease severity (percentage of coverage on the plot) with the field-observed disease incidence (percentage of infected ears on the plot). The evaluation of the models was carried out at the stage of grain maturation. As for the pixel level, the summation of both diseases' predictions showed a good correlation with the overall stress incidence observed in the trial (R^2 greater than or equal to 0.80) but the models did not allow diseases discrimination. In addition, the evaluation of the results indicates that FHB-infected ears with low severities are particularly challenging to detect.

These results suggest that the models can assess the general health status of the ears, however, further developments are needed to accurately discriminate individual stress types. This work also highlights areas for potential improvements in future studies, such as using a blackout enclosure for field image acquisitions. Such an enclosure combined with artificial lighting could enhance the quality of the spectral data. Additionally, it would provide more stable lighting, thereby improving the repeatability of the method. Furthermore, this setup could be used under cloudy weather, increasing the frequency of image acquisitions.

ACKNOLEDGEMENTS: This work was performed by the PhenWheat project funded by the Walloon Region, Service Public de Wallonie (SPW), Direction Générale Agriculture Ressources Naturelles Environnement (DGO3 - DGARNE), Direction Recherche et Développement, project number D65-1412/S1. This study has also contributed to the PHENET project funded by the European Union's HORIZON-INFRA-2022-TECH-01-01: R&D for the next generation of scientific instrumentation, tools and methods, under grant agreement N° 101094587. The authors are grateful to the team from the Crop Production Unit of the CRA-W for managing the field trials. They are also grateful to Benoît Scaut and Frédéric Debode as well as the technical staff of all CRA-W research teams involved.

Keywords
wheat,fusarium head blight,field,NIR hyperspectral imaging,phenotyping

P02.09.81

HYPERSPECTRAL IMAGING FOR PREDICTING FAT (OLEIC ACID) AND PROTEIN CONTENT OF SPANISH PISTACHIOS

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Pistachios are a valuable crop known for their nutritional and health-promoting properties, largely attributed to their fatty acid composition. Oleic acid (53–55%) and linoleic acid (29–32%) are the predominant fatty acids in pistachio oil, offering cardiovascular and antioxidant benefits [1]. Ensuring the quality of pistachios is essential for meeting consumer expectations and maintaining market competitiveness. Traditional methods for assessing quality parameters, such as protein content and fatty acid composition, are often destructive, time-consuming, and labour-intensive. In this context, non-destructive techniques like hyperspectral imaging (HSI) have gained attention for their ability to rapidly and accurately predict quality attributes without damaging the product [2]. This study explores the use of HSI combined with Partial Least Squares (PLS) and Support Vector Machine (SVM) models to non-destructively predict protein content and oleic acid levels in pistachios. To this end, pistachios from two distinct irrigation treatments (high and control) were analysed using a SPECIM IQ hyperspectral camera capturing spectral data in the 400–1000 nm range. The performance of the models was evaluated using the coefficient of determination (R2), Mean Absolute Error (MAE), and Root Mean Square Error (RMSE). For protein prediction, PLS achieved an R2 of 0.75, with MAE and RMSE values of 0.58 and 0.74, respectively, outperforming SVM (R2 = 0.73, MAE = 0.60, RMSE = 0.77). For oleic acid prediction, both models performed well (R2 > 0.90), but PLS demonstrated superior accuracy (R2 = 0.91, MAE = 0.60, RMSE = 0.78) compared to SVM (R2 = 0.90, MAE = 0.66, RMSE = 0.87). These findings highlight the potential of HSI-PLS as a valuable tool for quality assessment in the pistachio industry.

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ACKNOLEDGEMENTS: This work was supported by Project CDTI (IDI-20200822) and by MCIN/AEI/10.13039/501100011033 and European Union NextGenerationEU//PRTR, grant number RYC2021-033890. Co-financed by FEADER funds and Junta de Castilla y León (Spain). COST Action CA21142 titled Fruit tree Crop REsponses to Water deficit and decision support Systems applications (FruitCREWS), https://www.cost.eu/actions/CA21142/. Salvador Castillo-Gironés thanks INIA for the FPI-INIA grant number. PRE2020–094491, partially supported by European Union FSE funds. Dr. Raquel Martínez's contract has been supported by Regional Institute of Agri-Food and Forestry Research and Development of Castilla-La Mancha (IRIAF). Dr. Sergio Vélez's contract was supported by Beatriz Galindo Program, funded by the Spanish Ministry of Science and Innovation.

Keywords
Hyperspectral,Oleic,Pistachio,Protein,Quality

P02.09.82

NIR HYPERSPECTRAL IMAGING FOR CHEESE MACRONUTRIENT PREDICTION: COMPARISON BETWEEN CHEMOMETRIC AND MACHINE LEARNING APPROACHES

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Traditional methods for assessing cheese's nutritional content are often labor-intensive, destructive, and environmentally taxing. This study explores non-destructive techniques like Near Infrared Hyperspectral Imaging (NIR-HSI) and Near-Infrared (NIR) Spectroscopy combined with chemometrics and machine learning (ML) to predict fat and protein content in 73 cheese samples. By adopting a broad-based approach, we integrated a diverse range of cheese varieties, from fresh to seasoned cheeses, into a single model, aiming to enhance predictive accuracy. The cheese samples were acquired with a line-scan NIR-HIS system working in the 1000-1700 nm range. We evaluated multiple pretreatment methods, feature selection approaches, and model types to determine their predictive performance. Chemometric approaches, including Partial Least Squares (PLS) and its variants, were compared with ML models such as a Multilayer Perceptron (MLP), alongside variable selection techniques like CovSel and IPW-PLS.

For protein prediction, the best-performing chemometric model used EMSC (degree=6) pretreatment, achieving an R^2_pred of 0.96, MSEP of 2.61, and SEP of 1.61 without variable selection. The UVE-PLS model, also in the chemometric category, further enhanced accuracy with an R^2_pred of 0.98, but required 80 selected variables. For fat prediction, the IPW-PLS chemometric model with 15 selected variables achieved the highest R^2_pred (0.94) and RMSEP (2.15). ML models, such as the MLP, performed comparably, with the best MLP model yielding an R^2_pred of 0.94 for protein and 0.97 for fat, but without the benefit of interpretable variable selection. This highlights the advantage of chemometrics in providing practical insights into important wavelengths for fat and protein prediction.

Keywords
cheese,chemometrics,machine learning,quantitative prediction,hyperspectral imaging

INDUSTRY & PAT

P02.10.83

SEASONAL VALIDATION OF INDUSTRIAL NIR MEASUREMENTS OF GRASS AND SILAGE

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The nutrient contents of grass and silage are important for the dairy industry as it affects the quantity and quality of the milk produced by the cows. The forage is usually augmented by concentrate, e.g. soy or corn. Measurements of the nutrient contents of the silage will allow more precise allocations of concentrate, reducing the costs of the farm. Measurements of the grass will allow harvesting the grass when the nutrient contents are the highest, improving the yields.

In this project, NIR calibrations for dry matter, protein, neutral detergent fiber (NDF), organic matter digestibility (OMD), lactic acid, sugar, ammonia, and ash in silage, and dry matter, protein, NDF, OMD, and ash in grass have been deployed and used in industry. Due to its industrial use, the calibrations need to be valid for future seasons, as well as for samples from all of Norway. Therefore, the calibrations have been validated over several seasons. There have also been multiple types of validation. Validations have been done both with random samples, and with samples selected by spectral variation, i.e. by high and low X-scores in the various models.

This presentation focuses on the differences in performance estimates between the validations, how it relates to existing understanding of the chemical properties (e.g. direct versus indirect modelling: dry matter versus ammonia), and the impact of the modelling (e.g. preprocessing). By designing the validation around the specific issues that the calibration will be required to handle, in this case variations in sample origin and seasonal variation, we can achieve a more direct understanding of the calibration's robustness.

ACKNOLEDGEMENTS: We would like to acknowledge Tine for allowing us to share the results, and Norwegian Fund for Research Fees on Agricultural Products through the project "Precision Food Production" (no. 314111) for financial support.

Keywords
Silage,Grass,Industrial measurements,Validation

P02.10.84

MILK COAGULATION MONITORING USING A NIR PROBE (I): MCR-ALS AS A TOOL TO UNDERSTAND PHYSICAL PHENOMENA AND OPTIMIZE CURD-CUTTING DECISIONS

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Milk coagulation is a fundamental step in the production of Grana Padano DOP cheese, as it directly affects the quality and consistency of the final product. Finding the exact moment to stop the process is crucial for cheesemakers, who currently rely solely on their expertise to determine it. However, some mechanisms underlying this process remain poorly understood from a physical point of view. This study integrates near-infrared (NIR) spectroscopy with advanced chemometric techniques to investigate the coagulation process, aiming to provide a more standardized approach for its monitoring and understanding.

Experiments were conducted in two different dairies using two types of rennet: a traditional animal-derived rennet and a microbial rennet. For each type of rennet, a design of experiments (DoE) approach was employed to evaluate the effects of rennet concentration and pH level on a30 (curd firmness at 30 minutes) and k20 (curd firming rate), the two most common traditional reference parameters used to monitor the coagulation process. NIR spectral data were collected in real-time during coagulation, acquiring one spectrum every approximately 13 seconds for 30 minutes. The spectra were not preprocessed, as the scattering effect in the data is extremely important for the interpretation of the physical changes that occur during the process. The data were subsequently processed using Multivariate Curve Resolution–Alternating Least Squares (MCR-ALS) to extract components describing the various phases of the coagulation process.

The MCR-ALS algorithm successfully identified spectral profiles associated with key events during coagulation, allowing for a detailed characterization of the process dynamics. These profiles were then correlated a30 and k20. Results showed that the spectral components derived from the MCR-ALS analysis provided insights into the structural changes occurring during coagulation, offering a new perspective for interpreting the process.

The study highlighted differences in coagulation behaviour between the two types of rennet. Specifically, microbial rennet is more influenced by pH changes. This underscores the importance of tailoring process parameters to the specific rennet type to achieve optimal results. Furthermore, the findings suggest that the MCR-derived profiles could serve as robust indicators for determining the precise moment to halt coagulation and cut the curd, reducing variability and improving consistency in cheese making. Moreover, MCR-ALS, through the combined interpretation of spectral profiles and concentration profiles, provides valuable insights into the physical phenomena occurring during the coagulation process.

The ability to standardize the curd-cutting decision is crucial for maintaining the quality and authenticity of Grana Padano DOP. The combination of NIR spectroscopy and chemometric analysis demonstrated in this study offers a rapid, non-invasive, and reproducible method for monitoring coagulation. This approach not only enhances our understanding of the process but also provides a practical tool for optimizing production, ensuring that traditional cheese making practices can be adapted to modern quality control standards without compromising their heritage.

