Use of Iowa Eggshell Waste as Bio-Cement Materials in Pavement and Gravel Road Geo-Material Stabilization

Final Report
September 2024

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Institute for Transportation

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16. Abstract

This study investigated the utilization of eggshell powder (ESP) as a bio-based cementitious material for soil stabilization in Iowa, a state recognized as the leading egg producer in the United States. The prominence of egg production in Iowa results in substantial eggshell waste, a byproduct that, despite its high calcium carbonate content, remains largely underutilized. Eggshells, constituted of nearly 95% calcium carbonate, present an environmentally friendly opportunity to repurpose agricultural waste into beneficial construction materials. Addressing the dual challenges of waste management and soil stabilization, this study explored the potential of ESP derived from ground eggshells to enhance the properties of subgrade soils. Through a series of treatments—no treatment, oven drying, and calcination—the study assessed the effectiveness of ESP in improving soil engineering properties. Laboratory experiments with two types of Iowa soils mixed with up to 12% ESP demonstrated that calcination significantly optimized ESP's performance, particularly when the soil was combined with 3% additional water and cured at 40°C. The optimal addition rate for maximizing unconfined compressive strength (UCS) was identified as 6% to 8% ESP, which provided over a tenfold increase in UCS compared to soil without ESP. This enhancement was especially notable in loess soils, which exhibited marked strength improvements. Field simulations using the dynamic cone penetration (DCP) test further validated the laboratory results, indicating that the inclusion of calcined ESP substantially enhanced the tested soil's California bearing ratio (CBR). The findings underscore ESP's potential as an innovative and sustainable additive for soil stabilization, offering a practical solution to managing eggshell waste while contributing to the development of eco-friendly construction practices in Iowa. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses revealed that adding calcined ESP (1,830°F or 1,000°C) to soils resulted in a denser and more solid soil structure due to the formation of hydration products. This research not only addresses environmental concerns associated with eggshell waste but also highlights the broader application of agricultural byproducts in civil engineering, promoting sustainability in construction materials.

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EXECUTIVE SUMMARY

Soil stabilization is a critical process in construction and civil engineering, aiming to enhance the physical properties of soil to meet specific engineering requirements. Traditional methods of soil stabilization involve the use of materials such as lime and cement that, while effective, pose environmental concerns. The exploration of sustainable alternatives has led researchers to investigate the use of eggshell powder (ESP) as a bio-based cementitious material. Iowa, with its significant egg production, generates substantial quantities of eggshell waste, traditionally disposed of in landfills or used in limited agricultural applications. This study focused on the potential for utilizing this waste as a soil stabilizer to improve the engineering properties of local Iowa soils while contributing to sustainable construction practices and waste management.

The study involved a comprehensive laboratory testing program to evaluate the effects of ESP on the engineering properties of two types of Iowa soils (Soil A and Soil B) categorized based on their composition and grain size distribution. Three processing methods for ESP were investigated—air drying, oven drying, and calcination—to determine their impact on soil stabilization. The research methods included Atterberg limit tests and standard Proctor compaction tests to assess soil consistency and compaction characteristics, as well as tests to determine the unconfined compressive strength (UCS) and California bearing ratio (CBR) of soil samples mixed with up to 12% ESP at different moisture levels and under different curing conditions. The effects of untreated, oven-dried, and calcined ESP on soil were analyzed, with a focus on changes in soil plasticity, maximum dry unit weight, optimum moisture content, and compressive strength.

