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Effects of Processing Methods on the Physical Properties of Aquafaba Powder: Time-Domain Nuclear Magnetic Resonance Analysis

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ABSTRACT

Aquafaba, the water remaining after cooking chickpeas, has been a promising emulsifier and stabilizer in food products. Despite its potential, the variability in its composition and dry matter content poses challenges for its consistent use. This study aimed to enhance the dry matter content of aquafaba through different processing methods—microwave heating, microwave-infrared heating, and conventional boiling—and to evaluate how these methods affect the physical properties of the resulting powders. The experiment also explored the effect of overnight soaking of chickpeas on the dry matter yield. The powders produced were characterized using time-domain nuclear magnetic resonance (TD-NMR) to investigate their water absorption, hydration behavior, and emulsification properties. Results showed that microwave and microwave-infrared heating significantly increased the dry matter content compared to conventional boiling. Furthermore, overnight soaking of chickpeas led to a notable increase in dry matter yield across all processing methods. TD-NMR analysis revealed that microwave-infrared samples exhibited improved hydration rates and more stable emulsions over time compared to those processed with other methods. These findings suggest that alternative processing techniques, especially microwave-infrared heating, can improve the consistency and functionality of aquafaba as an ingredient in food products. By increasing the dry matter content and enhancing hydration properties, these methods may provide a more reliable plant-based emulsifier. This study contributes to the development of novel, sustainable approaches in food processing that can enhance the quality and performance of plant-based ingredients across various applications.

1 | Introduction

Chickpea (*Cicer arietinum* L.), also known as Bengal or garbanzo beans, belongs to Fabaceae family [1]. Chickpea contains approximately 60% of carbohydrates [2]. Among the different pulses, chickpea has been reported to have one of the highest values of protein content, which is around 20%–25% [3]. Albumin and globulins are the predominant proteins, comprising 8%–12% and 53%–60% of the total protein content, respectively [2]. Aquafaba, a by-product of the canned and frozen legume industry, refers to the viscous liquid obtained from cooking or soaking legumes, such as chickpeas, in water [4]. This liquid contains soluble plant compounds, including proteins, carbohydrates, and saponins, which give it unique functional properties, such as emulsification, foaming, and gelling, making it a valuable ingredient in various food applications [5]. The term "aqua" comes from the water, and *faba* denotes the bean. Aquafaba consists of 92%–95% water, 0.95%–1.5% protein (wb), and approximately 3.5% carbohydrates. It also contains saponin in the range of 4.5–30 mg/g, which has been shown

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to enhance the emulsifying property of the aquafaba [4, 6]. Functional properties of aquafaba come mainly from oligosaccharides, low molecular weight, and water-soluble proteins and saponins [7, 8].

Microwave heating has many potential uses in food industry because it is able to reduce the time and energy required for essential unit operations such as drying and evaporation [9]. In addition to regular microwave heating, MW-IR contains halogen lamps that supply near-infrared radiation and therefore provides higher penetration depth [10]. Moreover, a halogen lamp has the ability to heat the inside of the oven and the surface of the food material, and consequently, enhanced heating could be ensured [9]. During microwave heating, internal pressure gradients caused by rapid heating and evaporation can lead to pore formation within the food matrix [10, 11]. These pores play a significant role in improving moisture transport and may also facilitate the leaching of macro- and micronutrients during processing [12, 13]. Furthermore, microwave energy can increase the solubility of proteins through denaturation or degradation, further impacting the structural and functional properties of food [14, 15].

Time-domain nuclear magnetic resonance (TD-NMR) relaxometry is a simple, quick, low-cost, and nondestructive method [16, 17], which is based on the measurement of T_1 (*longitudinal*) and T_2 (*spin–spin*) relaxation times. In food products, T_1 represents the time required for excited protons to return to thermal equilibrium with their surroundings, providing insights into water binding, hydration, viscosity, and crystallization in various food matrices [18–20]. T_2, on the other hand, reflects interactions between protons and provides information about water mobility, which can be linked to adulteration, syneresis, and staling [21–27]. Decomposing the NMR signal helps identify different proton populations—primarily those in water, but also in other molecules such as carbohydrates and proteins [18, 25, 28]. This analysis is essential for investigating how processing methods like heating, cooling, or chemical reactions affect these proton components. Such processes alter the chemical environment and, consequently, the resonance frequencies of these protons, detectable as changes in the NMR signal. This approach allows us to characterize the specific modifications each processing method induces in the proton populations of various molecular components [18, 25, 28].

This study aims to explore conventional, microwave and microwave-infrared (MW-IR) extraction techniques that are known for their efficiency in extracting proteins and other compounds. These methods were chosen to increase the dry matter, saponin, and protein content of aquafaba. Different than the current aquafaba studies conducted by other researchers, saponin and amino acid content of powder aquafaba was characterized by LC-MSMS method. Another novel aspect of this study was to use TD-NMR relaxometry of the powders to investigate their behavior.

2 | Material and Methods

3 | Materials

Chickpeas (*C. arietinum*) (Reis Gıda A.Ş. Kastamonu, Turkey) were purchased from local market in Ankara, Turkey. Distilled water was used to prepare the chickpea solution. All the other chemicals were supplied form Sigma-Aldrich.

3.1 | Preparation of Aquafaba

Aquafaba was prepared following the method of [29] with slight modifications. First, chickpeas were washed, and excess water was discarded. Then, washed chickpeas were soaked in distilled water at a chickpea-to-water ratio of 1:3 (w/w) at 4°C for 16 h [30]. Afterward, to increase the solid content of aquafaba, different methods were applied (conventional heating, microwave heating, and MW-IR cooking) to increase dry matter content of aquafaba, which were explained below. Microwave heating and microwave-infrared were used as alternatives to the conventional "boiling" process. The abbreviations and process conditions are summarized in Table 1.

3.1.1 | Conventional Heating

Chickpeas were cooked in an electric pressure cooker at a chickpea-to-water ratio of (1:3) (Sinbo-Sco-5033, Istanbul, Turkey), where 500 g of chickpeas was subjected to 20 min cooking [29].