Keywords
Milk coagulation,MCR-ALS,Process monitoring,Process understanding

P02.10.85

AT WAR WITH BITTER ALMONDS: FAST DISCRIMINATION OF ALMONDS USING A NIR INSPECTION SYSTEM

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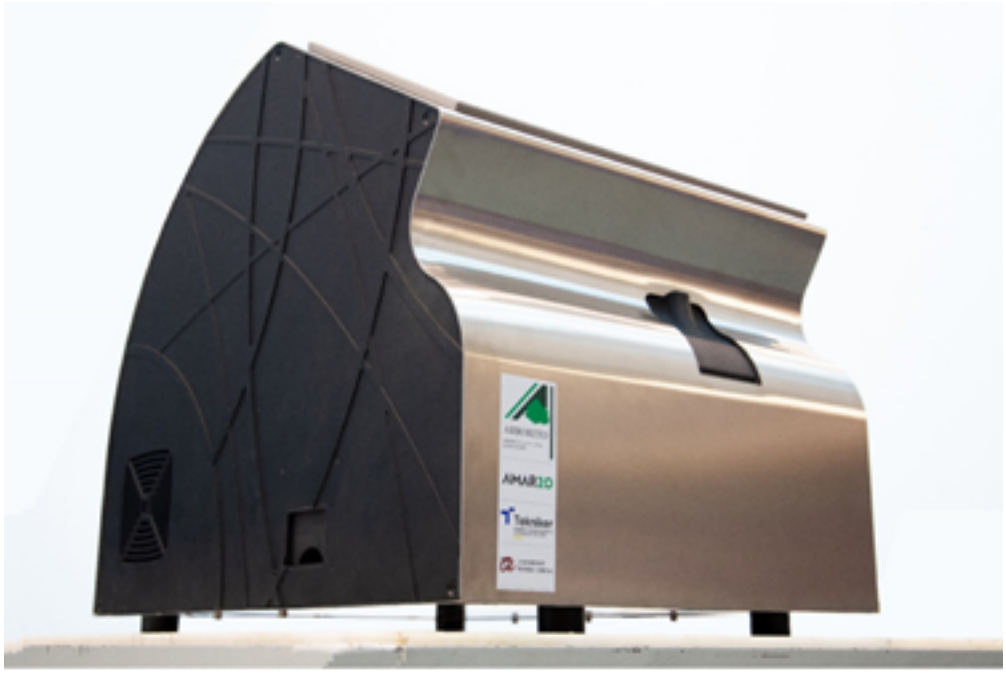
Due to the toxicity of amygdalin, a cyanogenic glucoside presents in bitter almonds but not in sweet almonds, certain markets, such as the USA, require an absence of bitter almonds in their imports. The Spanish production of almond presents a non-traceable proportion of bitter almonds (around 1%) and their presence in exported items may lead to the loss of customers and even to the veto of certain countries at the entrance of Spanish almonds. The search for technological solutions for the identification and discrimination of bitter almonds in the processing and production lines has revealed there are no commercial solutions focused on solving this problem.

This communication describes the process of developing a NIR inspection system for the detection of bitter almons. Based on promising results obtained with a limited number of almonds [1] a system capable of unitary detection and discrimination of bitter almonds during the reception stage of the product at the processing plant of the company ARBORETO S.A.T (Tarragona, Spain) was developed. The main goal was to guarantee a proportion of bitter almonds of less than 0.5% in the final batch. During the project it was necessary to deal with factors such as temperature monitoring and control of the NIR sensor, limited time for the unitary characterization of the batch (500 g of almond /min), the non-homogeneity of the amygdalin concentration on the surface of the almond, and the biochemical variability present in the Comuna almond variety selected for the project, being the latter the most problematic point to solve. The Comuna variety is composed of a group of subvarieties of almonds of different types depending on their geographical origin and this gives rise to a spectroscopic variability which is very difficult to model.

The core of the inspection system is a compact NIR sensor based on Fabry-Perot Interferometry and manufactured by Micro-Electro-Mechanical Systems (MEMS) manufacturing techniques. The NIR sensor was integrated into an opto-mechanical system together with a vision system and a sample removal system, all managed by a Labview-based control platform. The multivariate classification models were developed using Partial Least Squares Discriminant Analysis (PLS-DA) obtaining quite high sensitivity (TPR > 0.96) and specificity (TNR > 0.98) levels depending on geographical origin of the almonds but not being possible to obtain a good performance by a model for the whole Comuna variety. Considering the results, it was decided to optimize the models according to the reception centers from which the almonds came.

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Figure 1. NIR inspection system developed for bitter almonds detection.





ACKNOLEDGEMENTS: This work has been funded by Centro para el Desarrollo Tecnológico Industrial (CDTI) under project IDI-20150402

Keywords
Almonds,Amygdalin,Inspection system

P02.10.86

A FEASIBILITY STUDY FOR ACRYLAMIDE QUANTIFICATION IN BISCUITS USING VNIR SPECTROSCOPY

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Acrylamide, a carcinogenic compound formed during high-temperature processing of foods containing asparagine and reducing sugars, poses significant public health concerns. Since its detection in food in 2002, extensive research has aimed to mitigate its presence, resulting in regulatory measures such as the European Commission's Regulation (EU) 2017/2158 and the FoodDrinkEurope (Acrylamide Toolbox). These frameworks emphasize the monitoring and reduction of acrylamide levels in food products with high-frequency intake, including cereal-based items, potato products, and coffee. Despite these efforts, recent EFSA assessments indicate inconsistent reductions in acrylamide levels, underscoring the need for innovative and reliable tools to improve compliance and quality control in food production.

This study investigates the feasibility of using visible and near-infrared (VNIR) or near-infrared (NIR) spectroscopy as rapid, non-destructive tools for acrylamide quantification in biscuits, with the goal of developing a robust Process Analytical Technology (PAT) solution. A total of 98 samples of ground biscuits were analyzed across two experimental campaigns conducted in 2023 and 2024. The samples were collected directly from the production line immediately after the cooking phase in an industrial continuous oven. To maximize variability in acrylamide content, a Design of Experiment (DoE) approach considering seven production factors was implemented. Three spectrometers with distinct technical features and industrial applicability were tested: ZEISS Corona Process (from 350 to 1650 nm), Unity Spectra Star (from 680 to 2600 nm), and Bruker MPA (from 800 to 2500 nm). Reference acrylamide quantifications were carried out using liquid chromatography coupled with mass spectrometry (LCMS/MS).

Spectral data were processed using multivariate statistical techniques, including PCA for data exploration, EPO (External Parameter Orthogonalization) for domain adaptation, and PLS regression for predictive modeling. The model's performance was evaluated through internal (cross-validation) and external (test set) validation.

Overall, all the three instruments showed comparable results demonstrating promising predictive performances for an initial massive screening of the production trend (RMSE about 20% both in cross-validation and test set). Therefore, these findings highlight the potential of VNIR and NIR spectroscopy as a tool for real-time quality control of acrylamide in food matrices. Future studies will aim to expand sample variability to further enhance model robustness and support the industrial implementation of this technology for such purposes.

ACKNOLEDGEMENTS: The authors thank Barilla G.R. F.lli S.p.A. for their support and resources essential to this study.

Keywords
Acrylamide,Process Analytical Technology,Food quality,VNIR Spectroscopy,Bakery products

P02.10.87

RESEARCH ON QUANTITATIVE ANALYSIS ALGORITHM FOR NEAR-INFRARED SPECTROSCOPY BASED ON KAN CONVOLUTIONAL NETWORK

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NIR technology has gradually evolved into an indispensable core tool in agricultural production. The current algorithm faces accuracy issues caused by spectral overlap and signal noise, as well as overfitting problems due to insufficient generalization ability.

To address the challenges of limited accuracy, insufficient generalization capability, and overfitting in corn composition analysis, this study proposes a KAN convolutional neural network model based on the Kolmogorov-Arnold network. By incorporating B-spline basis functions and adaptive grids, the model efficiently captures the nonlinear features within the data. Furthermore, a sliding wavelength selection module is introduced to perform local feature extraction and identify the spectral bands that contribute most significantly to the prediction targets. Finally, the model's performance is evaluated using RMSEC, RMSEP, R^2, and RPD as metrics.This paper optimizes the regularization mechanisms in the KAN network by fine-tuning the regularization parameters for the convolutional and fully connected layers.For band selection, the sliding window size is configured as 5, with a selection threshold of 0.20. These optimizations effectively mitigate overfitting, while further enhancing the model's predictive performance and robustness.The results indicate that under the same preprocessing conditions (including Savitzky-Golay smoothing, Standard Normal Variate Transformation, and First Derivative), constructing the main convolutional model with the KAN network, and employing the KAN Linear Layer as the flattened fully connected layer, significantly improves model performance. Compared to traditional convolutional networks, the KAN network maintains high predictive accuracy even when the number of network layers is reduced to two. Its built-in hyperparameters, such as grid size, spline function order, and noise scaling, require no additional adjustment to exhibit strong generalization capability.

Experimental results demonstrate that, compared with the Conv model, the KANConv model reduces the number of neurons in the flattened layer by 65.96%. The model converges rapidly within the first 20 epochs of a total of 150 training epochs. On the training set, the coefficient of determination ([R_c]^2) of the new model reaches 0.9365, which is slightly lower than the Conv model's [R_c]^2of 0.9748. However, on the validation set, the KANConv model shows clear advantages, with predictive [R_p]^2increasing from 0.9069 to 0.9141, reflecting a 0.79% improvement. At the same time, the predictive error RMSEP decreased from 0.0527 to 0.0480, a reduction of 8.91%, indicating stronger generalization ability. Furthermore, the RPD value increased from 3.2774 to 3.4128, a rise of 4.14%, further demonstrating the model's robustness and reliability in practical applications. These results underscore its advantages and application potential in near-infrared spectroscopy analysis, particularly in resource-constrained and complex scenarios.

In summary, the KANConv model, based on the Kolmogorov-Arnold network and proposed in this paper, integrates sliding wavelength selection techniques to effectively mitigate the overfitting problem commonly seen in traditional algorithms on small to medium-sized datasets. It simultaneously achieves higher prediction accuracy and stronger generalization capability. Under limited data conditions, this method demonstrates excellent performance by significantly reducing the number of model parameters. This work offers a novel perspective and serves as a valuable reference for the further development of near-infrared spectroscopy analysis technology.

ACKNOLEDGEMENTS: This research was supported by theDevelopment and Application of MEMS-Based Near-Infrared Sensors (No. 2024GH17).

Keywords
Near infrared spectroscopy,Kolmogorov-Arnold network,KANConv,B-Spline Functions,Overfitting Mitigation

P02.10.88

STUCK ON EXPERIMENTAL VARIABILITIES: EVALUATING THE RELIABILITY OF CLASSIFICATION MODELS IN HYPERSPECTRAL IMAGING

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Near Infrared Hyperspectral Imaging (NIR-HSI) has already proven to be a helpful tool in many applications in agriculture, environmental monitoring, food quality control, mining, medical imaging, waste management, remote sensing, etc. For instance, in waste management, NIR-HSI enables the automation of plastic classification by capturing the unique spectral signatures of materials, offering significant benefits in speed and scalability for recycling processes [1].

However, the reliability of NIR-HSI models presents notable challenges in real-world applications. These models are frequently calibrated and validated under idealized conditions within controlled environments [2 3 4], while the conditions in an industry change unexpectedly, promoting biases and malfunction of the models. For example, a common pitfall arises when models are tested using data collected during the same scanning session as the calibration set. Although such data may originate from different spatial points, their similarity to the calibration environment often leads to an overestimation of model performance, neglecting the complexities and variability encountered in practical, operational settings.

