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Effect of Extreme Heating on the Mineralogy and Microstructure of Expansive Soil

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Abstract - Expansive soils pose significant challenges in geotechnical engineering due to their pronounced volume changes in response to moisture fluctuations, leading to substantial economic losses annually. This study investigates the effects of extreme thermal treatment on the mineralogical and microstructural properties of expansive soils. Soil samples collected from Al Ghat, Saudi Arabia, were subjected to a controlled heating regime of 600°C for two hours. Advanced analytical techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), particle size distribution (PSD) analysis, and energy-dispersive X-ray spectroscopy (EDS), were employed to evaluate changes in mineralogical composition, microstructure, and elemental composition. Results revealed significant alterations in the soil's properties, including particle aggregation, reduced fine particle content, and the formation of larger, more stable aggregates. XRD analysis indicated the disappearance of kaolinite peaks, suggesting dehydroxylation and the formation of metakaolin, while EDS analysis showed a reduction in oxygen content and enrichment of certain cations. These findings align with previous research, demonstrating that thermal treatment effectively reduces the expansive characteristics of soils by altering their mineralogical and microstructural properties. The study underscores the potential of thermal stabilization as a viable method for mitigating the adverse effects of expansive soils in geotechnical engineering applications.

Keywords**:** Expansive Soils; Thermal Stabilization; Mineralogical Transformation; Microstructural Analysis; Soil Swelling Potential

1. Introduction

Expansive soils present considerable challenges in geotechnical engineering due to their pronounced volume instability in response to changes in moisture content. Characterized by significant swelling and shrinkage cycles, these soils are especially problematic in arid and semi-arid regions, where seasonal variations in moisture levels are particularly pronounced. In the United States, the economic impact of damage caused by expansive soils is substantial, with annual costs estimated to range between $7 billion and $13 billion [1], [2]. Remarkably, this financial burden surpasses the cumulative damage attributed to all major natural disasters combined, underscoring the critical need for effective mitigation strategies.

Heat treatment has gained recognition as a viable method for stabilizing expansive soils, offering a potential solution to mitigate their undesirable volume change behavior. This technique entails subjecting soil to controlled elevated temperatures, which induces modifications in both its physical and chemical properties, thereby enhancing its stability. The underlying mechanism of thermal stabilization lies in the alteration of the soil's mineralogical structure, particularly targeting the behavior of swelling clay minerals such as smectite. Empirical studies have demonstrated that temperature plays a critical role in influencing soil consolidation behavior, with notable variations observed in compression indices as a function of thermal conditions [3]. These findings underscore the significance of temperature as a key parameter in the thermal stabilization process.

Recent research has highlighted the efficacy of thermal stabilization techniques in addressing the challenges posed by expansive soils. Experimental studies have consistently reported substantial enhancements in soil stability and a notable reduction in swelling potential after subjecting soils to controlled thermal treatment [4], [5], [6]. These advancements have spurred the adoption of thermal stabilization in diverse applications, including ground improvement, soil remediation, and infrastructure stabilization projects [7], [8], [9]. The growing body of evidence underscores the potential of thermal methods as a reliable and effective approach for mitigating the adverse effects of expansive soils in geotechnical engineering practices.

This study explores the effects of extreme thermal treatment on expansive soils, with a focus on changes in mineralogical composition and microstructural properties. Utilizing advanced analytical techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), particle size distribution (PSD) analysis, and energy-dispersive X-ray spectroscopy (EDS), the research examines the transformation of soil samples subjected to a controlled heating regime of

600°C for two hours. A detailed comparison between untreated and thermally treated soils is presented, highlighting key alterations in swelling potential, mineralogical structure, and microstructural characteristics. The findings contribute to the growing understanding of thermal stabilization mechanisms and their efficacy in addressing the challenges posed by expansive soils.