3.1.2 | Microwave Heating

Chickpeas were cooked in a microwave oven at a chickpea-to-water ratio of (1:3) (Arçelik, Istanbul, Turkey), where 600 g of chickpeas was subjected to 50 min cooking at the maximum power (900 W), which operated with 80% efficiency according to IMPI 2L test [31]. Chickpea-to-water ratio, power, and time were determined with preliminary experiments.

It is obvious that 50-min microwave heating time is not feasible at all. However, only after 50 min, dry matter was enough for spray drying. Same time was used in all microwave experiments. The goal was to see how microwave heating would change the properties of the powder.

3.1.3 | MW-IR

Chickpeas were cooked in a MW-IR oven at a chickpea-to-water ratio of (1:3) (GE Appliances, Louisville, KY, USA), where 300 g

TABLE 1 | Processing conditions that were used to increase the aquafaba yield.

Process	Abbreviation	Time (min)	Chickpea-to-water ratio
Conventional	PC	20	1:3
Microwave heating	MW-C	50	1:3
Microwave-infrared	MW-IR	50	1:3

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of chickpeas was subjected to 50 min cooking at 100% upper power, 100% lower power, and 50% microwave power.

In preliminary experiments for both microwave heating techniques, chickpea-to-water ratio, power, and time were tested. First, cooking time was held constant, whereas power settings and chickpea-to-water ratios were varied, testing 1:3 and 1:5 at 80% and 100% power. The highest dry matter yield was observed at a 1:3 ratio under 100% power. Then, cooking durations of 20–60 min at the same power setting were subsequently examined. It was determined that extending the cooking time to 40 min increased yield, whereas durations beyond 60 min were found to be ineffective due to rapid water evaporation, which impacted spray drying feasibility. Therefore, 50 min process was selected for further processing.

Due to the differing behaviors observed in the microwave treatments (MW-C and MW-IR), the same microwave power settings could not be used for both methods. Specifically, regardless of the container size and chickpea-to-water ratio, the MW-IR processing consistently caused overflow from the largest container that could fit in the microwave. To prevent this and accommodate the unique dynamics of MW-IR processing, the microwave power was accordingly reduced, whereas the upper and lower power settings were maintained at 100%. This adjustment was necessary to achieve comparable processing conditions without compromising the integrity of the experimental setup.

As seen in Table 1, chickpea-to-water ratios were kept same for all experiment. Parameters were adjusted to obtain the best dry matter yield as explained in preliminary section. In fact, treatment time was fixed as 50 min for each treatment except for the conventional (pressure cooker) method because it is a conventional production method for cooking chickpeas.

To ensure clarity, throughout this manuscript, "MW-C," "MW-IR," and "PC" refer to the powder or liquid aquafaba obtained from microwave-conventional, microwave-infrared, and conventional processing methods, respectively, rather than the processing techniques themselves when comparing results. However, when explicitly discussing the methods, these abbreviations refer to the processing techniques.

3.1.4 | Further Processing Techniques

Following the application of three different processing methods, the obtained aquafaba was treated in two ways before drying. In the first, the aquafaba was immediately drained and separated from the chickpeas, and in the second method, the chickpeas and water were kept overnight at 4°C and then filtered after 24 h. The former is abbreviated as "S" (n**on-soaked**), and the latter is abbreviated as "O" (**overnight**).

3.2 | Preparation of the Powder Aquafaba

A pilot-scale *spray dryer* (UNOPEX, İzmir, Turkey) was used to obtain powder aquafaba. Spray drying parameters were selected as 8–9 mL/min feed flow rate, 150°C inlet temperature, 83°C outlet temperature, and 80% aspiration rate [32]. Dried and powdered aquafaba was stored at room temperature in a glass container wrapped with parafilm.

To ensure consistency across analyses, powder aquafaba samples were prepared in batches over approximately 2 months, yielding 100 g per process. This batch preparation approach was used to minimize compositional variations that could arise. As food-based materials can naturally exhibit slight compositional differences, even within the same processing method, a single batch per condition was prepared and used across all experiments. This ensured better reproducibility and reliable comparisons in analytical results.

3.3 | Characterization of Liquid and Powder Aquafaba

3.3.1 | Dry Matter Content

The solid content of the liquid aquafaba was measured using an oven at 105°C, and the samples were kept in the oven for 3 h. Dry matter measurements were continued until the difference between measurements was less than 0.5% [33]. At least three replicates were used for the analysis.

3.3.2 | Water and Oil Holding Capacities (WHC/OHC)

Water and oil holding capacities (WHC and OHC) were carried out for powder aquafaba. For water holding capacity (WHC) measurements, 0.1 g of aquafaba powder was mixed with 10 mL of distilled water, whereas, for the oil holding capacity (OHC), 0.1 g of sample was mixed with 10 mL sunflower oil [34]. The mixtures were then centrifuged at 1960 × g for 30 min. After 30 min, the resulting supernatants were drained, and the centrifuge tubes containing the sediment were weighed. At least three replicates were used for the analysis.

W_0 is the weight of the dry sample, W_1 is the weight of the centrifuge tube and dry sample, and W_2 is the weight of the centrifuge tube and the sediment.

$$WHC\,/\,OHC = \frac{W_{2-}W_1}{W_0} * 100$$

3.3.3 | Hygroscopicity

Hygroscopicity was conducted for only powder samples. 1 g of powder aquafaba was spread in the petri dish to increase the surface area. Then, the petri dish was placed in a desiccator where the humidity and temperature were 75% and 25°C, respectively [35]. When equilibrium was reached, the hygroscopicity was calculated as

$$\text{Hygroscopicity} = \frac{\text{Final moisture content} - \text{inital moisture content}}{\text{Initial moisture content}}$$

3.3.4 | TD-NMR Relaxometry Experiments

TD-NMR experiments were performed using a 0.5-T (20.34 MHz) system (Spin Track GmbH, Kirchheim/Teck, Germany). These

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NMR experiments were carried out for both liquid and powder aquafaba. Four different types of measurements were conducted using TD-NMR Relaxometry which were explained in detail below.

