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Reliability of Multi-Method Mineralogical Identification in Expansive Soils

Sefa YILDIRIM*, Erdal COKCA

Middle East Technical University, Department of Civil Engineering, 06800 Ankara, Türkiye

Highlights

- Integrated XRD, SEM–EDX and methylene blue methods improve mineral identification.
- Cubuk clay contains smectitic-illitic minerals together with quartz and calcite.
- Reliable mineral identification requires combined analytical indicators.

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Abstract

The swelling characteristics and engineering performance of expansive soils are primarily governed by their clay mineralogy; however, mineralogical identification based on a single method may be insufficient for natural clays composed of multiple clay minerals together with non-clay constituents. This study aims to determine the mineralogy of a natural expansive clay from the Çubuk district of Ankara, Türkiye, using an integrated set of direct and indirect methods, and to demonstrate the limitations of relying on a single technique while proposing a practical combination of tests for more reliable mineralogical identification. Direct analyses included X-ray diffraction (XRD) and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX), while indirect indicators comprised methylene blue adsorption, Atterberg limits, clay fraction content (<2 μm) and activity. The results show that the Çubuk clay contains a significant smectitic (montmorillonitic) component together with non-clay minerals such as quartz and calcite. Methylene blue results indicate high cation exchange capacity and specific surface area, and SEM–EDX spectra support the presence of interlayer cations consistent with a smectite-dominant clay fraction exhibiting illite-smectite characteristics. The results highlight the advantage of a multi-method approach and provide a practical framework for more robust mineralogical characterization.

1. INTRODUCTION

Clay mineralogy exerts a primary control over the physical, chemical, and mechanical behavior of fine-grained soils, particularly in expansive clays, where swelling and shrinkage responses depend on their structure, composition, interlayer cations, and hydration mechanisms [1–4]. The amounts of smectite, illite, or mixed clay minerals govern cation exchange capacity (CEC), specific surface area (SSA), water adsorption, and volume-change potential. From an engineering perspective, inaccurate identification of clay mineral composition may lead to misestimation of swelling pressure, deformation potential, and long-term performance of foundations, pavements, and earth structures [5, 6]. Reliable mineralogical characterization is therefore essential not only for scientific classification but also for geotechnical risk assessment and design.

X-ray diffraction (XRD) is widely regarded as the reference method for mineral phase identification in soils and rocks [7–11]. By analyzing basal spacings and diffraction peaks, XRD enables differentiation among major clay groups such as smectite, illite, and kaolinite [7–10]. However, natural expansive soils commonly contain multiple clay minerals together with non-clay constituents such as quartz and calcite. In addition, mixed-layer phases, variable crystallinity, and peak overlap may complicate interpretation, particularly when bulk powder samples are analyzed without oriented clay preparations [7–11]. Under such conditions, reliance on XRD alone may lead to incomplete or uncertain mineralogical conclusions.

*Corresponding author, e-mail: tsefayildirim@gmail.com

For this reason, complementary analytical techniques are often used to support crystallographic observations [12]. Scanning electron microscopy (SEM) provides direct insight into particle morphology and fabric, revealing platy and flaky structures typical of 2:1 clay minerals. When combined with energy-dispersive X-ray spectroscopy (EDX), elemental composition can be assessed semi-quantitatively, offering additional evidence regarding layer type and interlayer cations [13–15]. For example, potassium is commonly associated with illitic structures, whereas sodium, calcium or magnesium may indicate smectitic interlayers [1, 13]. Although SEM–EDX does not replace XRD, it can provide valuable microstructural and compositional context that strengthens mineralogical interpretation.

Indirect indicators related to surface charge and physico-chemical behavior may also provide useful constraints. The methylene blue adsorption test allows estimation of CEC and SSA, parameters closely linked to clay mineral type and activity [16–18]. High CEC and SSA values are generally associated with smectite-rich clays [19], whereas lower values may suggest illitic dominance. Similarly, index properties such as liquid limit, plasticity index, activity, and clay fraction content (<2 μm) reflect the combined influence of mineralogy and particle size distribution and offer rapid screening-level insight into expansive behavior [1,15]. While these parameters do not directly identify mineral phases, they provide complementary evidence that can support or question conclusions derived from crystallographic analyses.