2. Materials and Methods

2.1. Soil Sample Collection and Preparation

The soil samples utilized in this study were collected from a site located near Al Ghat, a town approximately 250 kilometers northwest of Riyadh, Saudi Arabia. Laboratory testing conducted on the samples confirmed that the soil exhibits high plasticity characteristics, classifying it as CH according to the Unified Soil Classification System (USCS). Further classification using the AASHTO system placed the material within the A-7-6 category, indicative of its high clay content. Detailed engineering properties and classifications of the soil are summarized in Table 1. XRD analysis, illustrated in Fig. 5, identified kaolinite as the predominant clay mineral, with minor traces of illite. Notably, the absence of montmorillonite minerals in the soil composition was confirmed.

Table 1: Geotechnical properties and classifications of Al-Ghatt soil.

Parameter	**Value**
Specific gravity	2.8
Natural water content (%)	22
Dry unit weight (kN/m^3)	18
Consistency limits (%)	
Liquid limit	54
Plastic limit	27
Plasticity index	27
Shrinkage limit	16
Classification	
Unified soil classification system*	CH
AASHTO classification system	A-7-6

*CH refers to clay with high plasticity.

2.2. Thermal Treatment Procedure

The thermal treatment of clay specimens was performed using a Matest-manufactured electric laboratory furnace. This equipment features digital temperature controls for accurate thermal management and can achieve temperatures up to roughly 1000°C, making it ideal for high-heat applications. The device incorporates an even heat distribution mechanism to ensure uniform sample exposure, while its chamber, constructed with temperature-resistant components, maintains consistent thermal conditions throughout extended heating periods.

The experimental thermal treatment protocol involved subjecting the expansive clay specimens to a carefully controlled heating process. The samples were placed in the furnace and gradually heated until reaching a temperature of 600 °C, which was then maintained for a duration of 2 hours under constant monitoring. This specific temperature and duration were selected based on previous research indicating optimal conditions for achieving the desired mineralogical transformations [5]. Visual documentation of the specimens, presented in Fig. 1, illustrates the notable physical changes that occurred, comparing the original untreated clay material with the thermally modified samples. The post-heating specimens exhibited distinct alterations in both color and texture, providing initial evidence of the thermal treatment's effects on the clay's structural properties.