3.3.4.1 | T_1 Times of Powder Samples. For powder aquafaba, only T_1 times were measured as T_2 times were very short to measure. Saturation recovery pulse sequence was used for the T_1 measurements. The time of observation, initial step and number of points were set to 600,000 μs, 4000 μs, and 16, respectively.

3.3.4.2 | T_2 Relaxation Time of Liquid Aquafaba. For liquid aquafaba, on the other hand, T_2 values were measured. CPMG (Carr–Purcell–Meiboom–Gill) sequence was used to measure the T_2 relaxation times. Echo time was set to 8400 μs and the number of echoes changed between 1000 and 1300. The T_2 relaxation data were processed using a monoexponential fitting model to determine the relaxation times. The fitting was performed using a least-squares method, and the goodness of fit was evaluated using R-squared values to ensure accuracy.

3.3.4.3 | Emulsification Properties Determination of Powder Aquafaba by TD-NMR. For emulsification properties of powder aquafaba, 0.5-g aquafaba powder was dissolved in 20 mL of distilled water until it becomes homogeneous. Then, 5 mL of oil was added to mixture and homogenized at 7500 rpm for 5 min to form emulsion. T_2 relaxation time were measured for the first day and 14th day of emulsion. CPMG sequence was used to measure the T_2 relaxation times. Echo time was set to 8400 μs, and the number of echoes changed between 1000 and 1300.

The relaxation spectra were obtained by applying non-negative least squares (NNLS) analysis to the T2 decay curves. For this purpose, MATLAB was used for mathematical analysis, whereas one-dimensional NNLS analysis was performed using PROSPA software (Magritek Inc., Wellington, New Zealand).

3.3.4.4 | Hydration of Powder Aquafaba by TD-NMR. T_2 relaxation times were measured at 5-min intervals during the first hour and at 10-min intervals during the remaining 1.5 h, making a total observation period of 2.5 h by pipetting the 100 μL of water to 0.8 g of powder aquafaba evenly distributed in a glass vial. The rate of change of T_2 times was measured to interpret the hydration ability of powder aquafaba. For T_2 relaxation time measurement, echo time was set to 800 μs, and the number of echoes changed between 300 and 1300. Relaxation delay of 1000 μs was used for all measurements.

3.3.5 | Statistical Analysis

Analysis of variance (ANOVA) (Minitab Inc., Coventry, UK) was used to examine all experimental data in order to determine whether there had been a significant change between samples. A 95% confidence interval was utilized for the Tukey's comparison test. At least five replicates were used for all analysis. Before analysis, the ANOVA presumptions were verified, and if needed, outliers were eliminated from the data set.

4 | Results and Discussion

4.1 | Determination of the Dry Matter Content

Different production methods yielded different dry matter content in the aquafaba due to different mechanisms of each processing method (Table 2). The conventional cooking method yielded the lowest dry matter, which agreed with previous studies [4, 7]. Among the microwave processing methods, MW-C gave the highest dry matter, which was expected, because higher power was used in MW-C sample. This means that internal pressure generated by microwave was greater for MW-C sample than MW-IR sample. Internal pressure gradients form as a result of internal heating and evaporation and significantly improve the moisture transport, which leads to opening of the pores more [36]. Same microwave power could not be used for both microwave methods because independent of container and chickpea-to-water ratio, MW-IR processing caused overflow from the largest container that could fit in the microwave. Therefore, microwave power was reduced, and upper and lower power was set to 100%.

In addition to the processing methods, the effect of overnight soaking was also examined. It was observed that overnight soaking significantly increased the dry matter of aquafaba for all production methods possibly due to the prolonged interaction between chickpeas and water ($p < 0.05$). The combination of microwave processing methods and the overnight treatment appears to increase dry matter content more. The change in the dry matter content was greater for MW-C produced aquafaba (2.75%–5.33%). Microwaves penetrate into food and cause the internal temperature to rise.

TABLE 2 | Dry matter of liquid aquafaba and WHC, OHC, and hygroscopicity of powder aquafaba.

Process	Dry matter (%)	WHC	OHC	Hygroscopicity (%)
PC-S	1.12 ± 0.01^{e}	3.98 ± 0.24^{a}	3.93 ± 0.27^{a}	4.39 ± 0.36^{d}
Mw-C-S	2.75 ± 0.02^{c}	2.56 ± 0.17^{c}	3.51 ± 0.16^{b}	5.10 ± 0.26^{c}
Mw-IR-S	2.38 ± 0.02^{d}	1.47 ± 0.08^{d}	3.46 ± 0.17^{b}	0.44 ± 0.03^{f}
PC-O	2.37 ± 0.03^{d}	3.68 ± 0.15^{a}	3.87 ± 0.16^{a}	31.62 ± 0.31^{a}
Mw-C-O	5.33 ± 0.02^{a}	3.15 ± 0.12^{b}	2.87 ± 0.20^{c}	7.99 ± 0.22^{b}
Mw-IR-O	3.38 ± 0.01^{b}	2.67 ± 0.06^{c}	3.80 ± 0.12^{ab}	2.01 ± 0.10^{e}

Note: Values within the same column, indicated by different lowercase letters, are significantly different.
Abbreviations: MW-C = microwave, MW-IR = microwave-infrared, O = overnight processing, PC = conventional, S = non-soaked.

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High temperature generates water vapor inside the food and results in internal pressure gradient [37, 38]. As mentioned before, this internal pressure gradient was higher in MW-C method, which results in expansion of the pore more. Moreover, time could have been insufficient for the dry matter to diffuse to the liquid phase in the non-soaked procedure. Hence, soaking overnight enhanced the dry matter transfer to a greater extent.