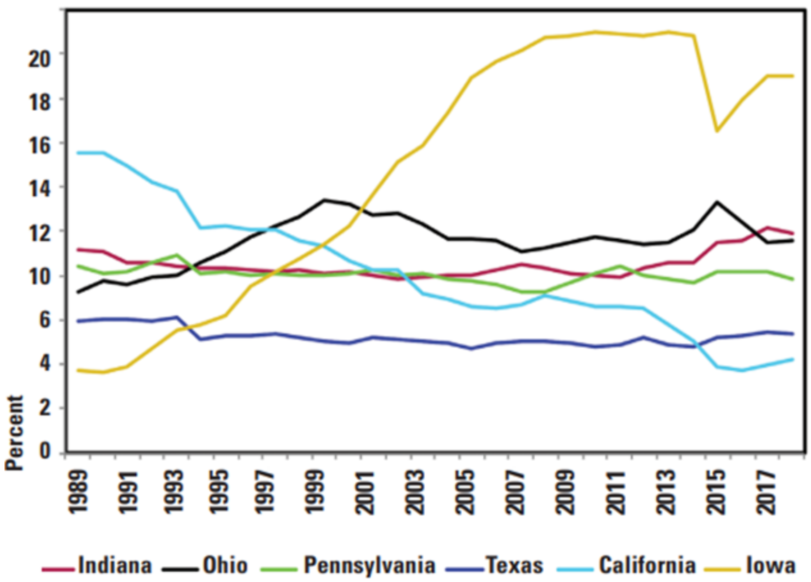
The addition of ESP, particularly calcined ESP, significantly and positively influenced the engineering properties of the soils studied. It was found that untreated and oven-dried ESP modestly increased soil plasticity and density by acting primarily as filler material without inducing significant chemical changes. Calcined ESP, rich in CaO, markedly improved soil strength through the hydration of lime to hydroxide, particularly when ESP was added at an 8% rate by dry soil weight and the soil was at optimal moisture content. The effectiveness of calcined ESP was enhanced under elevated curing temperatures, significantly increasing the compressive strength of Soil B by over 20 times. Furthermore, most of the strength development in calcined ESP-treated soil occurred within the first seven days of curing, with extended curing periods offering limited additional benefits. Follow-up laboratory-based dynamic cone penetration (DCP) testing validated the remarkable improvements to the soil's bearing capacity resulting from the addition of calcined ESP. To characterize the microstructure of untreated soil, ESP, and ESP-treated soil specimens, X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) technologies were employed.

This study highlights the promising potential of calcined ESP as an efficient and sustainable soil stabilizer. The ability of this material to significantly enhance soil strength, combined with the environmental benefits of repurposing eggshell waste, positions ESP as a valuable alternative to traditional stabilization materials. Future research could further explore the scalability of ESP treatment for large-scale field applications and its long-term durability under various environmental conditions.

CHAPTER 1: INTRODUCTION

Background and Motivation

Over the past few decades, Iowa's egg industry has seen rapid growth, solidifying the state's position as the leading egg producer in the United States, as shown in Figure 1. In 2018, the industry boasted approximately 57.7 million laying hens and produced more than 10 billion eggs, despite setbacks from the highly pathogenic avian influenza (HPAI) outbreak in 2015. The Iowa Egg Council reported nearly 16 billion eggs produced and more than $2 billion in total sales from September 2019 to August 2020, which highlights the industry's significant economic impact. A strategic shift towards producing eggs in liquid or dried form for the food manufacturing sector, a process utilizing integrated packaging and crushing facilities, is aimed at mitigating freight costs to coastal markets and enhancing Iowa's competitive edge over other states like Pennsylvania and California (Ibarburu et al. 2019). This strategy is expected to bolster the competitiveness of Iowa's egg industry.



Ibarburu et al. 2019

Figure 1. Market share of the top five egg-producing states and California

However, this booming industry also generates a substantial amount of eggshell waste. Composed primarily (up to 96%) of calcium carbonate ($CaCO_3$) (Chumlong et al. 2007), eggshell waste offers a diverse range of recycling opportunities, from simple uses in fertilizers and animal feed to more complex uses in materials for human consumption and heavy metal absorption and as catalysts in biodiesel production (Sim et al. 1983, Hossain et al. 2023, Saldanha et al. 2021). The versatility of eggshells extends to everyday life, including their use as biodegradable seedling pots in gardening.

In the realm of engineering materials, eggshells can serve as bio-based cementitious materials, enhancing the performance of products like fiber-reinforced concrete, masonry blocks, and so on. Due to their high calcium content, eggshells also offer the potential to be used as a calcium-based stabilizer material (CSM) capable of improving soil and aggregate binding through various chemical reactions. Sathiparan (2013) highlights the potential of eggshell powder (ESP) to serve as a sustainable construction material capable of replacing conventional materials due to its ability to react with soil constituents to form binding compounds. This innovative application of ESP underscores its value as a soil stabilizer, demonstrating the breadth of eggshells' utility and the potential for eggshell waste to contribute significantly to sustainable construction and environmental conservation efforts.

Internationally, the use of eggshells as a bio-based cementing material has shown success in applications ranging from cement replacement to soil stabilization and even in bone and dental implants. This success abroad underscores the potential for utilizing Iowa's eggshell waste to enhance the engineering properties of local geo-materials, potentially leading to more durable pavement and gravel road systems. It is hypothesized that such an innovative and sustainable approach may lead to stronger and more durable pavement foundations and gravel road systems in Iowa.