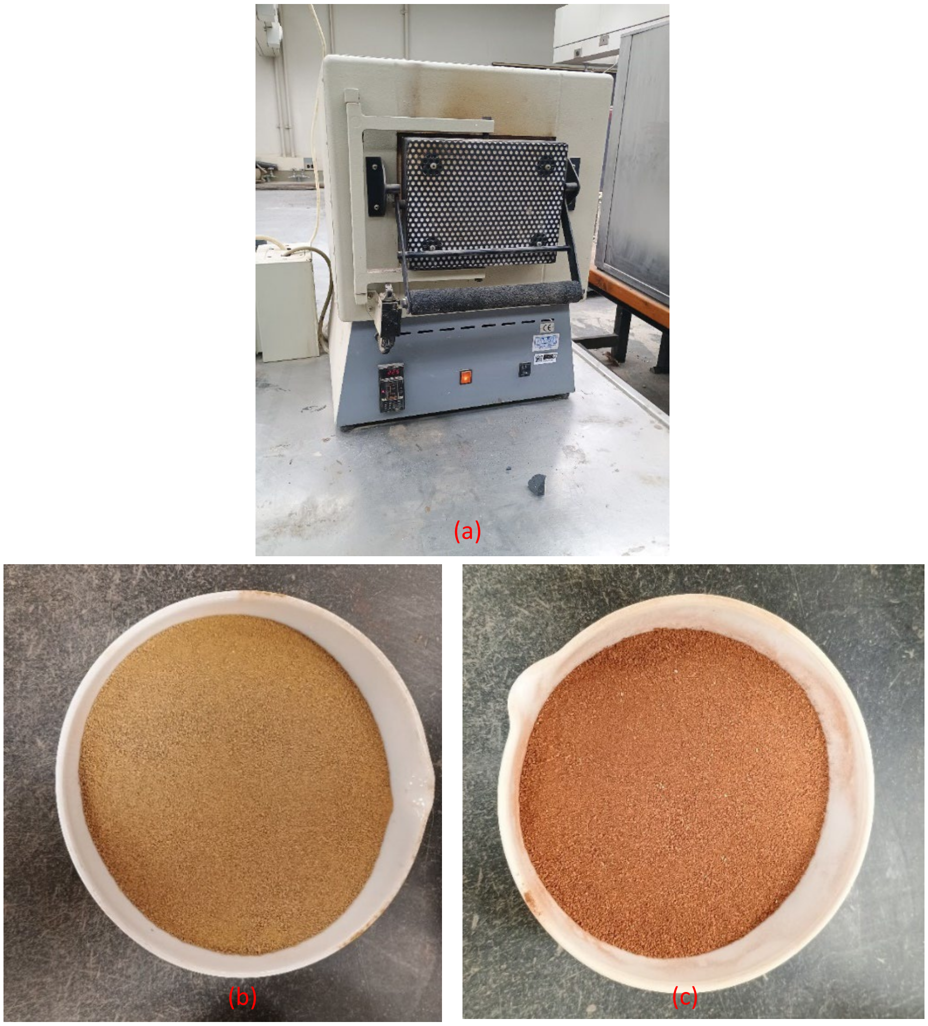


Fig. 1: Procedure of Thermal treatment of expansive soil sample: (a) Electrical furnace; (b) Untreated expansive soil; (c) Thermally treated clay sample.

2.3. Experimental Techniques

To investigate the effects of extreme heating on expansive soil, a series of advanced experimental techniques were employed to analyze both untreated and heated soil samples (heated to 600°C for 2 hours). XRD analysis was conducted using the Empyrean system by Panalytical to identify the mineralogical composition of the soil. PSA was performed using the LA-950 instrument by HORIBA to measure the particle size distribution of the soil samples. Additionally, SEM and EDS analyses were carried out using the TESCAN Vega system to examine the microstructural changes and elemental composition of the soil. These techniques provided a comprehensive understanding of the physical, mineralogical, and microstructural properties of the soil before and after thermal treatment.

3. Results and Discussion

3.1. Effect of extreme heating on Particle Size Distribution (PSD)

Fig. 2 shows the PSD for untreated and heat-treated expansive soil samples. The PSD curves demonstrate substantial differences between the untreated and heat-treated samples. The untreated clay shows a broader distribution curve with particle sizes ranging from 0.2 µm to approximately 200 µm, with 50% passing occurring at approximately 10 µm. In contrast, the heat-treated soil (600°C) exhibits a significant shift toward larger particle sizes, with the majority of particles distributed between 4 µm and 2000 µm, and 50% passing occurring at approximately 200 µm. This rightward shift in the PSD curve indicates particle aggregation and the formation of larger clusters due to thermal treatment. The untreated soil contains higher percentage of fine particles (<10 µm), while the heat-treated sample shows a marked reduction in fine particles an increase in coarser fractions. This transformation in particle size distribution suggests that thermal treatment promotes particle agglomeration and potentially reduces the soil's expansive characteristics by altering its fine particle content.