4.2 | WHC and OHC

WHC and OHC are mainly influenced by the chemical composition, microstructure such as size and distribution of the pores and surface area, and also the interactions between macromolecules. The microwave process can enhance the pore structure by creating localized heating and rapid vapor generation, leading to the expansion of internal spaces [10, 12]. This contributes to the formation of a more porous structure, which plays a critical role in water absorption, oil retention, and other functional properties. WHC plays an important role in baking industry in terms of reducing the moisture loss and providing freshness and moist mouthfeel [39]. It has been emphasized in different studies that composition has an effect on WHC and OHC [40, 41]. WHC and OHC results of aquafaba powders were given in Table 2. It was hypothesized that differences in the composition of aquafaba may lead to the observed differences in their properties. PC samples showed the highest WHC for both non-soaked and overnight aquafaba processing ($p < 0.05$). This may be due to the fact that the high pressure and temperature of the pressure cooker can damage starches and denature the proteins in the aquafaba, and hence, it becomes more gelatinous, which can enhance the water holding capacity of aquafaba. MW-C and MW-IR have lower WHC compared to PC for both non-soaked and overnight processing. Microwave treatment causes internal pressure, which enhances the opening of the pores. Therefore, dry matter transfer might have increased, and the transfer of higher molecular weight constituents such as starch and insoluble fiber that can decrease WHC could also have taken place more. It is known that chickpea contains a higher proportion of insoluble fiber compared to soluble fiber [2]. Because insoluble fibers and starch are weak water binders, they might have decreased WHC [42, 43]. The most known chickpea proteins are albumin and globulin, which are water soluble and salt soluble fractions, respectively. With the opening of the pores, globulin transfer that is the larger and insoluble protein fraction may have enhanced [44].

OHC, which is defined as the physical entrapment of oil molecules, is attributed to enhanced hydrophobicity [45]. OHC may be altered with processing conditions, composition, temperature, and particle size. Moreover, amino acid content, protein structure, and surface hydrophobicity is known to have a high impact on the OHC of proteins [46]. OHC is expected to increase with increasing concentration of nonpolar side chain of the proteins because these amino acids play an effective role in the protein–fat interaction [34, 47].

In this study, it was observed that overnight soaking did not have a significant effect on the OHC of MW-IR and PC powder aquafaba ($p > 0.05$). However, overnight soaking had a significant decrease in OHC of MW-C sample ($p < 0.05$). It was observed that overnight soaking had the opposite effect on WHC and OHC for MW-C sample did not change for other samples. WHC and OHC of aquafaba powders tend to show an inverse relationship. This is expected because a decrease in WHC might mean a decrease in polar groups, whereas an increase in OHC might mean a decrease in these polar groups [48, 49]. Besides change in the polarity, network created by macromolecules in the aquafaba powder might have also affected OHC. When oil is added to aquafaba powder, which is rich in macromolecules, a more viscous environment might have been formed [34]. Due to these macromolecules, it may be more difficult for oil droplets to separate and therefore they become held in the network. Also, insoluble fibers in powders might have been soluble in the oil phase, which again results in viscosity increase. These macromolecules and insoluble fibers could act as a gel network that traps the oil and changes OHC.

4.3 | Hygroscopicity

Hygroscopicity refers to a substance's ability to adsorb moisture from the surrounding air. In general, plant-based proteins tend to be hygroscopic due to the high content of free amino acids and peptides, which have good moisture adsorption ability [50]. Spray drying results in instant moisture loss and creates an amorphous structure and enables to produce highly hygroscopic powder samples [51]. That is why aquafaba powders are expected to be hygroscopic. It was worth noting that the final moisture content of the spray-dried protein powder can also affect its hygroscopicity.

The hygroscopicity of aquafaba after various processing methods displayed diverse patterns that were influenced by both microwave processing techniques and treatments shown in Table 2. Especially, the overnight treatment had a significant effect on hygroscopicity. This significant rise in hygroscopicity suggests that soaking chickpeas in water overnight leads in a stronger affinity for water absorption. In contrast, for all non-soaked procedures, each method showed lower hygroscopicity when compared to overnight processing, suggesting that the differences in the composition of aquafaba caused by the absence of an overnight soaking step, as well as the faster drainage and separation process, may reduce the water absorption capacity of the powders. As seen in Table 2, PC-O sample was the most hygroscopic samples followed by MW-C-O. On the other hand, Mw-IR (*both overnight and non-soaked ones*) were the least hygroscopic samples. Microwave and pressure-based treatments influence starch structure by inducing gelatinization, a process where heat and water disrupt the crystalline structure of starch granules. This leads to the leaching of amylose and deformation of amylopectin [52, 53]. Amylopectin, which is highly hygroscopic due to its branched structure allowing hydrogen bonding, loses its ability to bind water when its structure is deformed during gelatinization [52]. As will also be explained later in NMR experiments, MW-IR caused higher extent of gelatinization due to its heating mechanism. During gelatinization, amylose that is the amorphous part of starch let water penetrate through starch granules. On the other hand, amylopectin deforms and loses its ability to bind water [52, 54]. The decrease in hygroscopicity of the samples was explained with the amylopectin deformation of the branched structure.

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4.4 | TD-NMR Relaxometry

4.4.1 | T_1 Times of Powder Aquafaba

TD-NMR relaxometry is a nondestructive method to examine the mobility of protons under an external magnetic field [16]. T_1 value, which is also known as spin–lattice relaxation time, is highly dependent on the proton mobility. And in food samples, protons mostly come from water and water mobility is highly affected by dipolar interactions [18]. As dipolar interactions strengthen, the relaxation time of protons becomes shorter due to the restricted mobility, and T_1 values decreases. On the other hand, in some cases, as water content thus mobility increases, T_1 value increases [55].

In this study, changes in T_1 relaxation time were monitored while increasing the moisture content for all powder aquafaba for process changing from non-soaked to overnight as shown in Table 3 (only samples produced in the same way were compared based on being non-soaked or overnight). However, an unexpected trend for T_1 values was observed. Typically, moisture content and T_1 relaxation time show a positive correlation, where higher water content leads to increased T_1 values. However, in some samples, T_1 first decreased and then increased, which does not follow the expected trend.