Although several mineralogical determination methods are available, XRD is the most preferred and frequently used primary method. This approach may be sufficient for simple, well-crystallized systems but is less reliable for natural expansive clays composed of multiple clay minerals together with non-clay constituents and potentially mixed-layer phases [1,8]. Individual techniques may therefore produce partially overlapping or ambiguous indications. Despite this complexity, structured guidance on integrating mineralogical, microstructural, and physico-chemical indicators into a coherent interpretative strategy remains limited in the geotechnical literature. As a result, mineralogical conclusions in engineering studies may vary depending on the selected method, highlighting the need for a systematic multi-method evaluation framework.

The expansive clay sampled from the Çubuk district of Ankara, Türkiye, represents a natural multi-mineral system typical of many engineering sites. Previous studies on Ankara clays have focused primarily on swelling potential and mechanical behavior, whereas integrated mineralogical characterization combining multiple complementary techniques has received comparatively less attention. The Çubuk clay exhibits high plasticity and notable swelling characteristics, suggesting the presence of smectitic components, while geological context indicates the possible coexistence of illitic phases and non-clay minerals.

The present study aims to determine the mineralogical composition of this expansive clay through an integrated evaluation of direct (XRD, SEM, EDX) and indirect (methylene blue adsorption, CEC/SSA estimation, and index properties) methods, to assess the limitations of single-technique identification, and to propose a practical, tiered framework for reliable mineralogical interpretation of expansive clays. By systematically combining complementary lines of evidence, the study demonstrates that multi-method integration enhances confidence in mineralogical determination and provides a reproducible strategy for engineering-oriented soil characterization.

2. MATERIAL AND METHODS

The clay investigated in this study was obtained from a representative expansive clay deposit in the Çubuk district of Ankara, Türkiye. Bulk samples were collected and for mineralogical analyses, the material was air-dried, gently disaggregated, and passed through a No. 40 sieve.

Index properties were determined to provide indirect indicators supporting mineralogical interpretation. Atterberg limits were measured in accordance with ASTM D4318, and particle size distribution was determined using ASTM D6913. These parameters were primarily used to evaluate plasticity and clay fraction content relevant to mineralogical assessment, rather than for detailed engineering characterization.

The methylene blue adsorption test was performed according to NF P 94-068 to estimate cation exchange capacity (CEC) and specific surface area (SSA). Soil suspensions were titrated with methylene blue solution until the endpoint was identified using the filter paper spot method. CEC and SSA values were calculated according to the procedures defined in the standard. These parameters were used as indirect indicators of clay mineral type and surface activity.

Mineralogical composition was determined using X-ray diffraction (XRD). Analyses were performed on a MiniFlex 300/600 diffractometer equipped with a D/teX Ultra2 detector. The X-ray generator operated at 40 kV and 15 mA under θ/2θ scan mode. Samples were oven-dried at low temperature and pulverized prior to testing. Diffraction patterns were recorded over a 2θ range of 5°–60°, with a scan speed of 5°/min and a step width of 0.02°. Mineral phases were identified based on characteristic peak positions and reference diffraction data.

Microstructural observations were performed using a QUANTA 400F Field Emission scanning electron microscope. Samples were mounted and coated with a conductive layer prior to imaging. Observations were conducted at magnifications ranging from 1000× to 25,000× to evaluate particle morphology and aggregation characteristics.

Elemental composition was analyzed using an energy-dispersive X-ray spectroscopy (EDX) detector coupled to the SEM system. Spot analyses were conducted at multiple locations to identify major and minor elements present in the clay. SEM observations and EDX spectra were interpreted together to support mineralogical identification.