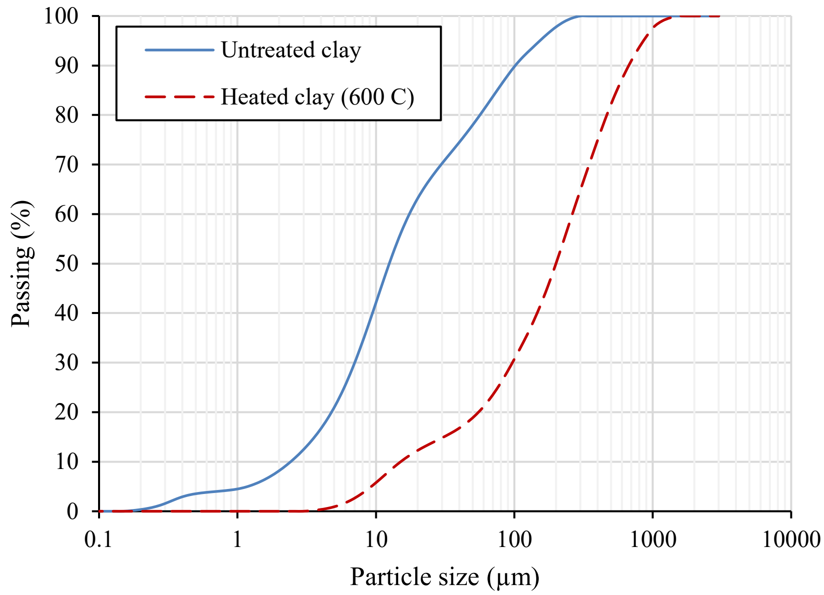
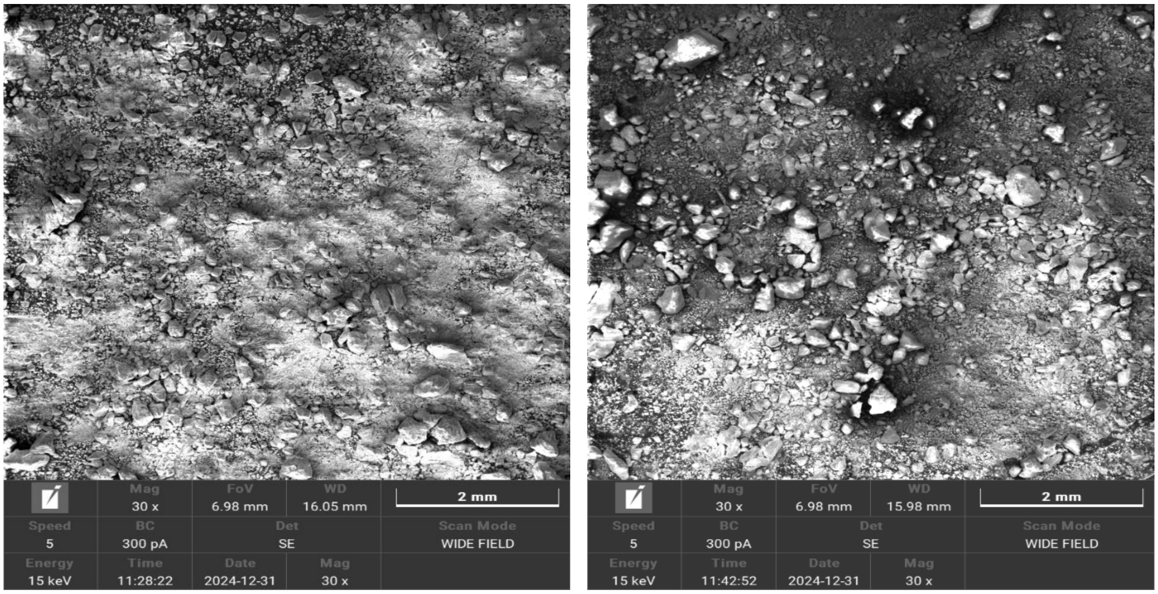


Fig. 2: Particle size distribution (PSD) for untreated and heat-treated expansive soil samples

3.2. Effect of extreme heating on SEM and EDS analysis

Fig. 3 illustrates SEM images of expansive clay samples at 30x magnification. The SEM images, captured at 30x magnification, reveal significant morphological differences between the untreated and heat-treated expansive soil samples. The untreated soil (**Error! Reference source not found.**a) exhibits a relatively dense and flocculated microstructure with smaller aggregates and less distinct particle boundaries. After heat treatment at 600°C (**Error! Reference source not found.**b), the microstructure shows notable modifications, characterized by larger, more clearly defined particles and increased void spaces between aggregates. This structural transformation can be attributed to the thermal modification of clay minerals, which likely resulted in the breakdown of the original clay structure and the formation of larger, more stable aggregates. The SEM images were obtained under identical operating conditions (15 keV beam energy, 300 pA beam current) to ensure comparative analysis.