Here, a phenomenon that should be enlightened is the *primary hydration sphere* (Figure 1) [56]. Due to the dipolar nature of water, bearing partial negative charge, oxygen positions toward to positively charged ion, whereas hydrogen positions toward to opposite charge [56]. Only a part of the water molecules interacts with ions and forms a spherical shell. This shell is called the *primary hydration sphere*. Within this layer, water is considered bound. The water molecules outside of this layer are called as bulk water and contribute T_1 [57].

In legumes, moisture content versus T_1 graph tends to have U-shaped curve [56]. A representative plot is given in Figure 1. To explain this behavior, studies on legumes are referenced, where moisture versus T_1 curves exhibit a U-shaped trend [56]. In this study, T_1 first decreases as moisture increases up to a critical point and then starts increasing. Although previous research has reported this transition occurring at around 20% moisture content, in our study, the same trend may be occurring within the 1%–9% moisture range (Table 3). The lowest T_1 point in this curve corresponds to the formation of a primary hydration sphere around macromolecules, which impacts water mobility. This phenomenon, explained by the water replacement hypothesis, accounts for the inverse relationship between water content and T_1 in very dry conditions (Region I). Beyond this point, T_1 increases proportionally with moisture content (Region II) [56].

In addition, the differences in T_1 may have arisen from the difference in crystallinity in aquafaba powders. Although MW-IR-S and MW-C-S showed higher moisture content, their T_1 times were the lowest ones, whereas PC-O had lower moisture content and longer T_1 times. These two opposite cases showed that aquafaba powders could differ in crystallinity. Accordingly, it is expected for overnight samples to have higher crystallinity. Crystallinity could be affected by amylopectin (crystalline part) and amylose (amorphous part) content of different aquafaba powders [58]. Soaking allows water to penetrate starch granules, partially disrupting their crystalline regions during gelatinization [59]. Upon drying, this can lead to the reorganization and stabilization of crystalline structures, resulting in increased crystallinity [60]. The extent of this effect is influenced by the relative amounts of amylopectin (the crystalline component) and amylose (the amorphous component) in the starch, as these components determine the structure and stability of the crystalline regions.

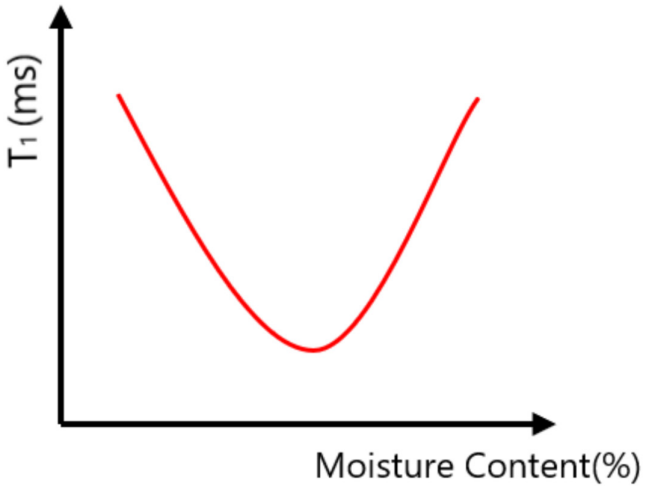


FIGURE 1 | Representative plot of T_1 versus moisture content for legumes.

TABLE 3 | Moisture content, aw, and T_1 times of powder aquafaba and T_2 times of liquid aquafaba.

Process	Moisture content (%)	aw	T_1 (ms)	T_2 (ms)
PC-S	8.66 ± 0.28^{a}	0.29 ± 0.00^{a}	75.40 ± 2.34^{b}	1426.20 ± 16.92^{a}
Mw-C-S	7.49 ± 0.26^{b}	0.24 ± 0.00^{b}	66.59 ± 2.82^{c}	1009.25 ± 36.47^{b}
Mw-IR-S	9.05 ± 0.08^{a}	0.28 ± 0.00^{a}	62.83 ± 1.49^{c}	983.00 ± 9.30^{b}
PC-O	1.05 ± 0.17^{e}	0.21 ± 0.00^{c}	77.80 ± 3.55^{ab}	974.74 ± 27.22^{b}
Mw-C-O	3.62 ± 0.28^{d}	0.14 ± 0.01^{e}	77.66 ± 2.54^{ab}	797.71 ± 16.88^{c}
Mw-IR-O	5.40 ± 0.08^{c}	0.16 ± 0.01^{d}	82.55 ± 2.74^{a}	687.27 ± 7.03^{d}

Note: Values within the same column, indicated by different lowercase letters, are significantly different.
Abbreviations: MW-C = microwave, MW-IR = microwave-infrared, O = overnight processing, PC = conventional, S = non-soaked.

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4.4.2 | T_2 Times of Liquid Aquafaba Samples

Another parameter investigated in this study was the T_2 times. The T_2 relaxation time, an important parameter that reflects molecular mobility in aquafaba, reveals unique variations that are influenced by different processing processes. T_2 results revealed that there is a strong negative correlation between T_2 (Table 3) and the dry matter content (Table 2). (Pearson correlation, $r = -0.76$, $p < 0.05$). PC-S, owing to the least dry matter content, gave the largest T_2 values, which was indicator for increased molecular mobility. This was in good agreement with previous studies. As the dry matter content increases, T_2 decreases due to reduced molecular mobility, as water acts as a plasticizer that increases T_2 by enhancing molecular flexibility and reducing intermolecular interactions [17].