This work studies the influence of multiple factors on NIR-HSI on reflectance mode and underscores the necessity of reliable validation methods that consider environmental variations, including changes in background color, lighting conditions, and acquisition session. To explore these challenges an illustrative dataset of five commercial plastic pellets (polybutylene terephthalate, acrylonitrile butadiene styrene, polypropylene, polystyrene, and polyamide 6) was selected. Spectral data were collected under different conditions with a SWIR (Specim®, Spectral Imaging Ltd., Oulu, Finland) camera ranging from 1000 to 2500 nm. Raw data and preprocessed data with scattering removal and/or Savitsky Golay derivative were tested to investigate spectral characteristics. Then, multiclass PLS-DA models were developed. External validation was conducted considering dataset acquired under calibration-like conditions and in scenarios designed to simulate real-world applications.

Our results reveal significant limitations in traditional validation approaches, which often fail to account for environmental variability. Models that performed well under controlled conditions struggled with real-world factors that negatively impacted model accuracy. Additionally, the variation in spectral signatures among plastics, depending on their physico-chemical characteristics, affects classification performance. Plastics with chemical composition extremely different from the others are easily differentiated, whereas those with common chemical compositions have overlapping spectral features that complicate accurate classification. These findings highlight the importance of incorporating environmental factors into validation protocols to ensure model robustness and adaptability in industrial applications.

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ACKNOLEDGEMENTS: This research was supported by the Innovation Fund Denmark under the project "HyperSort - Hyper-spectral imaging for advanced textile sorting and food control." Additionally, funding was provided by the Elkartek Programme of the Basque Government (Spain) under the SMART-EYE project (KK-2023/00021).

Keywords
Hyperspectral imaging,Validation protocols,Experimental variability,Plastic sorting,Multiclass

P02.10.89

NIR SPECTROSCOPY A FUNDAMENTAL TOOL FOR THE CONTROL OF DISCONTINUOUS FIXED BED REACTOR USED FOR PRODUCTION OF SUSTAINABLE COLLAGEN GLUE OBTAINED FROM RABBIT SKIN LEAVINGS

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Sustainability has become a huge concern in the glue industry, particularly in the sub-sector of glues for furniture. In the recent years, the market has demanded adhesives with lower environmental impact and higher sustainability. However, consumers are not willing to pay a premium for these eco-friendly options.

One approach to solve this apparent contradiction is to explore sustainable sources of raw materials that currently present no market value but could be used to produce industrial-grade performance glues.

Rabbit skin is one such raw materials. Historically known as "rabbit glues", its adhesive properties had made it a staple in the furniture sector for centuries. Today, rabbit glue is still used by artists and in restoration works. However, while it is valued for its quality, it does not meet industrial performance standards to be used in other applications as in furniture.

Cortadoria Nacional do Pêlo, one of the global producers for rabbit fur, generates over 3000 kg/day of fresh rabbit skin leavings which has no commercial use. Until the 80's, the company also produced rabbit glues. This presents a potential solution for the availability issue. However, despite having a steady supply of rabbit skin, the challenge of producing high performance industrial glue remains due to the quality variability.

Modern rabbit glues are mainly produced by a multi-step process that includes: extraction of collagen from fresh skin, denaturation, gelatinization reversion, and chemical modification.

The first phase of this process is highly complex and involves several steps, such as washing, degreasing, chemical and enzymatic hydrolysis. The unpredictability of this phase, is largely due to the variability in the properties of the fresh rabbit skin leavings, and it plays a critical role on final product's quality. The complexity was one of key factors behind the decline of the industrial collagen glues market in the 20th century.

To address the quality issue, one approach is to improve control over the process, particularly during the first phase steps. This can be achieved replacing the traditional heated batch reactor with a discontinuous fixed-bed reactor, that allows dynamic heating and controlled residence time.

In our research, a dynamic PCA and PLS model for the indirect prediction of a collagen glue performance was established. This model predicts collagen concentration, apparent molecular size distribution and isoelectric point using NIR spectroscopy, as a faster alternative to traditional methods, such as solid content determination, electrophoresis isoelectric focusing (IEF) and automated bond evaluation system (ABES) (bonding performance).

This paper presents the impact of using NIR spectroscopy, online residence time monitorization and control multi-step discontinuous fixed bed lab scale reactor to improve the performance of collagen glues.

Funding: This work was financially supported by: LA/P/0045/2020 (ALiCE) and UIDB/00511/2020-UIDP/00511/2020 (LEPABE) funded by national funds through FCT/MCTES (PIDDAC); Project FWFI (SI I&DT-Projects in co-promotion. Project LagusColl (SI I&DT-Projects in co-promotion, COMPETE2030-FEDER-01189700/17382) in the scope of Portugal 2030, co-funded by FEDER (Fundo Europeu de Desenvolvimento Regional) under the framework of POCI (Programa Operacional Competitividade e Internacionalização).

Keywords
NIR spectroscopy,collagen glues,on-line monitoring

INSTRUMENTATION

P02.11.91

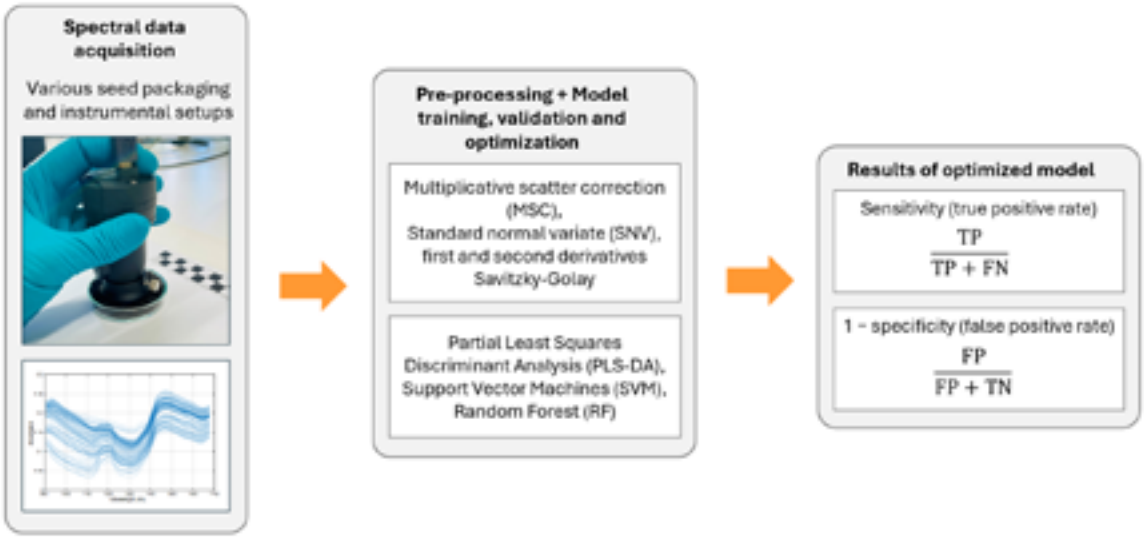
APPLICATION OF NEAR-INFRARED SPECTROSCOPY FOR SEED CLASSIFICATION IN HYDROPONIC INDOOR FARMING UNDER DIFFERENT PACKAGING CONDITIONS

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The demand for high-quality seeds suitable for hydroponic indoor farming is increasing as controlled environment agriculture (CEA) becomes a key solution for sustainable food production. Ensuring seed authenticity and quality is essential for optimizing germination, growth performance, and yield, especially in systems that rely on precise crop planning and automation. Traditional seed classification methods, such as visual inspection and chemical composition analysis, are often labor-intensive, time-consuming, and sometimes destructive, making them impractical for large-scale or real-time applications. This study explores the potential of handheld near-infrared (NIR) spectroscopy as a fast, non-destructive method for classifying hydroponic seeds, even when packaged in commonly used commercial materials such as uncoated and coated paper. The main objective was to evaluate whether NIR spectroscopy could accurately differentiate seed types through packaging, providing a rapid and efficient alternative for quality control in seed production and distribution.
For this purpose, we employed the MicroNIR 1700 ES (Viavi Solutions), a compact, handheld spectrometer operating in the 900–1700 nm range. A diverse set of seeds commonly used in hydroponic systems, including basil (Ocimum basilicum), mint (Mentha spp.), thyme (Thymus vulgaris), oregano (Origanum vulgare), chard (Beta vulgaris), and chicory (Cichorium intybus), were analyzed under three conditions: bare seeds, seeds in uncoated paper packaging, and seeds in coated paper packaging. Since packaging materials can introduce unwanted spectral interference, multiple measurement adapters were tested to optimize data acquisition.
To ensure robust classification, the spectral data underwent several pre-processing techniques, including multiplicative scatter correction (MSC), standard normal variate (SNV), first and second derivatives, and Savitzky-Golay filtering, to enhance spectral features and minimize noise. Machine learning models, including Partial Least Squares Discriminant Analysis (PLS-DA), Support Vector Machines (SVM), and Random Forest (RF), were trained and evaluated to determine the best approach for classification accuracy. The preliminary results are promising, showing that seed classification remains feasible even when packaging is present, although classification performance varied depending on packaging type and pre-processing approach.
This study provides valuable insights into the practical application of NIR spectroscopy for automated seed authentication in hydroponic farming, highlighting its potential for improving traceability, reducing fraud, and optimizing seed sorting in commercial production chains. The findings also emphasize the importance of choosing appropriate spectral acquisition strategies and data processing methods to mitigate packaging interference and enhance classification accuracy.

The detailed results, including model performance comparisons and optimal spectral processing strategies, will be presented at the conference.



ACKNOLEDGEMENTS: This work was supported by the VEMM Group, Sweden (Mälarenergi, Eskilstuna Energi och Miljö, VafabMiljö and Mälardalen University) under project AI2Sense - Pioneering frontier technologies for urban circularity and sustainability with AI and advanced sensors

Keywords
Seed classification,Machine learning,Packaging interference,Non-destructive analysis,Hydroponic indoor farming

P02.11.92

TOWARDS ACCREDITATION OF OLIVE OIL NEAR INFRARED SPECTROSCOPY ANALYSIS: GUIDELINES

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Extra Virgin Olive Oil (EVOO) is a high quality high price component of the Mediterranean diet and may be subject to food fraud. Official analyses are generally carried out using standardized chemical and physical methods, which are time-consuming and expensive which limits their use and then effectiveness. Under the fraud repression activities, the use of near-infrared spectroscopy (NIR) may be a cost-effective and quick method to screen large number of samples and identify suspect frauds.

The primary goal of this study is focused on the development of NIR analysis protocols and the construction and validation of NIR in order to test NIR performances with the aim to achieve NIR technique accreditation for olive oil analysis.

One of the first tests toward the accreditation of the method is to define instrument repeatability.

In this study a Fourier Transform spectrophotometer (FT-NIR), the MPAII: FT-NIR Multi Purpose Analyzer, supplied by Bruker was used. Spectra were collected with samples in 8mm glass vials that were heated at 40°C. No sample pretreatment was required.

NIR measurements were repeated on the same oil sample according to the following approach:

- Method I - 10 measurements repeated in succession leaving one vial inside the measuring chamber

- Method II - 10 repeated measurements by removing and reinserting the same vial into the measuring chamber

- Method III – 10 measurements on 10 vials filled with the same oil sample

The analysis results, provided by OPUS using the calibrations supplied by the manufacturer, were then processed to verify repeatability. The statistical analysis confirmed that repeatability for most of the evaluated parameters falls within the pre-established limits (CV%<5). The purpose of this stage is to build a calibration set that allows the development of calibration models used for the analysis of unknown samples.