Research Objective and Scope

The primary objective of this study was to investigate and validate the utilization of Iowa eggshell waste as a bio-based cementitious material for enhancing and stabilizing frost-susceptible soils and improving the quality of local aggregates currently employed in pavement and gravel road construction across Iowa. To achieve this overarching goal, the study set forth specific objectives:

- Identify and characterize Iowa eggshells
- Perform laboratory assessment of geo-material stabilization techniques using Iowa eggshells
- Demonstrate the success of and/or improve eggshell-based geo-material stabilization techniques for use in Iowa pavements and gravel roads and develop implementation recommendations for their real-world application

These objectives aimed to provide a foundation for the practical application of eggshell waste, transforming it from a disposal challenge into a valuable resource for infrastructure development. Through detailed characterization and rigorous testing, the study sought to establish a sustainable, environmentally friendly approach to improving soil and aggregate materials for better performing and more durable pavement and gravel road systems in Iowa.

Report Organization

This report is structured into seven chapters, as shown in Figure 2. Chapter 1 introduces the background, motivation, and objectives of the study. Chapter 2 discusses the comprehensive literature review on the use of eggshell waste as a soil stabilizing agent. Chapter 3 describes the research methodology. Chapter 4 presents the laboratory testing results. Chapter 5 describes the methods and results of a proof-of-concept demonstration of the technique. Chapter 6 presents the key findings and conclusions. Chapter 7 proposes recommendations for future research.