Mineralogical interpretation was performed by integrating crystallographic evidence (XRD), microstructural observations (SEM), elemental composition (EDX), and surface-charge-related parameters (CEC and SSA from methylene blue adsorption), supported by plasticity characteristics. Conclusions were based on the convergence of multiple independent indicators rather than reliance on a single technique.

3. RESULTS

3.1. Index Properties and Surface Activity Indicators

The investigated clay exhibits a liquid limit (LL) of 76%, a plastic limit (PL) of 24%, and a plasticity index (PI) of 52%. Based on the Unified Soil Classification System (USCS), the soil is classified as a high-plasticity clay (CH). Particle size analysis indicates a clay fraction of 61%, and the activity is calculated according to Skempton [20] (PI/clay fraction) as 0.85. The soil activity result, between 0.75 and 1.25, is categorized as normally active clay [20] and is considered an illite-type mineral (≈ 0.9) [15].

Methylene blue adsorption testing yielded a cation exchange capacity (CEC) of 28.6 meq/100g and a specific surface area (SSA) of 224.2 m^2/g. These values are within ranges commonly reported for active clay systems. The index and adsorption parameters provide quantitative indicators of clay mineral reactivity and surface properties.

According to the classification proposed by Mitchell and Soga [1], soils with a cation exchange capacity (CEC) in the range of approximately 15–50 meq/100 g are generally associated with illitic clay minerals, while typical specific surface area (SSA) values are about 80–120 m^2/g for illite and 600–800 m^2/g for montmorillonite. In the present study, the measured CEC value of 28.6 meq/100 g falls within the illite-type range. However, the SSA value of 224.2 m^2/g is higher than that expected for pure illite and falls between typical values for illite and montmorillonite. This combination suggests that the clay fraction exhibits predominantly illitic characteristics with a possible contribution from smectitic components.

3.2. X-Ray Diffraction (XRD) Analysis

The XRD pattern of the investigated sample is presented in Figure 1. The diffraction data reveal basal reflections characteristic of clay minerals together with distinct peaks attributable to quartz and calcite,

indicating the coexistence of both clay and non-clay mineral phases within the sample. The smectitic reflections are relatively broad, consistent with expandable clay minerals and possible structural variability, whereas quartz and calcite peaks are sharp and well defined at their characteristic positions. No separate illite phase was clearly resolved under the applied measurement conditions. Overall, the diffraction pattern suggests a multi-mineral assemblage dominated by smectitic clay minerals, with non-clay constituents.