The planning of this phase of the accreditation procedure is still ongoing, but potential guidelines have been outlined. The construction of the calibration set requires the definition of the quality parameters to evaluate and the number of samples to collect, involving ICQRF laboratories.

The choice of quality parameters is to be found in the legislation that defines olive oil classes according to the parameters assessed by ICQRF laboratories. Concerning the number of samples, it is recommended to collect approximately 100 samples as a desirable number for constructing a calibration set that can ensure reliable calibration curves for the quantification of unknowns. The aim is to collect a similar number of samples for each oil class (extra virgin olive oil, virgin olive oil, lampante oil), covering approximately the entire variability range for each parameter according to the threshold values set by law (Commission Delegated Regulation (EU) 2022/2104).

Once samples have been collected and analysed, analysis results are subjected to a validation procedure including the planning of ring tests. Ring tests are essential to ensure the quality control of the method and of the results of the tests conducted by the laboratory. They are carried out by organising inter-laboratory tests and comparing the results obtained by the participating laboratories. They aim to assess the reliability of the method by checking parameters such as accuracy, precision, reproducibility and uncertainty of the method. This is done using statistical methods which necessarily include the calculation of mean and standard deviation of results and indices such as coefficient of variation (cv%) and index of deviation (z-score). Therefore, they represent a useful tool to ensure the proper execution of analyses, the reliability of operators and the long-term quality of laboratories.

Olive oil quality parameters

Free Fatty Acids (%)	K232	K264	K268
K272	K270	ΔK	Peroxide Value (meqO2/Kg)
Polyphenols (mg)	PPP (%)	β-Sitosterol (%)	Brassicasterol (%)
Campesterol (%)	Waxes (mg/kg)	Waxes C40 (mg/kg)	Cholesterol (%)
Δ7-stigmastenol (%)	Diglycerides (%)	Erythrodiol + Uvaol (%)	Ethyl esters (mg/kg)
Total Sterols (mg/kg)	Stigmastadienes (%)	Lauric Acid (%)	Palmitic Acid (%)
Oleic Acid (%)	Linoleic Acid (mg)	Linolenic Acid (%)	Stearic Acid (%)
Myristic Acid (%)	Margaric Acid (%)	Arachidic Acid (%)	Behenic Acid (%)
Lignoceric Acid (%)	Gondoic Acid (mg)	Palmitoleic Acid (%)	

Tab 1.1 – List of olive oil quality parameters investigated using/with FT-NIR MPAII

Quality parameters	*CV%*		
	I	II	III
Free Fatty Acids (%)	0.268109	0.429281	0.605526
K232	1.382183	1.515055	1.202799
K264	1.221404	2.263213	2.60287
K268	3.889516	4.929908	5.119841
K272	0.847003	1.432289	0.845783
Peroxide Value (meqO2/Kg)	3.740541	3.494616	3.02416
β-Sitosterol (%)	0.023871	0.037585	0.034723
Waxes (mg/kg)	1.131769	2.415161	2.129566
Cholesterol (%)	2.375167	2.585105	2.896834
Ethyl esters (mg/kg)	3.83725	3.794557	3.455636
Total Sterols (mg/kg)	1.726429	3.169028	2.32672
Palmitic Acid (%)	1.182756	1.401592	1.797683
Oleic Acid (%)	0.041391	0.087276	0.091855
Myristic Acid (%)	0.258929	0.320097	2.726255
Margaric Acid (%)	1.429367	4.76152	3.647488
Arachidic Acid (%)	0.90966	2.065125	1.7222
Behenic Acid (%)	2.429345	2.077947	2.412421
Lignoceric Acid (%)	2.608659	2.694595	3.073484
Gondoic Acid (mg)	0.591591	0.764731	0.888936
Linoleic Acid (mg)	0.395599	0.698396	0.537068
Linolenic Acid (%)	0.457001	0.65465	0.576302
Stearic Acid (%)	0.520483	0.458486	0.658895
Waxes C40 (mg/kg)	14.02909	8.256591	6.905115
Δ7-stigmastenol (%)	2.835114	5.058153	6.33054
Diglycerides (%)	2.728443	8.306128	7.862824
Erythrodiol + Uvaol (%)	4.563753	8.530593	11.32055
Palmitoleic Acid (%)	1.299728	59.46646	4.33395

Tab 1.2 – Results of the statistical analysis associated with the NIR values for each parameter.

Keywords
Olive oil,Food fraud,NIR accreditation

P02.11.93

IDENTIFICATION OF CAFFEINE IN BEVERAGES AND FOOD SUPPLEMENTS USING NEAR INFRARED SPECTROSCOPY

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Background

Caffeine, known scientifically as 1,3,7-trimethylxanthine, is a natural alkaloid belonging to the xanthine group, commonly found in various plants such as coffee and cocoa beans, tea leaves, guarana berries, and kola nuts. In the last decades, the research has shown that caffeine can protect against several diseases, including Parkinson's, Alzheimer's, and dementia, due to its effects on the central nervous system [1,2]. Its psychoactive properties make it widely consumed and added to beverages and dietary supplements. However, excessive intake can lead to issues like insomnia, palpitations, anxiety, nervousness, gastrointestinal problems, headaches, and dizziness [3]. Thus, regulatory limits have been established for its inclusion in drinks and dietary supplements (32 mg/L and 200 mg per daily dose, respectively) [4]. The objective of this research was to validate a MicroNIR method for detecting and quantifying caffeine, aiming to enhance food safety control measures.

Materials and Methods

A MicroNIR method was set up for caffeine detection, which was validated by comparing its results with those obtained from a standard RP-HPLC-DAD method, commonly used for caffeine identification and quantification. RP-HPLC-DAD analyses were performed using a C18 column and isocratic elution with a binary solvent mixture (methanol and MilliQ water). Caffeine was quantified by the external standard methods and a calibration curve was created by dissolving caffeine standard in distilled water at eight different concentrations ranging from 1 to 480 µg/mL. RP-HPLC-DAD method was used to confirm microNIR accuracy and reproducibility.

Results and Discussion

49 energy and soft drinks have been analyzed by simply registering microNIR spectra and by RP-HPLC-DAD method. Preliminary results indicated that 22 energy drinks available at local discounts exceeded the permitted levels of caffeine, representing a significant risk for regular consumers. The study is ongoing to confirm these findings and validate them through chromatographic methods and also to extend the number of samples analyzed.

Conclusions

MicroNIR method validation will provide a straightforward application which could facilitate a quicker monitoring of caffeine in soft/energy drinks also thanks to the fact that it needs only a portable instrument. The investigations will be also extended to dietary supplements, thereby protecting consumer health.

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ACKNOLEDGEMENTS: Project funded under the National Recovery and Resilience Plan (NRRP), Mission 4 Component 2 Investment 1.3 - Call for tender No. 341 of 15 March 2022 of Italian Ministry of University and Research funded by the European Union - NextGenerationEU; Project code PE00000003, Concession Decree No. 1550 of 11 October 2022 adopted by the Italian Ministry of University and Research, Project title "ON Foods - Research and innovation network on food and nutrition Sustainability, Safety and Security - Working ON Foods

Keywords
MicroNIR,Caffeine,Energy drinks,Soft drinks,food supplements

P02.11.94

A NOVEL LOW-COST HANDHELD REFLECTANCE SPECTROMETER USING AN INTEGRATED MEMS/INGAAS SENSOR MODULE

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We present a novel handheld reflectance spectrometer designed for portability, precision, and affordability. This compact instrument combines discrete wavelength sensors with a MEMS tunable near-infrared (NIR) spectrometer, fabricated using standard semiconductor technology. Utilising standard manufacturing and packaging techniques, the system achieves scalability, volume production, and consumer-level affordability, making advanced spectral analysis accessible to a broad range of users.

The instrument's operational range is limited only by the window material, as it employs no refractive optics or gratings. For the proof-of-concept (POC) design, discrete wavelengths at 530 nm, 570 nm, 660 nm, and 850 nm were combined with a MEMS tunable spectrometer module covering 825–1700 nm (FWHM of 20 nm). This flexibility allows for precise configuration to meet specific application needs, including agricultural analysis, environmental monitoring, material characterization, and medical diagnostics.

Internal illumination sources ensure consistent sample illumination, while an integrated calibration system maintains high measurement accuracy. The MEMS spectrometer, comprised of a MEMS tunable filter integrated with an InGaAs photodiode, leverages standard semiconductor processes to enable robust, low-cost, and scalable manufacturing. Performance data, including spectral accuracy and resolution of the MEMS spectrometer, will be presented, showcasing the system's capability to deliver reliable reflectance spectra across diverse applications.

This handheld reflectance spectrometer exemplifies how an integrated MEMS/InGaAs technology can deliver a cost-effective and scalable solution without compromising performance.

Keywords
Reflectance Spectrometer,NIR Spectroscopy,MEMS Spectrometer,Portable Spectrometer

P02.11.95

ADVANCED CHEMOMETRIC DISCRIMINATION OF INTACT ORGANIC AND CONVENTIONAL BROWN RICE KERNELS: COMPARING NIR BENCHTOP, HAND-HELD NIR AND NIR HYPERSPECTRAL IMAGING

Elem Tamirys dosSantos Caramês [1], Michel Rocha Baqueta [1], Juan Antonio Fernández Pierna [2], Juliana Azevedo Lima Pallone [1], Vincent Baeten [2]

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The increasing world population demands higher yields in food production. Driven by this necessity, modern agriculture has adopted practices based on the use of chemical fertilizers, pesticides, high-yield seeds, and other inputs. However, in recent decades, these practices have been recognized for their negative impact on the environment and their association with deleterious effects on human health. In this context, organic farming systems, which avoid the use of these artificial inputs and pursue a more sustainable way to produce food, have gained market share. Alongside the growing interest in healthier lifestyles, the consumption of organic brown rice is increasing, making it a valuable product. At the same time, there is rising concern about analytical techniques capable of ensuring the authenticity of organic rice. Near-infrared spectroscopy (NIRS) has emerged as a fast and environmentally friendly possible alternative of analytical monitoring tool to ensure the quality of brown rice by distinguishing between organic and non-organic rice kernels. Thus, the present study aimed to test the performance of three different NIRS devices—benchtop, handheld, and hyperspectral imaging (NIR-HSI)—to develop classification models capable of distinguishing organic from non-organic brown rice kernels. The NIRS spectral data of 40 organic and 40 non-organic brown rice samples were obtained in bulks of 10 g each (approximately 200 grains). The NIRS devices operated within the following ranges and resolutions: 800 nm–2498 nm at 2 nm for the benchtop device, 908 nm–1676 nm at 6.2 nm for the handheld device, and 1100 nm–2400 nm at 6.3 nm for the NIR-HSI device. Partial Least Squares Discriminant Analysis (PLS-DA) was the algorithm applied to develop classification models. The Kennard-Stone algorithm was used to split the samples into calibration (66%) and test (33%) sets for the benchtop and handheld devices. For the NIR-HSI device, a hierarchical system was applied: in a first step, the spectral information of the rice kernels was extracted from the background and shadows present in the image by a PLS-DA model; in a second step, the mean spectra of only the rice kernels were obtained and used in the modeling process. To validate the imaging system, 12 rice kernels (not previously included in the calibration set) from 9 different brown rice samples (4 organic and 5 conventional) were combined, and a new image was captured to serve as the validation set. The performance of the final PLS-DA models was evaluated using sensitivity, specificity, and accuracy metrics. Additionally, the best-performing NIR-HSI PLS-DA model was tested in two prediction modes directly from the image: (i) pixel-by-pixel and (ii) majority-pixel per rice kernel, where each pixel of a single kernel was classified as organic or non-organic, and the class with the highest number of pixels determined the classification of the entire kernel. The three PLS-DA models developed based on the spectra data obtained by the NIRS devices tested demonstrated satisfactory performance, achieving sensitivity, specificity and accuracy rates ranging from 87.5% to 100%, for hand-held and NIR-HSI devices and from 93.3% to 100% for benchtop device. Among the tested devices, the benchtop NIRS showed outstanding results. These results indicate that NIRS technology in its different ways can be reliably applied to ensure the quality of organic brown rice.