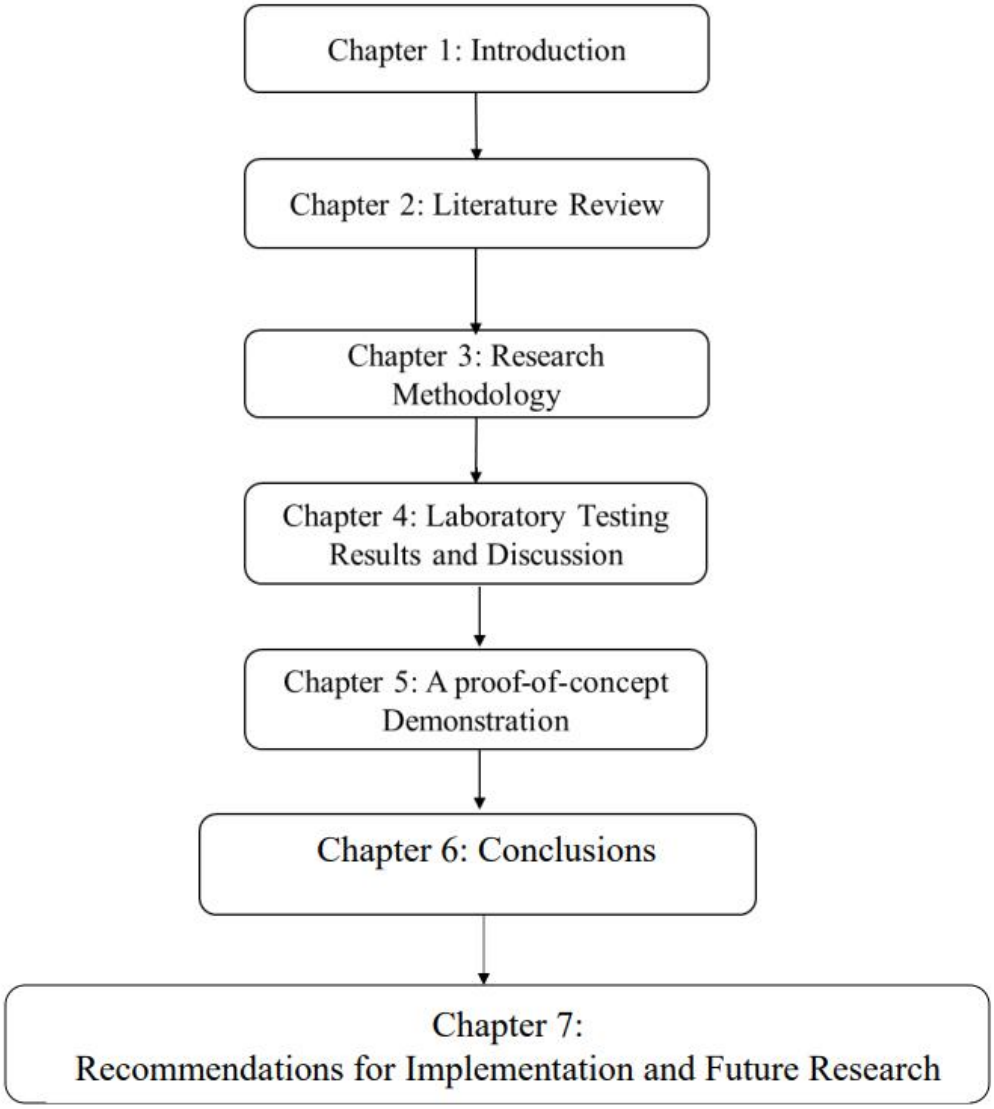


Figure 2. Report organization

CHAPTER 2: LITERATURE REVIEW

Overview of Geo-material Stabilization

Since the poor engineering properties of natural soil cannot provide the desired foundation for pavement structures, a common practice, termed soil stabilization, has been widely used to make soil strong enough to support pavement construction. Soil stabilization is a process for improving soil strength properties through mixing of additives. Traditional additives such as portland cement, lime, and fly ash have been widely used worldwide to stabilize soil, and extensive studies over many decades have investigated the use of traditional stabilizers in terms of their stabilizing mechanisms and mix design procedures. As one of the most commonly used stabilizing agents, lime is very effective for weak soils with high plasticity (Malkanthi et al. 2020). It can alter the engineering properties of clayed soil through pozzolanic reactions (Akula and Little 2020).

In a white calcium-based material, there are two types of lime products, quicklime (CaO) and hydrated lime ($Ca(OH)_2$), and the underlying mechanism of lime stabilization is the pozzolanic reaction. Similar to the cement hydration process, chemical reactions between the lime products (calcium-rich) and clay components (silica-rich) of soil mainly produce strong and stable gel structures termed calcium silicate hydrates (CSH). Secondary reactions such as flocculation, cation exchange, and carbonation can also occur in the presence of water and improve soil workability and strength capacity (Winterkorn and Pamukcu 1991). A simplified qualitative view of typical soil-lime reactions is as follows:

$$CaCO_3 \rightarrow CaO + CO_2$$
$$Ca^{2+} + 2(OH)^- + SiO_2(Clay\ Silica) \rightarrow C - S - H$$
$$Ca^{2+} + 2(OH)^- + Al_2O_3(Clay\ Aliumina) \rightarrow C - S - A$$
$$Ca^{2+} + 2(OH)^- + CO_2 \rightarrow CaCO_3 + H_2O$$

where C = CaO, S = SiO_2, A = Al_2O_3, and H = H_2O.

Much research has investigated the benefits and limitations of lime stabilization. Kavak and Akyarlı (2007) cooperated to conduct laboratory and field testing on green and brown clays treated with 5% lime, and a significant increase of up to 21 times in the soaked California bearing ratio (CBR) was obtained after 28 days. Keybondori and Abdi (2021) conducted a series of laboratory tests to investigate lime stabilization in clayed soil with high plasticity. They claimed that lime could reduce the plasticity of clay up to 94% and cause a reduction in the maximum dry density and optimum moisture content (OMC). Because lime is a rapid drying agent, the strength increase due to lime treatment may require waiting a couple of weeks or months to avoid long-term strength loss, and problems related to slow strength gain and lime's caustic properties must also be considered.

The disadvantages of conventional additives, such as their relatively high cost and lack of environmental friendliness, cannot be ignored, and such concerns have forced engineers to seek sustainable alternatives to traditional stabilizers. In recent years, the rapid development of nontraditional stabilizers has created hundreds of new products for soil stabilization. Among these additives, recycled materials like eggshells offer great potential for use in soil stabilization due to their rich calcium content (Sathiparan 2021).

Methods of Producing Eggshell Powder

Based on Iowa Egg Council reports (Ibarburu et al. 2019), Iowa produced more than 10 billion eggs between September 2019 and August 2020, and since approximately 70% of Iowa eggs are processed into liquid or dried egg form, a large number of eggshells are generated as food waste and generally dumped into landfills. Eggshells are composed primarily (i.e., up to 96%) of $CaCO_3$, indicating their significant potential to be recycled and reused for various purposes as calcium-based materials (Chumlong et al. 2007).

Typical recycling applications for waste eggshells include their use in fertilizer, animal feed ingredients, bone and dental implants, and bio-cement (Sathiparan 2021). For such purposes, eggshells must go through treatments such as grinding, heating, and bleaching to produce ESP, and the various treatment methods could significantly impact the calcium content of ESP. The grinding process is the first step to making ESP, and the duration and speed of grinding are two significant factors that can change the characteristics of ESP. Increases in rotational speed and grinding time elevate the Brunauer-Emmett-Teller (BET)-specific area (Tsai et al. 2008).