According to the results given in Table 3, T_2 values of liquid aquafaba samples decreased as dry matter content (Table 2) increased during overnight soaking. MW-IR-O has the lowest T_2 value, indicating reduced molecular mobility and possibly higher dry matter content. However, although dry matter content of MW-IR sample was increased during overnight soaking, T_2 decreased disproportionately compared to the MW-C sample, which had a significantly higher dry matter content. This unexpected decrease in T_2 may be attributed to the unique heating mechanism of MW-IR, which provides more uniform heating and enhanced vaporization compared to other methods. Following the MW-IR heating process, aquafaba was more viscous and had a browner color compared to the MW-C and PC samples. This difference is likely due to the longer penetration depth of MW-IR heating, which may have caused a greater extent of starch gelatinization. During gelatinization, starch molecules form a gel matrix that restricts water mobility by entrapping it within the structure [61, 62]. This entrapment reduces molecular mobility and results in shorter T_2 values for MW-IR samples. Thus, the differences in T_2 values can be linked to the extent of starch gelatinization.

4.4.3 | T_2 Times of Emulsified Powder Aquafaba Samples

In this study, the T_2 relaxation times of aquafaba emulsions were measured at two different time (Day 0 and Day 14) to understand how the mobility of water molecules within the emulsion changes over time (Table 4). T_2 relaxation times were used to analyze how freely water molecules can move within a system. Longer T_2 times mean that water molecules have more freedom to move, whereas shorter T_2 times mean that their movement is more restricted [25, 63]. In emulsions, this mobility is affected by how well the oil droplets are dispersed in the water phase. Once emulsification occurred, the T_2 times of the emulsions averaged somewhere between the shorter T_2 values of pure oil (1–50 ms) and the longer T_2 values of water (500–3000 ms) [63]. As emulsions age, they tend to become less stable, which can be observed through increase in T_2 times.

At day 0, the T_2 times varied depending on the preparation method used to create the aquafaba emulsions. MW-C-S allowed water molecules to move more freely, whereas PC-O restricted their movement more, likely due to how the oil droplets were distributed within the water phase. After 14 days, most of the emulsions showed an increase in their T_2 times. This suggests that as the emulsions aged, the water molecules gained more freedom to move, which is a sign of emulsion destabilization [63]. As the emulsions became less stable, the oil droplets began to come together (coalescence), and the two phases (oil and water) started to separate [64]. This phase separation reduces the interaction between oil droplets and water, allowing the water molecules to move more freely, hence the increased T_2 times [63].

TABLE 4 | T_2 relaxation time (ms) of aquafaba emulsions at Day 0 and Day 14.

Process	Day 0	Day 14
PC-S	$836.49 \pm 6.63^{c,B}$	$1184.90 \pm 53.03^{a,A}$
Mw-C-S	$999.23 \pm 5.72^{a,A}$	$965.91 \pm 20.64^{d,B}$
Mw-IR-S	$847.51 \pm 12.12^{c,B}$	$1035.73 \pm 14.21^{c,A}$
PC-O	$700.43 \pm 3.36^{d,B}$	$1048.03 \pm 19.83^{c,A}$
Mw-C-O	$881.33 \pm 7.40^{b,B}$	$1088.32 \pm 16.07^{bc,A}$
Mw-IR-O	$913.61 \pm 3.17^{b,B}$	$1123.33 \pm 42.76^{ab,A}$

Note: Values within the same column, indicated by different lowercase letters (a–d), are significantly different. Values within the same row, marked by different uppercase letters (A–B), show significant differences.
Abbreviations: MW-C = microwave, MW-IR = microwave-infrared, O = overnight processing, PC = conventional, S = non-soaked.

MW-C-S showed a decrease in T_2 time over the 14 days, indicating that this emulsion remained more stable over time. This means that the oil droplets in this emulsion were better able to stay dispersed in the water phase, preventing significant changes in water mobility and keeping the emulsion more intact.

4.4.3.1 | NNLS Analysis. The NNLS analysis showed the differences in water mobility and structural characteristics of the emulsions over time presented in Figure 2. The NNLS analysis of non-soaked samples (MW-C-S, MW-IR-S, and PC-S) revealed distinct stability trends over time. MW-C-S exhibited two peaks on both Day 0 and Day 14, with the peak at a lower T_2 time increasing in intensity on Day 14. This peak likely corresponds to protons associated with the oil phase, as oils typically exhibit shorter T_2 relaxation times due to restricted molecular mobility. The increase in this peak suggests a greater contribution of the oil phase over 14 days leading to a decrease in overall T_2 value. This trend shows that the emulsion remained relatively stable as water mobility was further restricted by the oil droplets [63]. MW-IR-S, in contrast, displayed a single peak on both Day 0 and Day 14, with the peak broadening slightly and shifting toward longer T_2 times by Day 14. This suggests minor structural changes within the emulsion, possibly due to slight rearrangements of water and oil droplets, resulting in a modest increase in water mobility while maintaining overall stability [65]. PC-S consistently showed a single, unchanged peak on both Day 0 and Day 14, yet the T_2 value increased over time. This may indicate subtle internal rearrangements within the emulsion matrix that allowed water molecules slightly more freedom to move, even though no significant destabilization was observed in the NNLS profiles.

The overnight samples (MW-C-O, MW-IR-O, and PC-O) displayed more significant changes in their NNLS profiles,