ACKNOLEDGEMENTS: We thank the Coordination of Improvement of Higher-Level Personnel (CAPES), the National Council for Scientific and Technological Development (CNPq) processes, the São Paulo Research Foundation (FAPESP) for financial support and all technical staff members at CRA-W involved in this study.

Keywords
Organic Food,Monitoring,Rice,NIR,Authenticity

P02.11.96

SETTING UP OF A COST-EFFECTIVE HYPERSPECTRAL PROTOTYPE FOR AGRI-FOOD PRODUCT MONITORING

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Hyperspectral imaging (HSI) for agri-food applications, although promising, faces challenges such as high cost and equipment requirements. However, advances in technology and 3D printing are enabling promising low-cost so-lutions that still need to be validated in the field. This work presents the de-velopment of a cost-effective hyperspectral prototype, built using 3D ele-ments and commercially available electronic components, and operating in the spectral range from 400 nm to 1000 nm. Two types of gratings have been compared, and different light source tested. The work presents the results of a few lab-scale tests on food matrices under controlled light conditions, which were conducted to evaluate the performance of the prototype. The da-ta were explored with PCA (Principal Component Analysis), which con-firmed the ability of the prototype to distinguish samples of different colours (first trial), assess the decay of different apple samples (second trial) and dif-ferentiate between healthy and damaged tissues (third trial). The experi-mental results were consistent, and both types of grating demonstrated ade-quate performance levels to envisage a future scale-up of the device.

Keywords
hyperspectral imaging,miniaturized,low-cost,monitoring

P02.11.98

AUTHENTICATION OF VEGETABLE OILS BY FT-NIR AND MULTIVARIATE ANALYSIS

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The geopolitical tensions, such as the war in Ukraine and climate change, can lead to emerging risks related to the quality and price fluctuations of ingredients. Economically motivated food adulteration, whether intentional or unintentional, poses a significant challenge for the food industry. For instance, oils and fats are essential ingredients in food production, and they may be adulterated with cheaper oils, resulting in a decrease in the nutritional value of the final product and potentially causing food safety issues. Various chromatographic techniques (such as gas or liquid chromatography) coupled with mass spectrometry, nuclear magnetic resonance (NMR), and high-resolution thin-layer chromatography have been employed to detect the authenticity of oils and fats; however, these methods are often time-consuming and expensive.

In this study, Fourier Transform-Near Infrared (FT-NIR) spectroscopy was evaluated as a rapid technique for monitoring oil authenticity in a factory setting. Fourth authentic samples of sunflower oil, rapeseed oil, and palm oil were adulterated in-house with vegetable oils at several concentration levels and divided into calibration and validation sets. Different chemometric and statistical treatments were applied, achieving a limit of quantification (LOQ) of 2-5% oil adulteration level, depending on the oil type. Additionally, several portable NIR devices were assessed as user-friendly and cost-effective alternatives to benchtop NIR instruments. The study demonstrated the potential of NIR instruments not only for detecting oil adulteration in the factory but also for oil quality control. Future work and some limitations of the study are also discussed.

ACKNOLEDGEMENTS: The authors would like to acknowledge the contributions of the project team members: John Corthesy, Charlotte Macron, Lorane Texari, and Fernando Vitorino da Silva, as well as the kind support from Bruker.

Keywords
vegetable oil,authenticity,FT-NIR,portable NIR,chemomatic

P02.11.98b

APPLICATION OF PORTABLE NIR SPECTROMETER IN QUALITY CONTROL DURING OLIVE PROCESSING

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The evaluation of moisture and oil content for whole olives, olive paste and olive pomace is crucial for olive oil processors. Reference methods, such as Soxhlet extraction, used for oil content determination in olive fruits are time- and solvent- consuming and labor intensive. The bench-top NIR instrument, which offers a more comprehensive analysis, remains financially out of reach for most operators. Thus, there's a growing demand for faster, portable, accessible and low-cost solutions throughout the processing. In the present work, we collected hundreds of samples of whole olives, olive paste, and olive pomace from various origins or processors spanning the Mediterranean region, and the reference values were determined using the oven-drying moisture method and the Soxhlet oil extraction method. Subsequently, the spectra of the samples were scanned using the IAS-Olive portable NIR analyzer. Finally, the calibration models were established based on the PLS method. Results indicate a satisfactory correlation between the reference method and NIR methods, with all R2 greater than 0.90. The SECV of moisture in whole olives, olive paste, and olive pomace is 1.95, 0.68, and 1.31, respectively. The SECV of oil content in whole olives, olive paste, and olive pomace is 1.51, 0.62, and 0.25, respectively. A cheap, portable instrument has been shown to be a viable alternative to large bench-top instruments.

Keywords
portable,whole olives,olive paste,olive pomace,quality control

PETROCHEMISTRY, POLYMERS & TEXTILE

P02.12.99

IN-LINE MONITORING OF CONVERTING PROCESSES BY HYPERSPECTRAL IMAGING

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In industry, the trend is more and more going towards supplementation of the conventional control of relevant process parameters by direct monitoring of the quality and the characteristics of the actual production output. Recently, this also includes typical converting processes such as coating, impregnation, lamination, chemical finishing, or even printing. Aside from the determination and control of quantitative parameters such as application weight, thickness, drying state, etc., monitoring of the homogeneity is of outstanding importance. Hyperspectral imaging (HIS) is the perfect tool for this task, because it can scan large areas (e.g. wide webs) very fast and in noncontact mode. Moreover, with an adequate calibration model, it can provide quantitative data about a broad range of various physical or chemical parameters in case that they are reflected in the specific NIR spectra [1]. Furthermore, it has been shown that HIS is particularly powerful in the detection of inhomogeneities resulting from nonuniformities of the converting process. In general, HIS mainly provides information about the surface of the scanned material, but in some cases even hidden inhomogeneities in the depth can be detected. This presentation will present selected examples from various technical processes such as coating, textile finishing and lamination and drying.

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Keywords
hyperspectral imaging,process control,coatings,textiles,homogeneity

P02.12.100

DETECTING GASOLINE IN DIESEL: A PORTABLE NIRS APPROACH

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The contamination of diesel oil with gasoline has been a recurring problem in Brazil. This contamination occurs mainly due to the shared use of storage tanks at retail stations. Contamination with gasoline causes changes in the physicochemical properties of diesel, especially in the flash point temperature, a safety parameter that defines storage and transport conditions [1]. The National Agency of Petroleum, Natural Gas and Biofuels (ANP) inspects fuel distribution bases and service stations to identify such irregularities. In this case, samples are collected and assessed for quality using analysis methodologies that are expensive, time-consuming, destructive and generate waste [2]. This encourages the search for alternative analytical techniques that are fast, accurate and in loco. Near-infrared spectroscopy (NIRS), using portable technology combined with pattern recognition and calibration techniques, offers an attractive approach for rapid, non-destructive analysis. Its portability allows field applications and generates no residue [3]. Therefore, this work proposes a method that uses NIRS portable instruments and classification and calibration techniques to identify and quantify diesel contamination with gasoline. 70 contamination-free diesel B S10 samples were used in the training set, while 70 samples contaminated with different concentrations of gasoline C (1.0% to 8.0% v/v) were used as the test set, in addition to 10 samples seized in inspection actions. All samples used were provided and evaluated for quality specifications by the Fuel Laboratory of UFPE (LAC-UFPE). Two portable NIR instruments, operating in different bands, NeoSpectra - 1350 to 2550 nm and MicroNir - 900 to 1676 nm, and benchtop FT-NIR (Frontier Perkin Elmer), were used to acquire the spectra in transflectance mode. One-class models DD-SIMCA (Data Driven SIMCA) and OCPLS (One-class Partial Least Squares) were evaluated using different pre-processing techniques (SNV, derivatives and mean centering), in addition to PLS (Partial Least Squares Regression) regression models for quantifying the contamination. The DD-SIMCA models were more efficient than the OCPLS models in identifying the contaminated samples, where the benchtof FT-NIR instrument showed the best performance (100% sensitivity, specificity and accuracy rates), which was expected given its characteristics. The NeoSpectra instrument, with information in the combination region, achieved sensitivity, specificity and accuracy rates of 100%, 86.7% and 90.1%, respectively. For the MicroNIR, operating in the overtone region, the DDSIMCA model achieved sensitivity, specificity, and accuracy rates of 100%, 98.3%, and 98.8%, respectively, proving it to be statistically more sensitive and accurate than the NeoSpectra. The quantification of contamination levels by the PLS models was satisfactory, with emphasis on the MicroNIR instrument, which obtained RMSECV (Root Mean Squared Error of Cross Validation) and RMSEP (Root Mean Squared Error of Prediction) of 0.45% and 0.41%, respectively, and coefficients of determination (R2) greater than 0.98. The analysis of 10 real samples using the models developed with spectra obtained from the MicroNIR instrument achieved 100% correct classification, and in the quantification, the RMSEP 0.42% and R2 0.98. Considering the ease of in loco inspection, the accuracy of the obtained models, and the response time, the presented proposal is relevant and can be useful as a screening analysis in monitoring the quality of diesel oil.

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ACKNOLEDGEMENTS: INCTAA/CNPq, FACEPE, LAC-UFPE, PRH 30.1-ANP

Keywords
Diesel oil,Gasoline,Potable NIRS,Control quality

PHARMA & BIOTECH

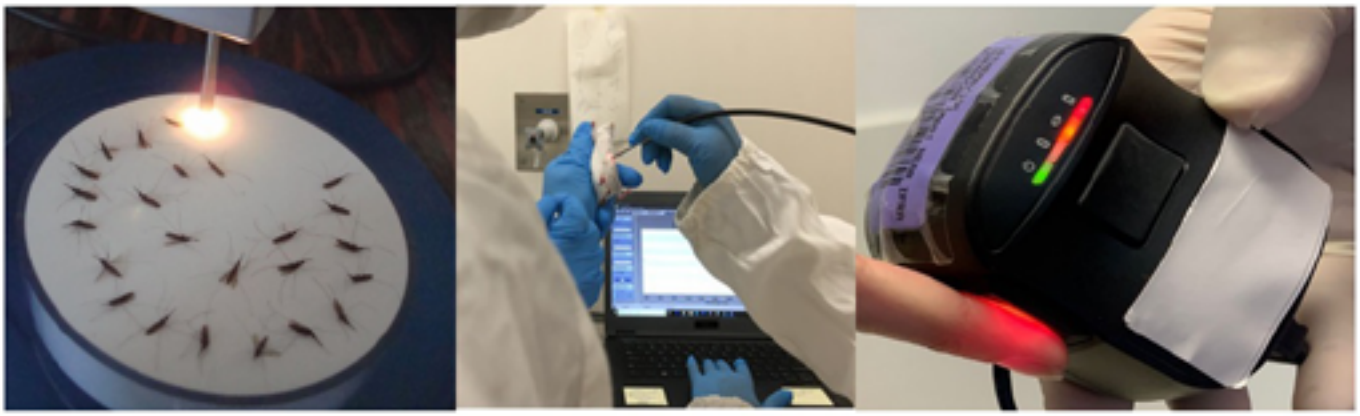
P02.13.101

SURVEILLANCE OF HUMAN PATHOGENS USING NIR

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Over the last decade, we have partnered with researchers around the world to demonstrate the evidence of the application of NIR coupled with machine learning algorithms for rapid characterisation of mosquitoes into age, species identity and infection status. More recently, we have demonstrated the capacity of the tool to detect infections including malaria, Dengue virus, Ross River virus, Barmah forest virus and soil transmitted helminths in experimental mice, human samples such as blood or stool samples and non-invasively through the skin of human subjects in the case of malaria. We will present a summary of these findings, the progress made so far and our future applications of the tool in the field.