Heating is another vital treatment influencing the calcium composition of ESP. During heating, calcite ($CaCO_3$) in eggshells can decompose to CaO and CO_2, resulting in noticeable weight loss. Sathiparan (2021) summarized the effects of oven drying and calcination on the weight loss of eggshells by reviewing several studies. The authors concluded that oven drying (80°C to 120°C) could produce ESP with up to 50% CaO, while calcination (400°C to 1,000°C) could produce ESP with up to 75% CaO. Elevated temperatures up to 700°C could produce a constant weight loss of less than 4% due to the removal of moisture and organic matter in eggshells. Once the temperature reaches between 700°C and 900°C, decomposition of $CaCO_3$ into CaO will occur, leading to a weight loss of approximately 40%. A high temperature greater than 900°C could decompose all $CaCO_3$ in eggshells and result in a weight loss of approximately 45%. Bleaching, an optional process involving the use of NaOH, isophthalic acid, ethanol, or a combination of these, can create a purified form with a calcium concentration of over 98% (Shuhadah and Supri 2009, Pliya and Cree 2015).

In summary, treatment of eggshells can be divided into three steps. First, clean, grind, and sieve the eggshells to get ESP. Second, air dry, oven dry, and calcinate the ESP or use combined heating methods to obtain ESP with a high calcium concentration. Third, bleach the ESP to obtain purified CaO powder.

Review of Eggshell Powder as a Soil Stabilizer

Several independent research efforts related to the effects of using ESP in soil have been documented. Because ESP contains a high amount of CaO, it can accomplish the same cation exchange, flocculation, and pozzolanic reaction as other lime products. A study carried out by Prasad et al. (2016) assessed the effects of ESP on clay strength and claimed that 15% is the optimum rate to achieve a strength increase of up to four times. Amu et al. (2005) investigated the engineering properties of lime-ESP-stabilized expansive clay and showed that combining 3% lime with 4% ESP is optimal. Consoli et al. (2020) examined ESP for potential use as a stabilizer in sand and found that hydrated lime consisting of ESP could achieve higher strength than calcitic hydrated lime and eggshell-based CaO (quicklime). Alzaidy (2019) performed unconfined compressive strength (UCS), shear, and CBR tests on ESP-treated clayed soil, with results revealing that 5% ESP-treated soil could achieve the highest strength parameters and that an increase in ESP content could reduce the swelling potential of the soil. Saldanha et al. (2021) evaluated the environmental impacts of eggshell limes and commercially available limes using life-cycle assessment (LCA). Their study claimed that the application of eggshell limes could reduce ecosystem damage by up to 65% compared to traditional limes. Sathiparan et al. (2021) reviewed more than 10 articles and created a comparison figure to evaluate the effects of ESP on soil CBR values and UCS. According to Sathiparan et al. (2021), an increase in ESP content elevates the soil CBR values and strength capacity.

Summary

The Iowa egg industry produces a massive number of eggs every year, resulting in a massive quantity of eggshells typically disposed of in landfills. However, since eggshells contain a high concentration of $CaCO_3$, they have great potential to be reused as a soil stabilizer similar to lime. This new application can enhance the economic value of the Iowa egg industry.

Eggshells can go through grinding, heating, and bleaching to become an ESP-based lime product. The high content of CaO in ESP results in a stabilization mechanism that relies on the hydration of lime to hydroxide, cation exchange, and flocculation in clay to form strong soil structures. Extensive research efforts have concluded that ESP could enhance soil strength properties and reduce soil swelling, indicating that ESP could fully or partially replace lime in soil stabilization. Replacing lime by utilizing ESP also reduces environmental impacts and construction costs compared to traditional lime. Although several previous laboratory studies have investigated the performance of ESP-based stabilizers, their application has been restricted by many factors, such as a lack of treatment standards and mixing guidance and inadequate field validation.