Fig. 4 and Table 2 show EDS analyses for expansive soil samples before and after treatment. The EDS analysis of untreated and heated expansive soil at 600°C reveals notable changes in the elemental composition due to thermal treatment. In the untreated soil, the predominant elements are oxygen (69.59 atomic %, 53.63 weight %), silicon (13.01 atomic %, 17.60 weight %), and aluminum (7.67 atomic %, 9.966 weight %), which are characteristic of clay minerals. After heating to 600°C, the atomic and weight percentages of oxygen decreased to 65.97% and 49.66%, respectively, indicating the loss of hydroxyl groups and water molecules during dehydroxylation. The percentages of aluminum and silicon remained relatively stable, with slight increases to 7.91% and 13.23% (atomic %), respectively, suggesting minimal structural changes in the aluminosilicate framework. However, significant increases were observed in calcium (3.70 atomic %, 6.97 weight %), sulphur (3.04 atomic %, 4.59 weight %), and magnesium (1.35 atomic %, 1.55 weight %), likely due to the decomposition of organic matter and the formation of new mineral phases. These changes highlight the impact of thermal treatment on the soil's elemental composition, particularly the reduction in oxygen content and the enrichment of certain cations, which contribute to the altered physical and chemical properties of the heated soil.

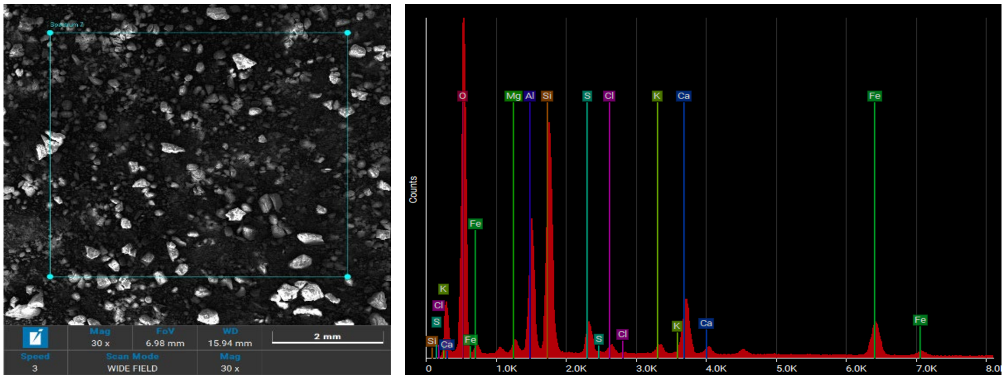


(a) (b)

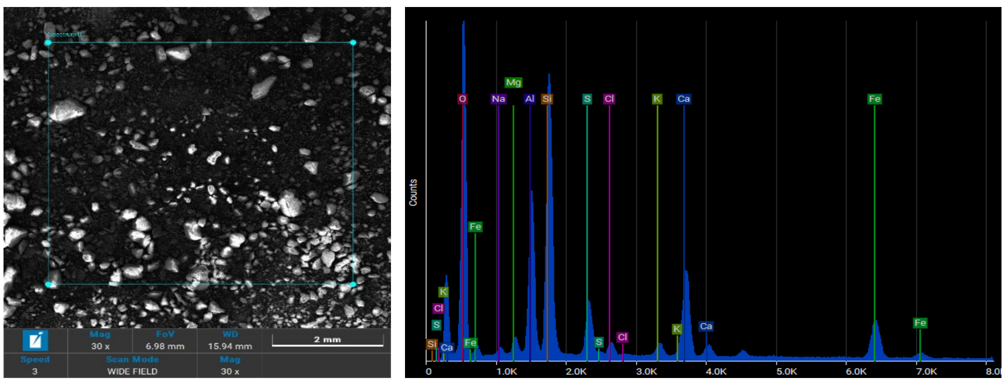
Fig. 3: SEM analysis of expansive clay samples at 30x magnification: (a) Untreated expansive soil; (b) Heated expansive soil (600 °C).