ACKNOLEDGEMENTS: We acknowledge the contribution made to this project by technicians and research assistants from the University of Queensland, Instituto Oswaldo Cruz, and James Cook University

Keywords
infectious pathogens,Non-invasive,NIR,machine learning algorithms

P02.13.102

USING FNIRS FOR EVALUATING EMPATHY AND SOCIAL DECISION-MAKING IN NON-HUMAN PRIMATES

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Non-human primates serve as important models for investigating social behaviors, including empathy and decision-making. This study aims to explore the role of social context in shaping these behaviors in long-tailed macaques using functional near-infrared spectroscopy (fNIRS). By employing fNIRS, we seek to examine the neural correlates associated with empathy and social decision-making processes in this species.

To facilitate our investigation, we developed a cognitive testing system utilizing a transparent touchscreen display that allows pairs of macaques to interact face-to-face while performing tasks under various social conditions, such as audience effects, envy, competition, and altruism. The fNIRS technology will enable us to monitor brain activity related to these interactions, providing insights into the mechanisms underlying social decision-making.

We anticipate that this approach will reveal how social facilitation influences performance and empathic responses, even in the absence of explicit results. Through this research, we aim to contribute to a deeper understanding of the neural and cognitive foundations of social behavior in non-human primates and inform future studies on the evolution of empathy and decision-making in primates and humans.

ACKNOLEDGEMENTS: This work was supported by the Center for Excellence in Brain Science and Intelligence Technology (Institute of Neuroscience)

Keywords
functional near-infrared,Social interaction,Empathy,Cooperation,Primate

P02.13.103

EVALUATION OF COMMERCIAL PSYLLIUM HUSK QUALITY USING NEAR-INFRARED SPECTROSCOPY BASED ON A COMPACT SPECTROPHOTOMETER

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Psyllium husks are mostly commercialized worldwide in bulk as dietary fiber supplements. The quality of the product is assessed by a time-consuming (32 h) destructive testing procedure aiming to determine its swell volume (SV), which correlates with its soluble and insoluble fiber contents. Psyllium rusk products may be contaminated with their seeds. Also, they look like several other grain flours and flakes, favoring adulteration. This work aimed to evaluate the psyllium husk quality and to identify adulterated products employing near-infrared spectroscopy (NIRS) performed by a compact, low-cost spectrophotometer.

The NIR reflectance spectra of 98 samples of commercial psyllium products were acquired using a NeoSpectra compact spectrophotometer operating in the range 1350 to 2550 nm, with a spectral resolution of 10 nm at 1500 nm, and a signal-to-noise ratio of 5,000. The raw spectra are shown in Figure 1a. The samples have their SV determined following a conventional procedure. An additional set of 20 adulterated samples was produced by the addition of wheat bran (at two granulometry), amaranth flakes, and flaxseed flour in the range of 5 – 50% (m/m) to a good-quality psyllium rusks product.

Data transformation (standard normal variate (SNV), and 2nd derivative by the Savitsky-Golay algorithm, 9 points window, 2nd degree polynomial), and multivariate analysis (PCA, and PLS) and SIMCA classification were conducted with the software package Unscrambler 10.5 (Aspen Technologies). The transformed spectra set, after the remotion of outliers, is shown in Figure 1b.

The best spectral range to evaluate the psyllium quality and to estimate the SV is 2012 – 2377 nm. Figure 1b shows the transformed spectra set and the relevant spectral features in this range. The spectral range covers information about the content of polysaccharides characteristic of psyllium rusks, protein, and starch found in common adulterants and psyllium seeds. All commercial product spectra show changes in the spectral features across this spectral region. Good-quality products in the sample set were selected based on their spectra in the range and their SV values, which must be higher than 30 mL g-1. A PCA model was constructed using good-quality samples, and SIMCA was employed to classify the remaining samples and the purposely adulterated samples.

Visual observation of the spectra of the 98 commercial samples of psyllium in that spectral range, the results of principal component analysis (PCA), SIMCA classification, and the results of the partial least squared (PLS) regression allows concluding that about 80% of the commercial samples employed in this study are adulterated or contain seed core material, presenting composition that is significantly different from that expected for good-quality commercial psyllium rusks. This fact is corroborated by their low swell volume (8 – 27 mL g-1). Samples purposely added with wheat bran, amaranth flakes, and flaxseed flour allowed for verification of the potential of NIR spectroscopy to detect adulterated psyllium rusks and corroborate the conclusions about the low quality of most commercial products. The proposed NIRS method, based on a partial least square (PLS) regression model, can determine the SV of commercial products with a root mean square error of external validation (RMSEP) of 3.5 mL g-1 in the range 8 – 50 mL g-1.

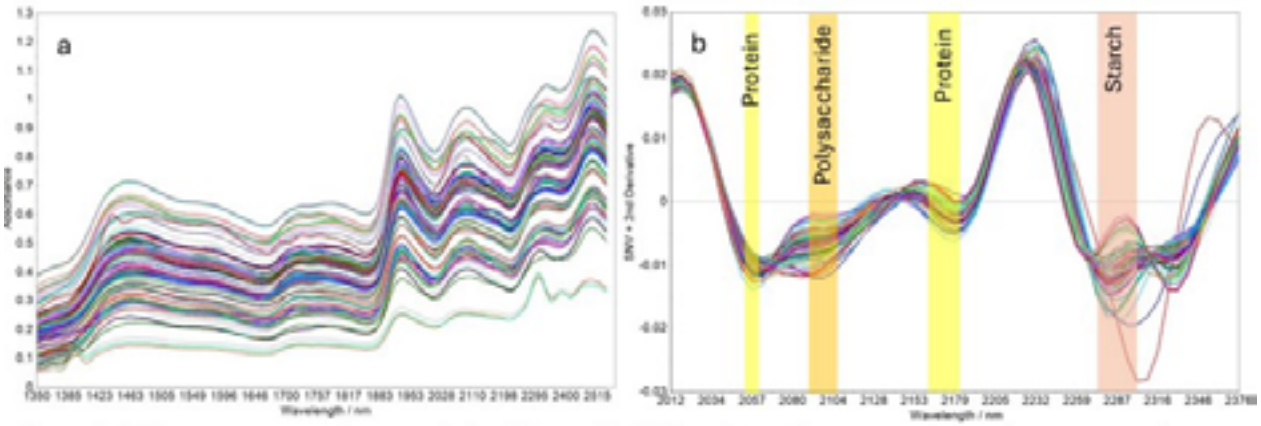


Figure 1. (a) Raw spectra set of commercial psyllium rusks; (b) Transformed spectra set in the relevant spectral range.

ACKNOLEDGEMENTS: CNPq/INCTAA: grant number 465768/2014-8; FAPESP/INCTAA: grant number 2014/50951-4; CNPq: grants number 305649/2021-3, 304199/2019-2; CAPES/PROCAD-SPCF: grants number 88887.608496/2021-00, 88887.808376/2023-00, 88887.838359/2023-00

Keywords
Psyllium Rusks Quality,Near-infrared Spectroscopy,Swell Volume,SIMCA Classification

P02.13.104

SPECTRAL INSIGHTS: TRANSFORMING CANNABIS SATIVA L. QUALITY CONTROL THROUGH NEAR-INFRARED TECHNOLOGY

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Cannabis sativa L. is a complex and versatile plant that has historically been surrounded by controversy due to its recreational use. In recent years, however, growing attention has been drawn to its proven therapeutic benefits, such as pain management and the treatment of epilepsy. The rapid growth of the cannabis industry has resulted in the need for reliable, efficient, and cost-effective analytical tools to ensure product quality, safety, and regulatory compliance. In this context, Near-infrared spectroscopy (NIRS) has emerged as a transformative technology, offering unparalleled potential for multi-compound analysis in a single, non-destructive measurement. Furthermore, its portable nature enables real-time and on-site analysis, thus allowing the monitoring of the plant throughout its growth without the need of harvesting.

The application of NIRS goes far beyond the main constituents in Cannabis sativa L., i.e., cannabinoids and terpenes. This technique can also be applied for the determination of moisture content and micro/macronutrients, among others. This ability of NIRS to provide simultaneous quantification of multiple analytes within seconds is achieved through advanced chemometric models, reducing the need for labour-intensive and time-consuming conventional techniques, such as liquid and gas chromatography. Furthermore, it can also provide qualitative information, including cultivation and variety identification. Additionally, NIRS avoids sample preparation, making it an environmentally friendly solution for routine analysis.

In this poster, the predictive models already developed in our laboratory will be highlighted. Equations for the determination of individual and total cannabinoids, moisture content (both in dried and fresh vegetal material) and micro/macronutrients will be detailed, together with the techniques employed to obtain the reference data, like liquid chromatography for cannabinoids, Karl-Fischer for moisture determination and Kjeldahl for nitrogen content, among others. Along with all this information, the predictive results for each model will also be included. Furthermore, future perspectives will be included, such as the improvement of some of the models and the development of new ones.

The application of NIRS in our laboratory has streamlined our daily operations and decreased the analysis costs, thus demonstrating the potential of NIRS for the quality control of Cannabis.

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Keywords
cannabis,near-infrared spectroscopy,cannabinoids,terpenes

SUSTAINABILITY & RECYCLING

P02.14.105

THE USE OF MICRONIR TO PREDICT COMPOSITION DURING CO2 CONVERSION INTO FUEL ADDITIVES

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With the increase in anthropogenic CO2 emissions, the natural balance of the carbon cycle has been altered, contributing to global warming and climate change. In this context, carbon capture, storage and utilization strategies have gained prominence, which includes the design of integrated carbon capture and conversion processes. In this circular carbon economy concept, CO2 is considered a valuable C1 building block for CO2-derived chemicals such as intermediates, monomers, construction materials and fuels, changing the perception of this waste product [1]. Among the products of interest that can be obtained from the conversion of CO2, carbonates stand out, such as diethyl carbonate (DEC), which is considered a promising building block for preparing polycarbonates and other more complex carbonates. DEC possesses a high fraction of oxygen, making it an interesting fuel additive with lower toxicity and more environmentally friendly than its competitors methyl-tert-butyl ether (MTBE) and ethyl-tert-butyl ether (ETBE) [2].