CHAPTER 3: RESEARCH METHODOLOGY

Experimental Materials

Natural Soil

To assess the effects of using Iowa eggshells to modify a soil's engineering properties, the researchers collected two types of local subgrade soil representing typical soil types found in Iowa (Figure 3). Soil A, sourced from beneath a granular road in Buchanan County, upon microstructural analysis displayed a significant presence of quartz (SiO_2) and calcite ($CaCO_3$). Soil B, an expansive soil type known as loess, was collected from a quarry in Pottawattamie County, and it consisted of albite (($NaAlSi_3O_8$), calcite, and quartz. The grain size distribution of both soils, detailed in Figure 4, revealed Soil A to be comparatively coarse due to its lower fines content. The engineering characteristics of these soils are summarized in Table 1, which incorporates classifications from both the American Association of State Highway and Transportation Officials (AASHTO) and the Unified Soil Classification System (USCS). According to these classifications, Soil A received an A-4 classification and was categorized as a clayey soil (CL-ML), while Soil B received an A-6 classification and was also deemed clayey. Furthermore, the Atterberg limits and compaction characteristics outlined in Table 1 indicate that Soil B exhibited a higher plasticity, optimum moisture rate, and maximum dry unit weight compared to Soil A, highlighting distinct differences in the soils' physical properties.

Figure 3. Collected subgrade soils

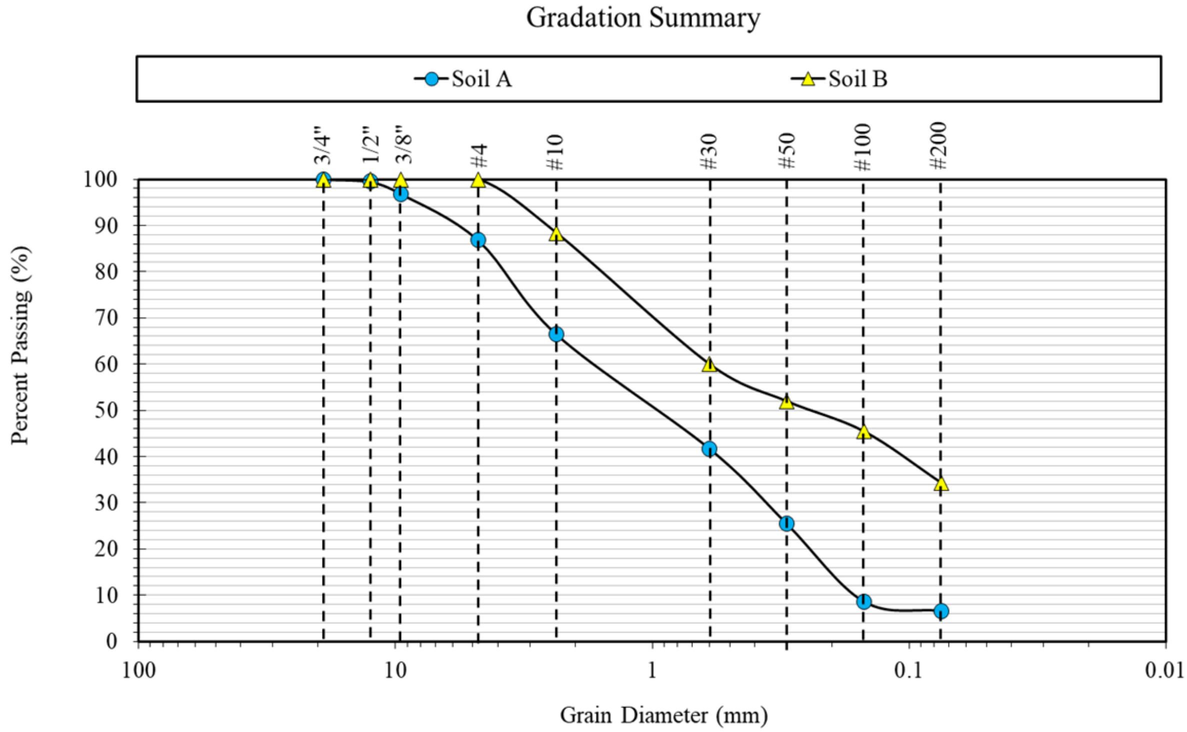


Figure 4. Grain size distribution for subgrade soils

Table 1. Engineering properties of two different soils investigated

Property	Soil A	Soil B
Classification		
AASHTO (group index)	A-4	A-6
USCS group symbol	CL-ML	CL
USCS group name	Silty Soil	Clayey Soil
Atterberg limits		
Liquid limit (LL), %	28.1	28.0
Plasticity limit (PL), %	19.3	16.5
Plasticity index (PI), %	8.8	11.5
Proctor Test		
Optimum moisture content (OMC), %	13.1	14.7
Maximum dry unit weight ($\gamma_{d\ max}$), lb/ft^3	103.2	107.7