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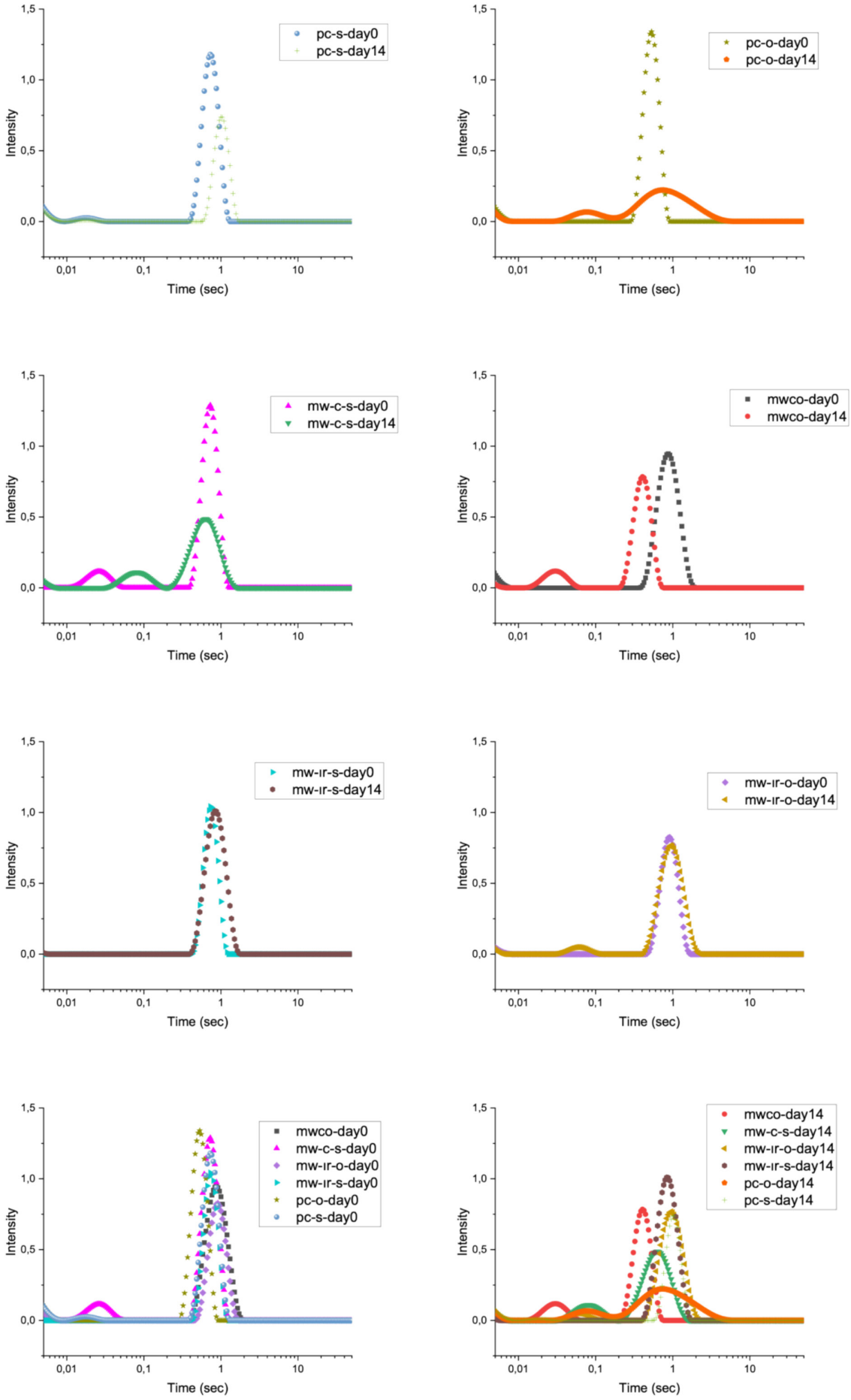


FIGURE 2 | Legend on next page.

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FIGURE 2 | NNLS analysis of T_2 proton pools in aquafaba emulsions on Day 0 and Day 14.

reflecting greater destabilization over time. MW-C-O started with a single peak on Day 0, indicating uniform water mobility, but developed two distinct peaks by Day 14. This shift suggests destabilization due to phase separation and coalescence, confirmed by the increase in T_2 value, which indicates increased water mobility [66]. MW-IR-O also began with a single peak on Day 0 and developed two peaks by Day 14, with the second peak representing oil contribution (lower value) and first peak appearing in a similar position as Day 0 but slightly shifted toward longer T_2 times [18, 63, 67]. This suggests moderate destabilization, as the emulsion structure broke down and allowed more freedom of water movement. PC-O exhibited the most dramatic change, starting with a sharp single peak on Day 0 and developing a very broad profile with two peaks by Day 14. This broadening reflects severe destabilization, where coalescence and phase separation led to distinct water environments and significantly increased T_2 values. These results indicate that overnight soaking generally results in emulsions with higher initial uniformity but greater destabilization over time.

4.4.4 | Hydration

TD-NMR relaxometry seems to be a powerful tool to estimate the hydration behaviour of powder food samples. Hydration is explained as the ability of a powder to absorb or bind water [58]. At very low moisture levels, water is tightly bound to nonpolar or ionic groups. As the amount of moisture increases, the water molecules become more disordered, leading to higher mobility and increased T_2 relaxation times [68]. Therefore, the hydration ability of any powder or food material may easily be detected by measuring the T_2 relaxation times. At the start of a hydration process ($t = 0$ min), when water is first added, T_2 values are initially high because water remains in a free, unbound state. As hydration progresses, water interacts with the powder matrix, leading to gradual binding and reduced mobility, which is reflected in a decrease in T_2 values (Figure 3). This trend continues throughout the 2.5-h observation period, during which T_2 relaxation times were recorded at 5-min intervals for the first hour and at 10-min intervals thereafter.

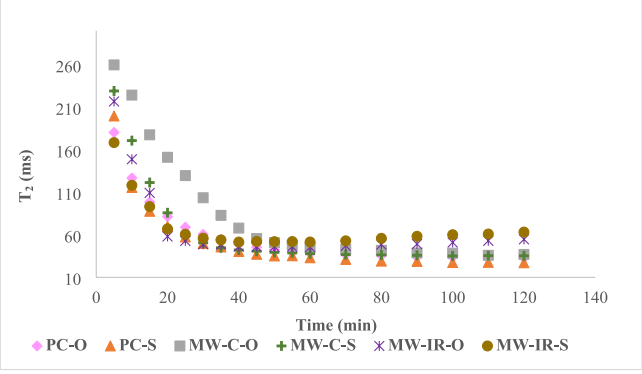


FIGURE 3 | Hydration behavior of powder sample over time.