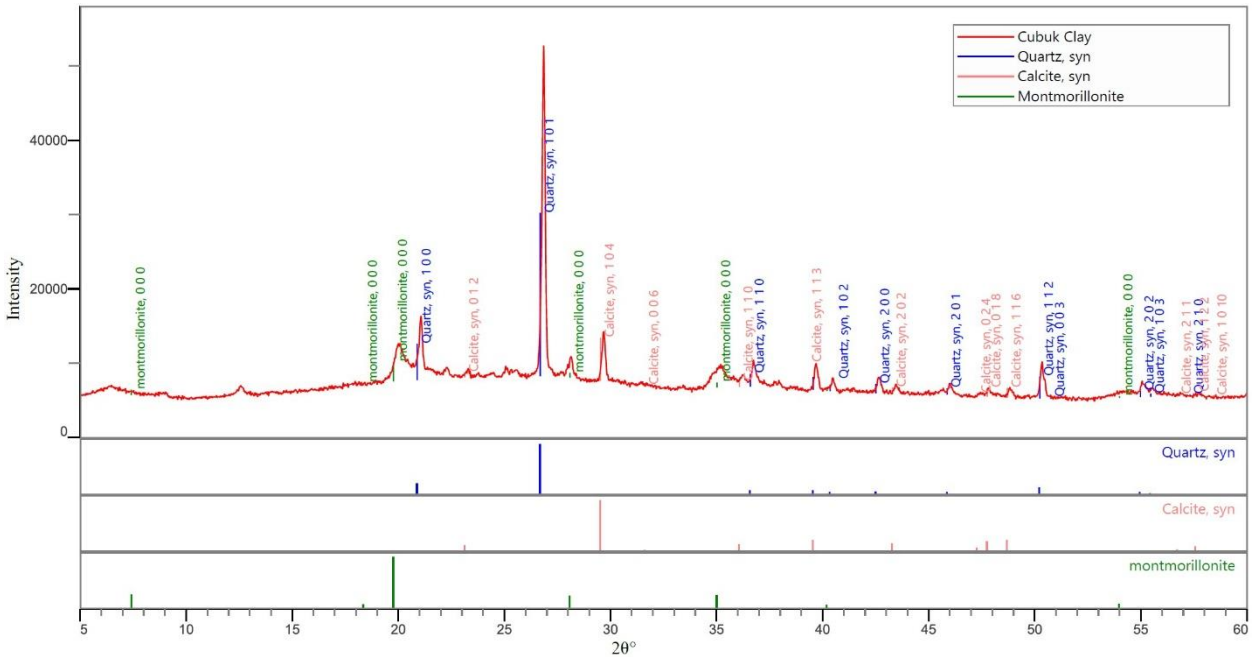


Figure 1. *X-ray diffraction (XRD) pattern of the investigated clay sample with indexed reflections of montmorillonite, quartz, and calcite [21]*

3.3. Scanning Electron Microscopy (SEM) Observations

Representative SEM micrographs of the investigated clay are presented in Figure 2(a–d). The images were obtained at different magnifications in order to capture both the overall microfabric and the detailed particle morphology.

Figure 2(a) shows a lower-magnification image (1000×, scale bar 100 µm) illustrating the general morphology of a clay aggregate. The specimen appears as a compact, irregularly shaped mass with a rough and heterogeneous surface texture. The surface is not smooth; instead, it exhibits fine-scale undulations and micro-roughness, indicating that the macro-aggregate is composed of much finer constituent particles.

Figures 2(b–d) present higher-magnification images (25,000×, scale bar 4 µm) focusing on different areas of the clay matrix. At this scale, the material is clearly composed of thin, plate-like and flake-shaped particles. These particles display irregular outlines and variable sizes, typically in the micrometer range. In Figure 2(b), stacked and partially overlapping platelets are visible, forming an interlocking arrangement. The particles appear to be oriented in multiple directions rather than forming a uniform parallel alignment.

Figure 2(c) shows a more densely packed region of the matrix, where numerous small plate-like particles adhere to larger surfaces. A distinct angular fragment is also visible in the lower part of the image, indicating the coexistence of particles with different shapes within the same microstructural domain.

Figure 2(d) highlights another compact zone characterized by closely packed platy particles forming a layered texture. The boundaries between individual flakes are discernible, and particle contacts predominantly occur face-to-face and edge-to-face, contributing to the aggregated fabric.

Overall, the SEM observations demonstrate that the investigated soil consists of aggregated clusters of thin, platy particles forming a heterogeneous microstructure. The multi-scale imaging confirms the presence of micro-aggregated clay domains with irregular particle stacking and interparticle contacts, providing morphological evidence consistent with a fine-grained clay matrix.