Ethanol oxidative carbonylation appears as an interesting green route for DEC production because it involves the direct reaction of CO2 and ethanol, two abundant reactants, yielding water as the only byproduct. However, this route involves a reversible reaction limited by equilibrium and by the hydrolysis of DEC caused by the co-produced water [3]. Consequently, intense activity research has been conducted in the field, aiming to develop new catalysts and dehydration methods to overcome these limitations [4, 5]. None of them has focused on the development of reliable characterization techniques. This work proposes the use of a portable and low cost MicroNIR spectrophotometer (1700 ES, Viavi Solutions), operating from 908 to 1676 nm, combined with multivariate analyses to quickly estimate the composition of mixtures containing DEC, ethanol and water. For the development of this work, we performed chemometric analyses, including spectra preprocessing procedures, wavelength interval selection and data splitting studies to extract accurate chemical information from the spectra of synthetic mixtures with or without suspended catalyst (cerium dioxide). The best preprocessing procedure was the first derivative determined with SG using a 7-point window and second order polynomial followed by mean centering of variables. Moreover, the backward interval partial least squares regression algorithm (biPLS) allowed the proper selection of spectral intervals, reducing the number of inputs necessary to the model development, while maintaining low values for both RMSEP and latent variables. On the other hand, the presence of DEC introduces complexity to the NIR spectra of the mixtures. Despite that, the joint use of biPLS and preprocessed spectra allowed us to obtain an adequate PLS model for DEC composition, with RMSEP of 0.424%. Extending the approach for the other compounds, we obtained RMSEP of 0.348% for ethanol, RMSEP of 3.410% for water, and determination coefficient beyond 0.99 for all of them, even in the presence of a catalyst suspended in the mixture. This is very comforting if we are to explore this technique for real-time monitoring of actual catalytic synthesis of DEC.

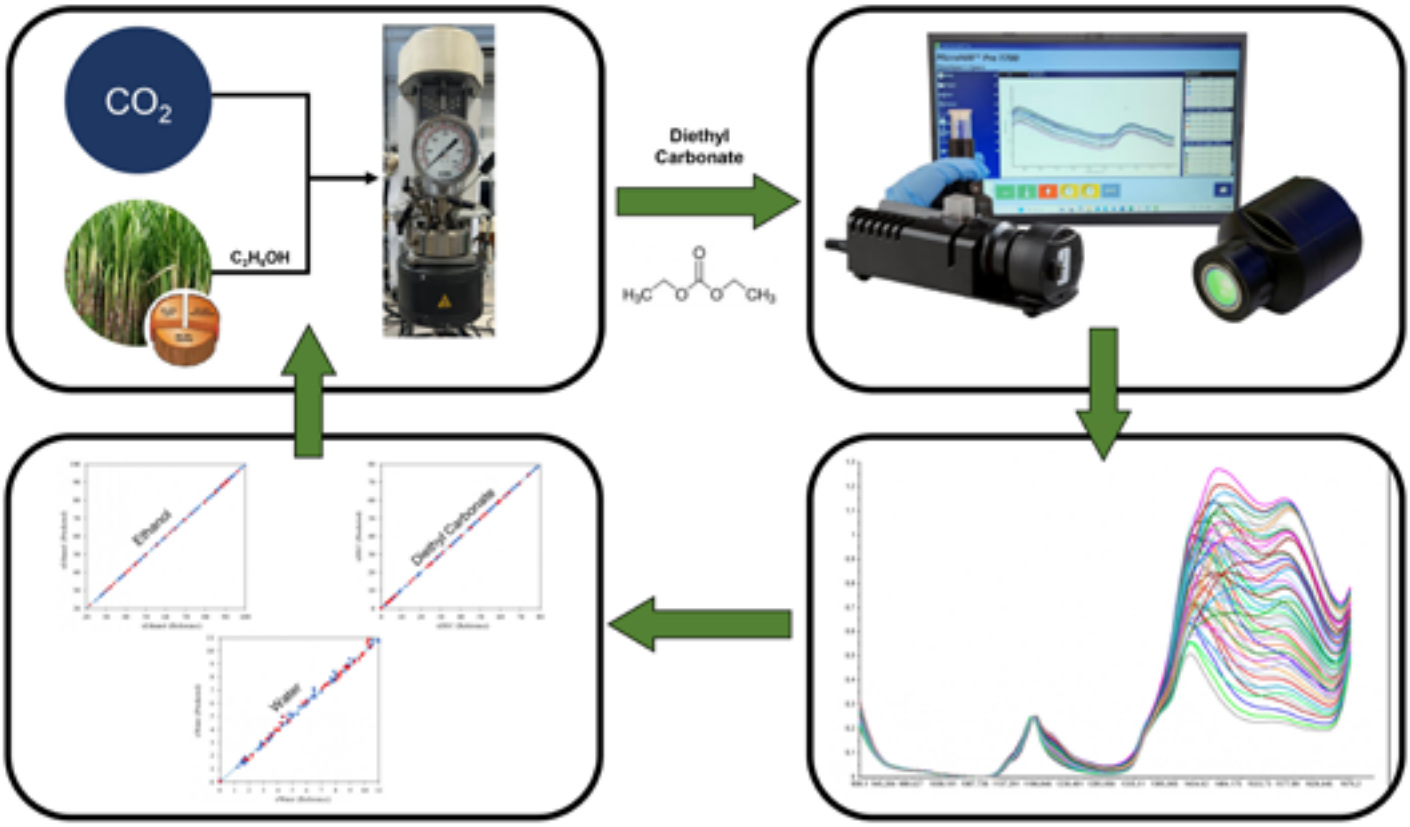
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ACKNOLEDGEMENTS: Authors wish to acknowledge the Brazilian funding agencies ANP/PRH, Finep and CNPq.

Keywords
Interval Partial Least Squares,MicroNIR,Organic Carbonate

P02.14.106

BENCHTOP VERSUS HANDHELD NIR SPECTROSCOPY FOR OLIVE MILL POMACE COMPOSTING MONITORING

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Olive mill pomace (OMP), the major byproduct of olive oil extraction, presents a significant environmental challenge. Composting is a promising OMP valorization strategy, transforming it into a valuable organic amendment for agricultural soils in the Mediterranean region [1]. However, successful field application requires precise monitoring of the composting process, including chemical composition analysis and maturity assessment. This study [2] investigates the applicability of NIR spectroscopy for monitoring OMP composting, comparing the performance of a benchtop Fourier Transform NIR (FT-NIR) spectrometer with a handheld miniaturized NIR device (VIAVI Micro-NIR 1700ES). For the latter, two different strategies were used to increase the representativeness of the spectra acquired for non-grinded samples, namely, the analysis with a windowed sapphire collar at different points and the rotation during measurement. Key compost quality parameters (pH, electrical conductivity (EC), C/N ratio, and organic matter content) were estimated during the composting process within a full-scale facility using Partial Least Squares (PLS) and Support Vector Machine (SVM) regression algorithms.

Results showed very good performance for the FT-NIR spectrometer in predicting compost quality parameters, with most achieving residual predictive deviation (RPD) values exceeding 2.0. For instance, pH prediction using SVM regression attained an RPD of 3.8 and a root mean square error of prediction (RMSEP) of 0.26, while LOI prediction yielded an RPD of 2.7 and an RMSEP of 2.0 % using PLS. On the other hand, the handheld NIR spectrometer provided reasonable predictions. For example, EC achieved RPD and RMSEP values around 2.0 and 0.60 dS m-1, respectively, demonstrating its suitability for rapid assessment of salinity-related parameters. In the case of pH, the narrower spectral range (908-1600 nm) resulted in poorer prediction in comparison with FT-NIR, whose broader spectral range (extending up to 2500 nm) captured critical absorption bands, such as those related to carbonates. Notably, the micro-NIR successfully predicted the C/N ratio, with the spinner accessory enhancing accuracy for intact samples. RMSEP values were comparable to or even better than those of the laboratory instrument, especially with the SVM model. In general, when analyzing intact samples with the miniaturized spectrometer, sample rotation decreased RMSEP values by 20 %, but performance was comparable to static measurements when using the sapphire windowed collar. In these conditions, averaging spectra from five replicate measurements in different positions compensated for sample heterogeneities and yielded reasonable prediction accuracy. Preprocessing techniques, such as scatter correction and derivatives, improved model performance by mitigating the effects of sample heterogeneity.

These findings highlight the potential of the handheld spectrometer for practical in-field applications. Based on the evaluation of the statistical parameters (RMSEP, RPD, and Passing–Bablok regression), all models could be considered good and reliable for analytical tasks. Consequently, miniaturized NIR spectrometers have the potential to become valuable spectral sensors for real-time compost process management. Their portability, speed, and cost-effectiveness make them particularly well-suited for on-site compost monitoring in large-scale facilities.

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ACKNOLEDGEMENTS: Project PID2020-118673RB-I00 by MCIN/AEI/10.13039/501100011033 and M.P.R. - FPU22/00016 fellowship (Spanish Ministry of Universities)

Keywords
Compost,FT-NIR,Handheld NIR spectrometer,SVM regression,PLS regression

P02.14.107

EVALUATION OF TWO NEAR-INFRARED SPECTROSCOPY TECHNOLOGIES FOR OLIVE POMACE CHARACTERIZATION IN BIOREFINERIES: A COMPARATIVE STUDY

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Olive pomace (OP), a major byproduct of olive oil production, is a complex lignocellulosic material with significant potential for valorization in biorefineries. To optimize conversion processes, obtain value-added products and establish its market value, precise and real-time characterization of OP is essential [1]. Continuous monitoring of key parameters, such as heating value (HV), moisture (M), ash (A) and volatile (V) content, is crucial for process control; however, traditional laboratory analyses are time-consuming, destructive and costly. In this context, near-infrared spectroscopy (NIRS) has emerged as a robust, non-destructive and rapid analytical technique for determining various parameters in organic matrices [2]. This study evaluates and compares two NIRS instruments suitable for at-line, in- line and on-line analysis for OP characterization: a FT-NIR Matrix F spectrometer (Bruker Optics) and a Perten DA-7300 (Perkin Elmer) diode array spectrophotometer. Both operate in reflectance mode, covering spectral ranges of 834.2-2502.6 nm and 950-1650 nm, respectively.

NIR spectra of intact and lyophilized OP were collected, and modified partial least square (MPLS) regression models were developed to predict eleven constituents - elemental composition (nitrogen (N), hydrogen (H), oxygen (O) and carbon (C)), protein (P), fiber, acid and neutral detergent fiber, lignin (L), antioxidant capacity (AC) and total polyphenols (TP) - as well as four physicochemical parameters: HV, M, A and V content. The first and second derivatives were evaluated as spectral signal preprocessing techniques.

The performance of the models developed was evaluated based on their coefficient of determination (R^2), standard error of cross-validation (SECV) and ratio of performance to deviation (RPD) to assess their precision and accuracy in predicting the studied parameters. For lyophilized samples, both instruments generally exhibited good predictive performance for most parameters. However, Matrix outperformed Perten in predicting L, AC and TP, although these parameters were not well predicted overall (R^2 = 0.40, 0.55 and 0.52 respectively). Perten instrument demonstrated higher precision in predicting C (R^2 = 0.85, SECV = 0.96, RPD = 2.62), O (R^2 = 0.86, SECV = 1.15, RPD = 2.69), P (R^2 = 0.71, SECV = 0.78, RPD = 1.85), HV (R^2 = 0.77, SECV = 419.59, RPD = 2.07) and A (R^2 = 0.85, SECV = 0.72, RPD = 2.54). Matrix instrument provided more accurate predictions for N (R^2 = 0.80, SECV = 0.15, RPD = 2.19) and H (R^2 = 0.72, SECV = 0.36, RPD = 1.3). For intact samples, Perten outperformed Matrix in all predicted constituents, achieving the best prediction values for M (R^2 = 0.783, SECV = 3.309, RPD = 2.131), V (R^2 = 0.813, SECV = 2.987, RPD = 2.313) and fibers (R^2 > 0.6).