Table 2: EDS Analyses of untreated expansive soil and heated soil at 600 °C.

Element	Untreated clay		Heated clay (600°C)	
	Atomic %	**Weight %**	**Atomic %**	**Weight %**
Aluminum	7.67	9.966	7.91%	10.04%
Calcium	2.75	5.31	3.70%	6.97%
Chlorine	0.60	1.03	0.74%	1.23%
Iron	2.49	6.70	2.33%	6.13%
Magnesium	1.20	1.41	1.35%	1.55%
Oxygen	69.59	53.63	65.97%	49.66%
Potassium	0.59	1.12	0.64%	1.17%
Silicon	13.01	17.60	13.23%	17.48%
Sulphur	2.10	3.24	3.04%	4.59%



(a) Untreated clay soil



(b) Heated clay soil (600°C)

Fig. 4: EDS analyses for clay soil: (a) Untreated expansive soil; (b) Heated expansive soil (600 °C).

3.3. Effect of extreme heating on XRD Analysis

Fig. 5 show the XRD analyses for untreated and heated soil under 600 °C for 2 h. XRD analysis revealed significant structural transformations in the expansive clay soil after heating to 600°C. Notably, the peaks in the range of 10-12°, which are characteristic of illite, sepiolite, and kaolinite, were absent in the heated sample, indicating structural alterations of these clay minerals. In contrast, the quartz peak at 26.7° remained stable, consistent with its known thermal stability at 600°C. Similarly, the calcite peak at 29.5° persisted, suggesting its stability under the same heating conditions. Overall, there was a notable reduction in peak intensity and the number of peaks, suggesting significant structural changes within the clay matrix. The disappearance of kaolinite peaks is indicative of dehydroxylation and transformation into metakaolin. These observations are consistent with the expected behavior of clay minerals subjected to dehydration and dehydroxylation processes at elevated temperatures.