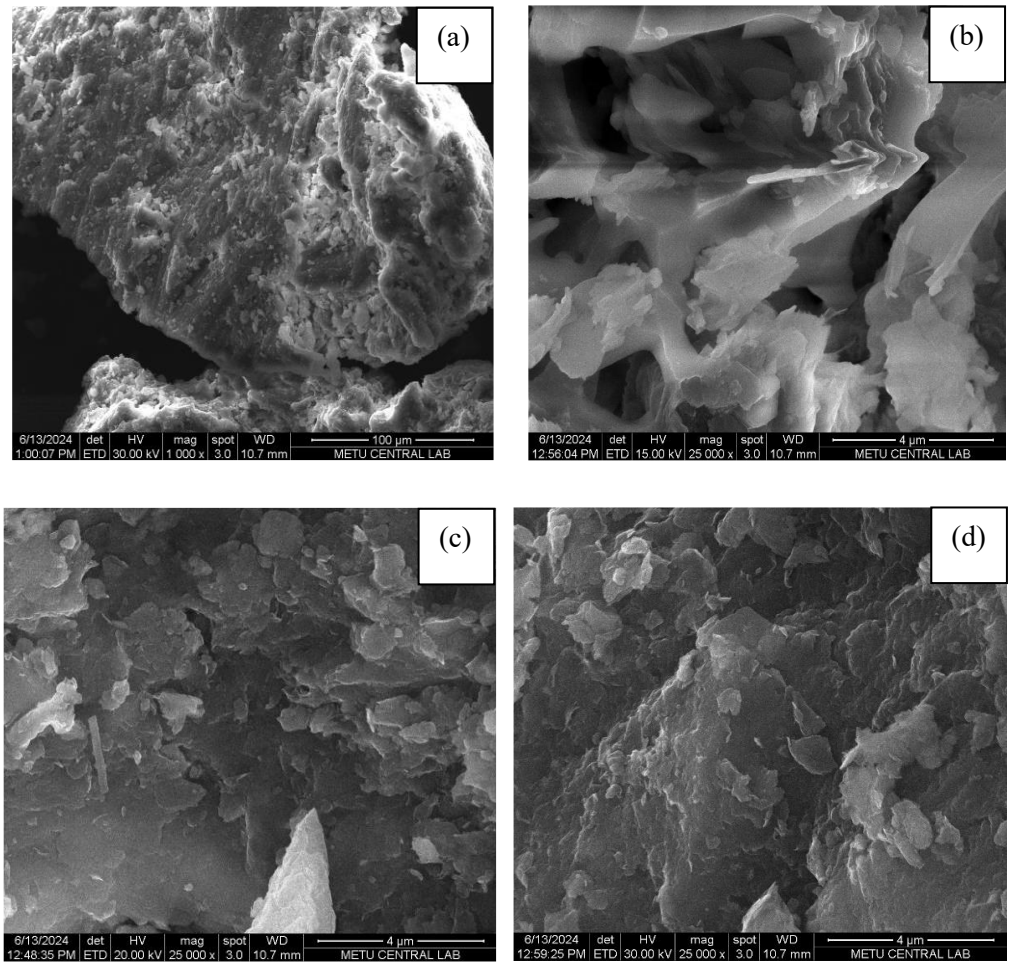


Figure 2. *Multi-scale SEM micrographs showing the microfabric and particle morphology of the investigated clay: (a) general aggregate structure (1000×); (b–d) detailed platy particle morphology (25,000×)*

3.4. Energy-Dispersive X-Ray Spectroscopy (EDX) Results

EDX spectra indicate that the clay matrix is dominated by O, Si, and Al, with recurring Mg, Ca, K, and Fe, and occasional Na. The EDX results obtained from six different analysis spots are presented in Table 1. In this table, the Mean (wt.%) values represent the arithmetic average weight percentage of each element calculated from the analyzed spots, while the Range (wt.%) values indicate the minimum and maximum measured concentrations, reflecting the compositional variability within the examined micro-areas.

Table 1. *Summary of EDX elemental composition (wt.%) of the investigated clay based on multiple micro-analytical spots*

Element	Mean (wt.%)	Range (wt.%)
O	47.94	45.05–53.10
Si	31.34	27.56–40.90
Al	10.72	5.47–12.22
K	2.26	1.88–2.51
Mg	1.52	0.36–1.88
Ca	1.94	0.17–2.67
Fe	5.04	3.99–5.84
Na	0.17	0.13–0.21

The repeated co-occurrence of (Si, Al, O) with Mg and Ca/Na is compatible with 2:1-type clay mineral chemistry, in which interlayer cations and octahedral substitutions are common. The persistent presence of K is consistent with illitic components or illite–smectite interstratification. Notably, one analyzed spot is Si-rich and Al-poor, suggesting the presence of a quartz grain within the clay matrix, in agreement with SEM observations and XRD results.