The findings confirmed that online NIRS technology, combined with multivariate analysis, is a promising tool for characterizing OP. This approach allows for real-time monitoring of both its chemical composition and physicochemical properties, which are critical for optimizing biorefinery processes, to enhance process efficiency and product quality.

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ACKNOLEDGEMENTS: This work was funded by the Spanish Ministry of Science and Innovation (projects PID2019-105936RB-C21 and TED2021-130596B-C22). M. Carmona-Cabello acknowledges funding from the University of Cordoba (Margarita Salas grant UCOR01MS) and EPSRC (project 1856492). N. Sanchez-Avila thanks the Spanish Ministry of Economy and Competitiveness for an FPI grant.

Keywords
Olive pomace,Biorefinery,Near-infrared spectroscopy,Online analysis

P02.14.108

NEAR INFRARED SPECTROSCOPY AS IN SITU ANALYSIS TECHNOLOGY OF AGRICULTURAL AND FORESTRY BIOMASS FOR ENERGY USE.

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Search alternatives to fossil fuels have led to the use of raw materials of different origins to be more environmentally friendly. The use of biomass as a solid fuel is a source that is considered an alternative and assists in the transition away from elements such as coal and petroleum derivatives. Some types of biomasses of agricultural and forestry origin are used for energy production processes. Although it is considered to have less impact on the production of greenhouse gases, it must be considered that they continue to produce them, such as Nox. In addition to the intrinsic composition of elements such as chlorine and sulfur, which can cause corrosion in boilers. The better or worse combustion of biomass is also correlated with the amount of moisture and calorific value, which affects the development of efficient combustion and the production of pollutants particulates. There are also parameters that have a direct economic impact, for example, high moisture content reduces the biomass quality. Therefore, the quality of biomass is a vitally important element if it is to burn efficiently and help to be an energy source that really serves as a transition from fossil fuels. Traditional analysis is costly, requires a lot of analysis time, generates solvents that must be managed by specialized agents and requires qualified personnel to carry out some of these analyses. As an alternative to traditional analysis, technologies such as near infrared spectroscopy (NIRS) have emerged fast, reliable, non-invasive, non-destructive, simple to use and non-contaminant technique. The main objective of this study is the application of NIRS technology in the analysis of biomass, but also its use in situ, using portable technology, which allows much more efficient decision making, reducing costs and improving the production model of the plant. Two types of biomasses were analyzed, one of agricultural origin that includes a mixture of corn stover and cereal straw and another subset of wood chips of forest origin, comprising a mixture of different species, including pine, eucalyptus, oak and poplar, two predictive models were developed, one for agricultural species and another for forest species. A partial least squares regression model and several spectral signal pretreatments combining standard normal variate (SNV) and detrend (DT) or multiplicative scatter correction (MSC) were performed, as correction methods for the scattered light phenomenon and two derivative treatments for spectral signal smoothing, one first (1,4,4,1) and one second (2,4,4,1), were carried out, resulting in a set of four different chemometric treatments (1,4,4,1+SNV+DT; 2,4,4,1+SNV+DT; 1,4,4,1+MSC; 2,4,4,1+MSC), from the comparison of these we chose the one with the best result for each constituent. A total of 13 constituents were analyzed for agricultural biomass, obtaining an excellent fit for the R^2 statistic in the case of moisture (0.97) and chlorine (0.89) and a good fit for carbon (0.74), hydrogen (0.75), nitrogen (0.77), higher heating value (HHV) (0.76), HHVb (0.73), lower heating value (LHV) (0.72) and LHVb (0.83). For forest biomass, 11 constituents were analyzed, obtaining excellent R^2 results for moisture (0.99) and with a good separation between high, medium and low values for ash (0.56), HHV (0.63), LHV (0.64), hydrogen (0.61) and sulfur (0.60). Considering the results, it can be concluded that near infrared spectroscopy has great potential to be used in field characterization process of agricultural and forest biomasses for energy use. In this way, it has potential use for the characterization of solid fuels, thus reducing analysis time and costs and allowing it to be used in power plant routines for quick and effective decision-making.

Keywords
Near infrared spectroscopy,In situ analysis,Biomass,Energy production,Circular economy

P02.14.109

ENHANCING BIOGAS PRODUCTION WITH NEAR-INFRARED SPECTROSCOPY: A GREEN TECHNOLOGY FOR SUSTAINABLE ENERGY

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Minimizing food waste while seeking sustainable and environmentally friendly energy solutions is increasingly important. Biogas presents a promising renewable energy source by converting organic waste into energy and supporting a circular economy. However, its production remains complex due to variations in feedstock composition, which affect methane yield and process efficiency. Additionally, obtaining representative feedstock samples is challenging due to the heterogeneous nature of biogas substrates, impacting the accuracy of traditional analytical methods. Conventional laboratory analyses for feedstock characterization are often time-consuming, labor-intensive, and require specialized equipment, limiting real-time decision-making in biogas plants. Addressing these challenges requires innovative approaches, such as advanced analytical techniques, to optimize biogas production and improve its viability as a renewable energy solution. Near-Infrared Spectroscopy (NIRS) has emerged as a rapid, non-destructive tool for assessing key feedstock properties. As a green technology, NIRS offers an environmentally friendly alternative to conventional chemical analysis methods, reducing the need for hazardous reagents and extensive sample preparation [1].

This study examines the potential of both benchtop and portable NIR spectroscopy for classifying biogas feedstocks and predicting critical parameters such as moisture content, volatile solids, and ash content. By applying chemometric techniques, NIRS can differentiate various feedstock types—including agricultural residues, food waste, manure, and sewage sludge—based on their spectral fingerprints. Additionally, this study explores how NIRS can address sampling challenges by providing a more efficient and reliable method for analyzing heterogeneous feedstocks, improving accuracy in biogas process monitoring.

Integrating NIRS into biogas operations could replace time-consuming traditional laboratory analyses, enabling real-time, on-site feedstock assessment. This approach has the potential to enhance adaptive process control, optimize substrate mixing, and improve digestion efficiency. By facilitating more efficient feedstock characterization, NIRS could contribute to more stable biogas production, increased methane yields, and greater economic viability of anaerobic digestion systems. Ultimately, adopting NIRS technology may help advance the transition toward more sustainable and cost-effective biogas energy production.

ACKNOLEDGEMENTS: Tomasz Pawel Czaja, Søren Balling Engelsen, Why nothing beats NIRS technology: The green analytical choice for the future sustainable food production, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy Volume 325, 2025, 125028

Keywords
Biogas production,Feedstock,Portable NIR,Sustainable energy

THEORY & FUNDAMENTALS

P02.15.110

SEPARATION AND INTEGRATION OF IMAGE INFORMATION IN THE VISIBLE AND NEAR-INFRARED SPECTRAL RANGES USING ABSORPTION PROPERTIES OF COMMERCIAL DYES

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By studying the absorption properties of dyes [1], we introduce the concept of twin colours and twin dyes. Dyes that appear visually identical in the visible part of the spectrum are referred to as twins, as their absorption properties in this spectral range are the same [2]. However, these dyes exhibit different absorption properties in the near-infrared (NIR) spectrum, ranging from 700 to 1000 nm [3]. This difference in absorption enables new possibilities for applications in various fields, including art [4], industry [5], and forensics.

Experimental work involving the mixing of dyes and examples of their application focuses on tempera dyes. A spectrogram of light absorption in the spectral range from 400 to 1000 nm was created for each newly developed dye. During image analysis, different light blockades at specific wavelengths were used to highlight variations in absorption between the visible and near-infrared spectral ranges. For the visible part of the spectrum, numerical values in the L*a*b* colour space were assigned to each dye mixture, facilitating precise tracking of colour differences.

The experimental process involves designing and creating mixtures of twin dyes while minimizing the delta E value between two or more twins. The goal is to achieve identical colours in the visible spectrum while preserving differences in the near-infrared range. Twin dyes were created for brown, orange, green, and blue tones. Examples of the separation and integration of image information in the two spectral ranges, achieved using commercially available dyes, are also presented. This research opens up possibilities for a wide range of applications, including the analysis and creation of new visual effects.

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ACKNOLEDGEMENTS: The authors conducted this research independently.

Keywords
absorption,twin dyes,tempera,visible spectral range,near-infrared spectral range

P02.15.111

IDENTIFYING PHASE PATTERNS IN GLYCOLYTIC OSCILLATIONS FOR SUPPORTING BAND ASSIGNMENTS IN THE NEAR-INFARED REGION

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[Introduction and Object]

Band assignment in the NIR spectrum is challenging because of many overlapping bands due to OH, CH, and NH overtones and combinations. For biological samples, it is more complicated because of the significant influence of water. To solve these problems, we focused on an oscillation reaction observed in the glycolytic pathway of yeast. The pathway intermediates periodically oscillated with the same frequency but differs in phase during the glycolytic reaction [1]. The period of the glycolytic oscillation in yeast has been observed not only in intact cells but also in cell-free extracts. The phase relationships among pathway intermediates have been identified through stepwise sampling for enzyme activity measurements and mathematical modeling [1,2]. We aim to separate the NIR spectral patterns for each metabolite group based on the phase differences. As a first step, we experimentally confirmed the phase relationships of metabolites given by theoretical models [1, 2] by monitoring yeast extracts during glycolytic oscillations using a ^{1}H-NMR.

[Materials and Methods]

Baker's yeast (Saccharomyces cerevisiae) was crashed and extracted to prepare a yeast extract. To induce glycolytic reaction, 1 M trehalose was added to the extract. After that, it was stared to measure soon by ^{1}H-NMR (Bruker, AVNEO-800). Measurements were performed at approximately 65 seconds intervals for two hours at 25 degrees Celsius. ^{1}H-NMR spectra were preprocessed and analyzed by using TopSpin 4.2.0 software and Python.

[Results and Discussion]

From the ^{1}H-NMR spectra, 33 pathway intermediates were identified. Time-series data of area intensities for each peak were used to classify the pathway intermediates into four groups: constant, monotonically increasing or decreasing, and oscillatory. Fourier transformation of the time-series data of the oscillating group revealed that they had a common frequency. The period derived from this frequency was approximately 16 minutes. Next, we examined the phase relationships of the pathway intermediates. Using ATP as a reference, NAD+ and ADP showed an antiphase relationship, while glucose-6-phosphate included glucose peak showed a phase difference of approximately 133°. These results support the relationships suggested by previous research [1, 2]. This is the first study to directly observe the oscillations of the pathway intermediates in yeast extracts and clarify the phase relationships. The analysis of NIR spectra using phase differences and the band assignment will be presented in a separate paper.

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ACKNOLEDGEMENTS: This work was supported by JSPS KAKENHI Grant Number JP23K26687.

Keywords
Yeast extract,Glycolytic oscillation,1H-NMR,Metabolites

ROMA NIR2025