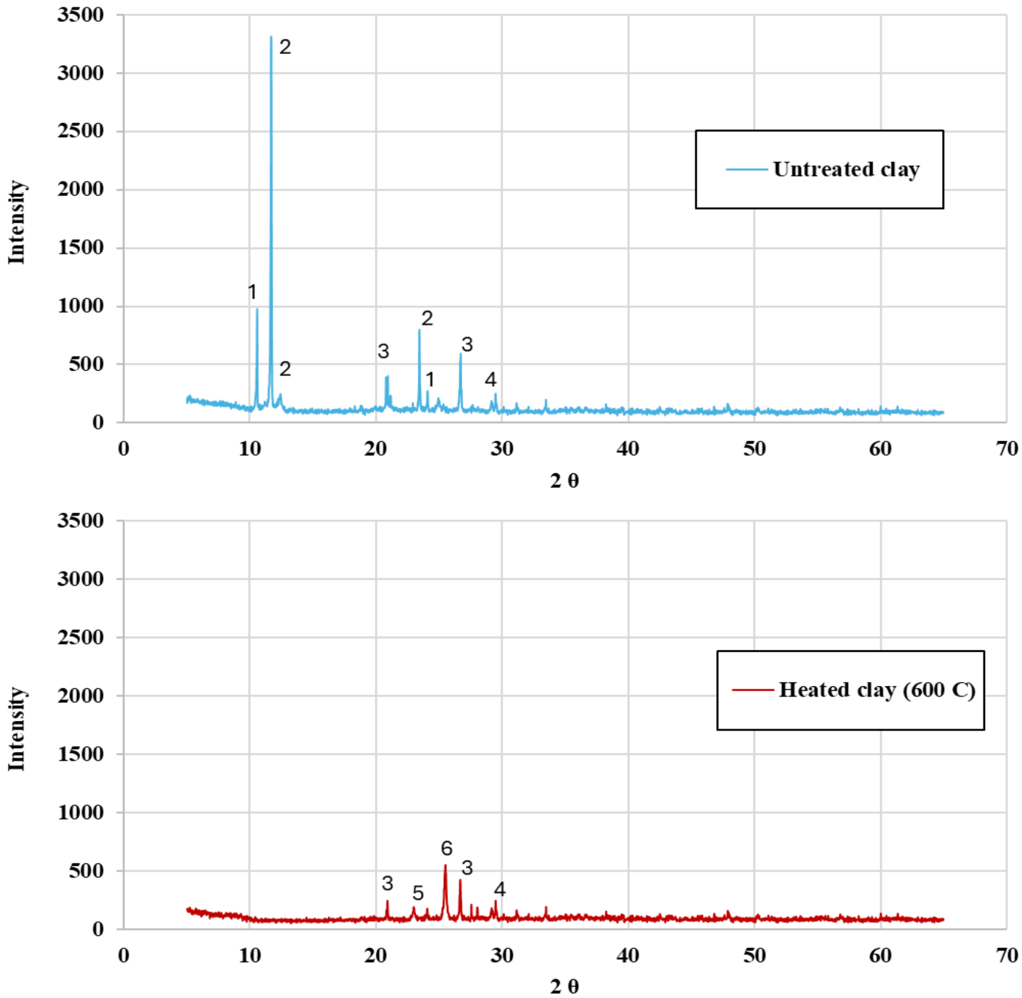


Fig. 5 XRD analysis for untreated clay soil and heated soil under 600 °C for 2 h (1=illite; 2=kaolinite; 3=Quartz; 4=calcite; 5=Dehydrated clay phases; 6=Transformed kaolinite).

3.4. Integration with Previous Research Findings

The findings of this study align well with previous research on the effects of thermal treatment on expansive soils. Wang et al. [5] demonstrated that heating expansive soils at specific thresholds, such as 600°C for bentonite, effectively converts them into non-expansive materials by altering their clay mineral structure. This is consistent with the current study, where heating at 600°C led to significant changes in mineralogical composition, as evidenced by the disappearance of kaolinite peaks in the XRD analysis and the formation of metakaolin. Similarly, the reduction in oxygen content observed in the EDS analysis supports the dehydroxylation process, which is a key mechanism in reducing the soil's expansiveness. Li et al. [10] observed rapid increases in mean particle diameter and density at lower temperatures, which parallels the current study's findings of particle aggregation and increased particle size at 600°C, as shown in the PSD analysis.

Kabubo et al. [11] and Yao et al. [6] further support the effectiveness of thermal treatment in mitigating expansive soil behavior. Kabubo et al. [11] reported improvements in strength characteristics and reductions in plasticity and swelling potential after heating black cotton soil to 600°C, consistent with the current study's observations of reduced fine particle content and increased particle size. Yao et al. [6] noted similar transitions from fine to coarse particles using microwave heating, aligning with the PSD results of this study. Overall, the results of this study corroborate previous findings while providing new insights into the specific changes in elemental composition, mineralogical phases, and particle size distribution induced by thermal treatment at 600°C.

4. Conclusion

This study has demonstrated that extreme thermal treatment at 600°C for two hours induces significant changes in the mineralogical and microstructural properties of expansive soils, effectively reducing their swelling potential. The application of advanced analytical techniques, including XRD, SEM, PSD, and EDS, provided comprehensive insights into the mechanisms underlying these transformations. Key findings include the aggregation of soil particles, reduction in fine particle content, and the formation of larger, more stable aggregates, as evidenced by PSD and SEM analyses. XRD results confirmed the dehydroxylation of kaolinite and its transformation into metakaolin, while EDS analysis highlighted a reduction in oxygen content and enrichment of cations such as calcium, sulphur, and magnesium. These changes collectively contribute to the stabilization of expansive soils, reducing their susceptibility to volume changes. The results align with previous research, reinforcing the efficacy of thermal treatment as a reliable method for mitigating the challenges posed by expansive soils. This study not only advances the understanding of thermal stabilization mechanisms but also provides practical insights for its application in geotechnical engineering, particularly in ground improvement and infrastructure stabilization projects. Future research could explore the long-term performance and field applicability of thermally treated expansive soils to further validate this approach.

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