EDX should be regarded as a supportive mineralogical tool rather than a stand-alone method. Because it determines elemental composition within a limited micro-volume and does not directly resolve layer structure or basal spacing, its primary value lies in (i) confirming Si–Al-dominated clay domains, (ii) identifying interlayer cation tendencies (K versus Ca/Na/Mg), and (iii) detecting non-clay grains (e.g., silica-rich quartz) that help explain XRD peaks and microfabric observations.

3.5. Summary of Mineralogical Indicators

The results obtained from index properties, methylene blue adsorption, X-ray diffraction, SEM observations, and EDX analyses provide a set of complementary mineralogical indicators for the investigated clay. Each analytical method yields specific information about the sample's composition and characteristics.

The high liquid limit and plasticity index indicate a highly plastic clay with a significant clay fraction. The calculated activity value indicates moderate to high clay-mineral reactivity. Methylene blue adsorption results indicate elevated CEC and specific surface area, suggesting a clay fraction with considerable surface activity.

XRD analysis identifies the presence of smectitic and illitic clay minerals together with non-clay constituents such as quartz and calcite. SEM observations reveal platy and lamellar particle morphologies consistent with clay-sized particles forming an aggregated microstructure. EDX spectra confirm the presence of major aluminosilicate elements (Si, Al, O) and minor elements including K, Ca, Mg, and Fe. The main mineralogical indicators obtained from different analytical methods are summarized in Table 2.

Table 2. *Integrated summary of mineralogical indicators derived from different analytical methods*

Method	Parameter/Observation	Value/Finding	Mineralogical Indication
Atterberg limits	LL,PL,PI	LL=76%, PI=52%	High Plasticity clay (CH)
Clay fraction	<2 µm	61%	Clay- rich soil
Activity	PI / Clay fraction	0.85	Normally active clay [20] illite-type mineral (≈ 0.9) [15]
Methylene Blue	CEC	28.6 meq/100g	Illite-type
	SSA	224.2 m^2/g	Illite and montmorillonite type
XRD	Diffraction peaks	Smectite + Illite; quartz, calcite	Mixed clay mineral assemblage
SEM	Particle morphology	Platy, lamellar	2:1 Clay morphology
EDX	Elemental composition	Si, Al, O dominant; K, Ca, Mg present	Illitic-Smectitic Composition

4. DISCUSSION

4.1. Indirect Indicators and Their Mineralogical Implications

The plasticity characteristics (LL = 76%, PI = 52%) and clay fraction (61%) indicate a highly plastic, clay-rich soil. The calculated activity value (A = 0.85) falls within ranges commonly reported for illitic to mixed illite–smectite systems rather than strongly smectite-dominant clays, which typically exhibit higher activity values. This highlights a known limitation of plasticity-based indicators: while they are sensitive to mineralogy, they are not uniquely diagnostic because they are also influenced by gradation, particle aggregation, and pore-fluid chemistry.

The measured CEC (28.6 meq/100g) and SSA (224.2 m^2/g) fall within ranges commonly associated with clays containing expandable components. However, overlapping ranges between illitic and mixed-layer clays are widely reported in the literature. Therefore, although adsorption-based parameters indicate a clay fraction with notable surface activity, they cannot independently resolve mineral proportions. This reinforces the need to interpret indirect indicators alongside crystallographic and microstructural evidence.

4.2. Interpretation of Direct Indicators

XRD analysis confirms the presence of smectitic and illitic clay minerals together with quartz and calcite. The smectitic basal reflection occurs near typical d-spacing values associated with expandable 2:1 clays, while illite peaks are also identifiable. However, interpretation of bulk XRD patterns in natural soils can be affected by peak overlap, variable crystallinity, and the presence of mixed-layer structures. In particular, interstratified illite–smectite phases may produce broadened or intermediate reflections, complicating phase identification.