Eggshell Materials

Based on phone communication with five local egg farms, it was found that in Iowa, most eggshells are typically discarded into landfills immediately after use, with only a minor fraction being recycled for livestock feed or used as agricultural lime material. The eggshell waste examined in this study was sourced from Rose Acre Farms, Inc., located in Guthrie County, Iowa. The eggshells provided for the research were minimally processed, having only been roughly washed and crushed. Quantitative analysis revealed that approximately 100 eggshells weigh around 1.3 lb, underscoring the need for effective management and utilization strategies to handle this type of waste. An examination of the condition of these materials showed that they contained a significant amount of water and eggshell membranes, as depicted in Figure 5, and emitted a strong sulfur smell. To make these materials suitable for further use, a cleaning process was necessary to eliminate the egg residues and remove the unwanted eggshell membranes, and using disinfecting bleach solution was an effective method.

Figure 5. Raw eggshells

Figure 6 details the cleaning process for raw eggshell wastes that utilizes a chemical solution to effectively remove the egg residues and eggshell membranes. For a 5-gallon bucket of eggshells weighing roughly 28 to 32 lb, the procedure required mixing 3 gallons of 7.5% sodium hypochlorite (SHC) disinfecting bleach solution with 1 gallon of water, resulting in a 4-gallon diluted 5.5% SHC solution. This specific mixture was used to soak one bucket of eggshells for about 24 hours, ensuring that the egg residues and eggshell membranes were fully dissolved. Following the soaking period, the eggshells were washed once more then allowed to dry thoroughly. After completing this cleaning process, the weight of the odorless, clean, and dry eggshells remaining in the 5-gallon bucket was significantly reduced to approximately 18 to 22 lb. This procedure highlights an effective approach to preparing eggshell waste for further application by removing organic contaminants and reducing the material’s overall weight.

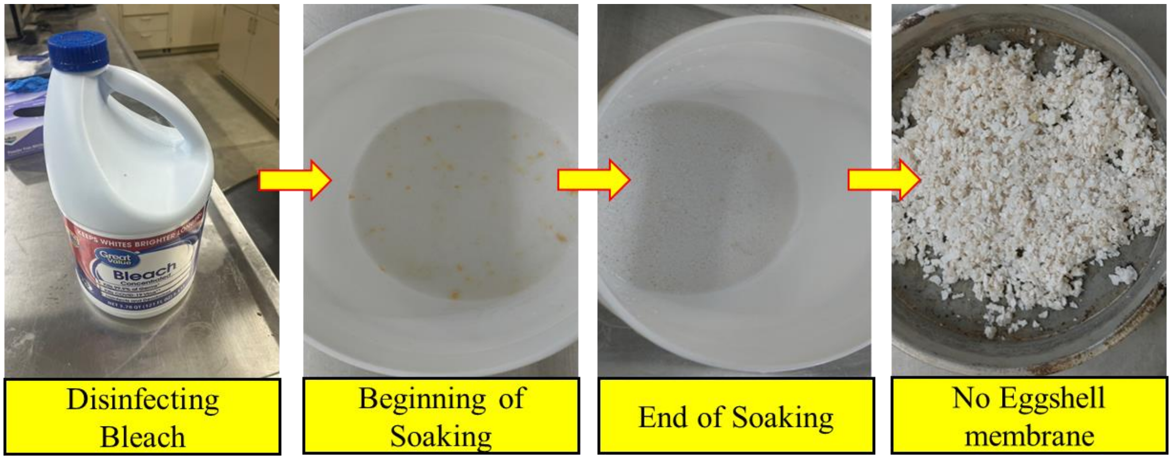


Figure 6. Procedure for cleaning eggshell materials

Following the cleaning procedure, the purified eggshell materials were prepared for further processing using a ball mill that ground them into ESP. To facilitate efficient grinding, the ball mill was pre-loaded with a specific combination of grinding media consisting of 180 1½ in. flint balls, 540 1 in. flint balls, and 1,260 5/8 in. steel balls. A single batch of clean eggshells weighing between 18 and 22 lb was then loaded into the ball mill for grinding. The process took approximately 10 minutes at a rotational speed of 30 revolutions per minute (RPM), as depicted in Figure 7. This grinding operation resulted in the production of ESP, characterized as a fine, white powder.