Powder aquafaba samples showed different hydration behaviour as can be seen in Figure 3, indicating that different production techniques affected the properties of the powder. Hydration curve of these powders was fitted to a mono exponential decay equation, which is shown below:

$$y = a * \exp^{-b*x} + c$$

where "a" expresses the initial T_2 value and "b" is the decay rate and "c" corresponds to the equilibrium T_2 value, which indicates the minimum water mobility reached at the end of the hydration process.

Constant "a" refers to the initial value which corresponds to the initial T_2 value corresponding to the first measurement taken immediately after water was added to the powder aquafaba. Higher "a" values indicate the presence of more free water, which results in longer T_2 values. Another important parameter that describes hydration kinetic of aquafaba powder is mainly ***b*** the decay rate of the hydration process, indicating how quickly water is absorbed by the powder. The highest "b" value was observed for the non-soaked samples indicating faster hydration, whereas the lowest "b" value was observed in MW-C-O sample, which suggests slower water absorption (Table 5) [69]. Non-soaked samples generally exhibited higher hydration rates than overnight-soaked samples. MW-IR-S had the highest "b" value, followed by MW-C-S and PC-S, indicating faster water absorption in non-soaked powders. This trend suggests that non-soaked powders contained more accessible water-binding sites, likely due to a greater proportion of amorphous starch and less structural reorganization during processing. In contrast, overnight-soaked samples showed lower "b" values, indicating slower hydration kinetics. MW-C-O had the lowest "b" value, followed by PC-O, whereas MW-IR-O maintained a relatively high hydration rate. The slower hydration rates in overnight samples may be attributed to higher starch crystallinity, which limits water accessibility by reducing available hydrophilic sites. The variations in hydration behavior among samples may be influenced by differences in their protein composition and structure, which

TABLE 5 | Hydration curve constants of powder samples.

Process	a	b	c
PC-S	233.20 ± 16.40^{c}	0.074 ± 0.00^{b}	36.55 ± 2.47^{bc}
Mw-C-S	337.43 ± 10.47^{b}	0.092 ± 0.02^{ab}	34.71 ± 2.07^{c}
Mw-IR-S	306.67 ± 19.64^{b}	0.111 ± 0.01^{a}	49.33 ± 2.46^{a}
PC-O	199.39 ± 21.06^{c}	0.066 ± 0.01^{c}	34.87 ± 3.20^{c}
Mw-C-O	302.23 ± 5.81^{b}	0.050 ± 0.00^{d}	33.94 ± 3.05^{c}
Mw-IR-O	403.10 ± 20.62^{a}	0.110 ± 0.02^{a}	40.44 ± 2.85^{b}

Note: Values within the same column, indicated by different lowercase letters, are significantly different.
Abbreviations: MW-C = microwave, MW-IR = microwave-infrared, O = overnight processing, PC = conventional, S = non-soaked.

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are affected by the processing methods used. Previous studies have shown that thermal and mechanical treatments can lead to the denaturation, aggregation, or selective removal of certain protein fractions, altering the powder's ability to interact with water [69]. Additionally, starch gelatinization and crystallinity changes play a key role in water absorption capacity and hydration kinetics [70].

As can be seen from the hydration plots (Figure 3), different processing methods may have resulted in the removal of different protein fractions. After a short time (< 40 min), a sharp decrease in T_2 was observed for all samples, indicating that aquafaba powder has a strong tendency to absorb or bind water. After around 60 min, the hydration of powders seemed to be complete for all samples as no significant change was obtained for T_2 values.

The sharpest decrease was observed for the non-soaked samples, whereas the smoothest decrease was determined with MW-C-O sample when "b" constants were examined. This difference may arise from the variations in the composition of the powder aquafaba being different. As an example, globulin and albumin differ in solubility and albumins are known with higher water solubility than globulins, which could influence hydration behavior [2]. Substances that are highly water soluble generally tend to have stronger hydration forces, meaning that water molecules are expected to form a tighter water shell around these substances. On the other hand, substances that are less soluble in water generally tend to have weaker hydration forces, meaning that water molecules are expected to form a less tight water shell around these substances. In the PC-S samples, more hydrophilic constituents could have leached out, whereas substances that have relatively less tendency to interact with water might have leached out in the MW-C-O samples.

However, starch crystallinity and gelatinization also played a major role. Overnight soaking and MW-IR treatment likely caused greater starch gelatinization, disrupting the crystalline structure and altering the hydration properties of the powders. MW-C-O, which had the lowest hydration rate (b value), also exhibited signs of higher crystallinity. Crystalline starch structures limit water absorption by restricting the availability of hydrophilic sites, slowing down hydration. In contrast, non-soaked samples, which showed a faster hydration rate, likely contained more amorphous starch regions, allowing water to penetrate more easily.

5 | Conclusion

This study demonstrates the significant impact of different processing methods on the physical properties of aquafaba powder, particularly in terms of hydration and emulsification behaviors. Using TD-NMR, water mobility in powders obtained through conventional, microwave, and MW-IR processing was successfully characterized. The results indicate that MW-IR processing enhances both the dry matter content and hydration capacity of aquafaba, making it a functional ingredient. TD-NMR provided valuable insights into the relationship between processing methods and water dynamics, confirming its utility as a noninvasive and effective tool for analyzing food matrices. This research contributes to the growing body of knowledge on plant-based ingredients and highlights the potential of TD-NMR techniques in food science.

Author Contributions

Elif Gokcen Ates conceptualized and designed the study, conducted experiments, performed data analysis, and wrote the initial draft of the manuscript. Gokcem Tonyali Karsli assisted in the experimental design, and contributed to data analysis and wrote the initial draft of the manuscript. Ozcan Dilara Ozcan was responsible for conducting experiments and data collection. Emin Burcin Ozvural supervised the research, reviewed the manuscript, and provided feedback. Mecit H. Oztop, as the principal investigator, conceptualized the study, secured funding, supervised the entire project, and revised the manuscript.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

Data will be available on request.

Peer Review

The peer review history for this article is available at https://www.webofscience.com/api/gateway/wos/peer-review/10.1002/mrc.5521.

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