Without oriented mounts and glycolation/heating treatments, XRD results may indicate the presence of smectite but may not reliably distinguish between smectite-dominant and mixed-layer systems. In engineering practice, this distinction is important because swelling behavior and surface reactivity depend strongly on the proportion and nature of expandable layers. Thus, while XRD provides primary mineral identification, its interpretative resolution may be limited when applied as a single method to heterogeneous natural clays.

SEM observations provide microstructural context (particle shape, aggregation, fabric) that complements XRD. The platy/lamellar morphology observed here is consistent with 2:1 clay minerals and supports the

presence of layered silicates indicated by XRD. While SEM cannot uniquely identify mineral species, it helps distinguish clay-rich fabrics from silt-dominated matrices and reveals aggregation that can influence both plasticity and adsorption results.

EDX further supports interpretation through elemental signatures. The dominance of Si–Al–O is consistent with aluminosilicates, while the presence of K is commonly associated with illitic components and Na, Ca and Mg may reflect exchangeable interlayer cations and/or carbonate association (consistent with calcite identified by XRD) [14,15]. Importantly, EDX is semi-quantitative and spot-based; therefore, it is most defensible when used to corroborate XRD rather than to serve as a standalone mineral identification method.

4.3. Integrated Mineralogical Interpretation

Taken together, the dataset supports that the investigated Çubuk clay is a mixed-mineral expansive clay. XRD confirms smectitic and illitic phases with quartz and calcite, SEM shows clay-dominated platy morphologies, and EDX indicates elemental constituents consistent with layered aluminosilicates with K-bearing and Ca/Mg-bearing signatures. Indirect indicators (CEC/SSA and plasticity-based metrics) are consistent with an active clay fraction containing an expandable component. The key implication is that mineralogical confidence increases when independent "signatures" converge: crystallography (XRD), morphology (SEM), chemistry (EDX), and surface activity (methylene blue-derived CEC/SSA). This convergence is particularly valuable for natural soils where mixed mineral assemblages are expected and where single-method interpretations can be sensitive to sample preparation and measurement conditions.

4.4. Practical Implications and Recommended Test Combinations

From an engineering perspective, the objective is often not only to name minerals but to reliably anticipate swelling-related behavior and to classify risk. On that basis, a practical, tiered combination of tests is recommended:

Tier 1—Rapid screening: Atterberg limits + clay fraction + activity, complemented by methylene blue (CEC/SSA). This tier provides fast, cost-effective constraints on whether an expansive (smectitic) component is likely.

Tier 2—Definitive identification: XRD for mineral phases (including non-clay minerals). Where possible, oriented mounts and glycolation/heating treatments should be used to improve resolution of smectite and mixed-layer behavior.

Tier 3—Validation and context: SEM–EDX to document clay fabric and corroborate chemical signatures (e.g., K-bearing and Ca/Mg-bearing indications), supporting interpretation in mixed systems.

This approach aligns the diagnostic power of each method with its limitations, thereby improving the reproducibility of mineralogical conclusions for natural expansive clays.

4.5. Future Research Directions

Future work can strengthen mineral identification and reduce uncertainty by: (i) performing oriented-clay XRD with glycolation/heating to better resolve expandable and mixed-layer phases; (ii) quantifying mineral proportions using Rietveld refinement or comparable quantitative methods; (iii) integrating additional physico-chemical tests such as exchangeable cation analysis or FTIR/TGA to cross-check clay type; and (iv) examining how pore fluid chemistry (salinity, valence) modifies both methylene blue response and swelling behavior. Such additions would further improve the robustness of multi-method mineralogical characterization for engineering applications.

The combined evidence indicates that the investigated clay is a mixed-mineral expansive clay containing both smectitic and illitic components together with non-clay minerals. The results highlight that reliance

on a single identification method may be insufficient for natural clays with heterogeneous mineral composition. An integrated evaluation using complementary techniques provides a more reliable basis for mineralogical interpretation in engineering practice.

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DECLARATION OF ARTIFICIAL INTELLIGENCE (AI) USE

Generative AI agents or large language models (LLMs) were used only to improve clarity and quality of the English language presentation of the manuscript.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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