Figure 7. Ball mill machine and resulting ESP

Thanks to its capacity to grind vast quantities of eggshells into a fine powder efficiently, a ball mill is highly effective for large-scale production of ESP. In cases where a ball mill is not available, a coffee grinder presents a viable alternative for small-scale ESP production. Such adaptability allows for continued production of ESP under varying conditions and scales of operation. Figure 8 compares the grain size distribution of clean eggshell materials processed using both a coffee grinder and a ball mill. The comparison clearly demonstrates that the ball mill is capable of producing the finest ESP, achieving particle sizes smaller than those passing through a No. 10 sieve (0.0787 in.). This finer grain size of ESP produced by the ball mill potentially increases the specific surface area of the powder, in turn enhancing the efficiency of chemical reactions involving ESP. This attribute is particularly beneficial in applications requiring a high degree of reactivity, such as in the production of bio-based cementitious materials or in soil stabilization efforts, where the finer powder can more readily participate in the necessary chemical processes. This grinding process showcases an innovative recycling approach for eggshell waste.

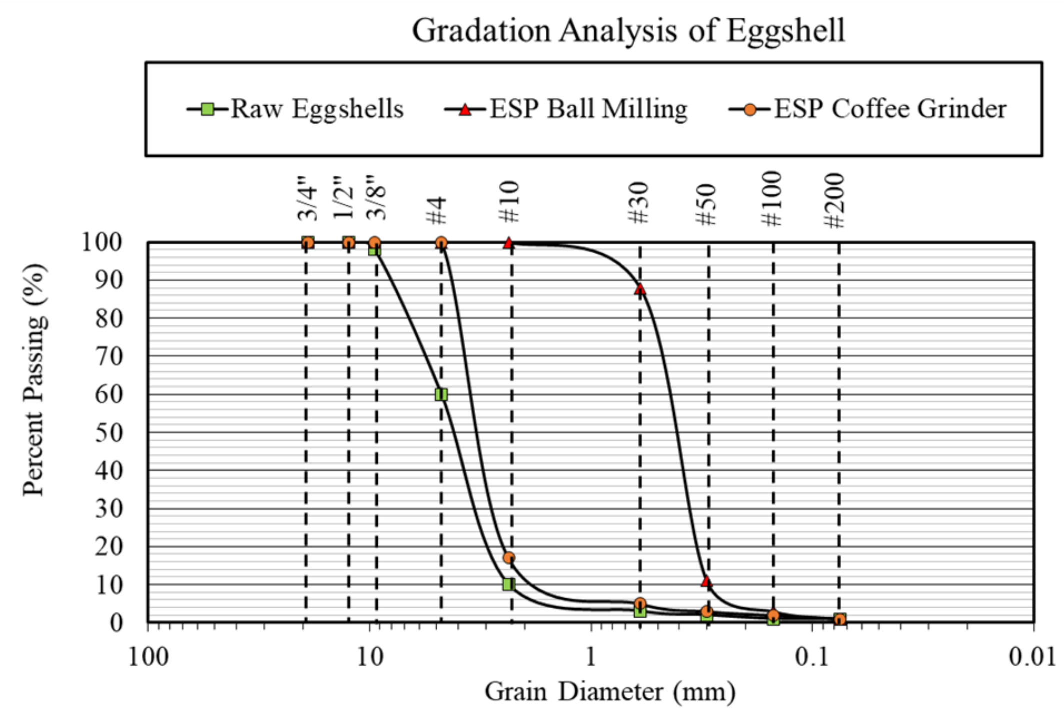


Figure 8. Gradation of eggshell materials and ESP

Processing Eggshell Powder

The literature review highlighted that ESP possesses a high content of $CaCO_3$ that can be enhanced to increase the CaO content through various methods such as calcination, bleaching, or a combination thereof. However, since bleaching involves the use of different chemical additives and additional washing and cleaning procedures, making it a relatively costly and time-consuming approach, calcination emerges as a more cost-effective alternative for processing ESP. This study focused on calcination as the primary method for processing ESP, comparing calcined ESP with air-dried and oven-dried ESP for comprehensive analysis. Oven drying in the study (Figure 9) involved using a conventional oven and resulted in ESP with a light-yellow color, which was distinct from the air-dried version. The oven-dried ESP exhibited no significant mass change (<3%) after 12 hours of heating at 220°C.

Figure 9. Oven-dried ESP (220°C)

Calcination, conducted at very high temperatures in a conventional furnace, converts $CaCO_3$ in ESP to CaO. The literature suggests that this decomposition reaction requires temperatures of at least 800°C, but this study opted for 1,000°C to ensure complete purification of ESP in the furnace (Figure 10). In this study, the process transformed ESP into a black powder after one hour of heating, due to the burning of organic matter, and eventually back into a fine white powder after five hours, reflecting the completion of the decomposition process. Due to safety considerations, a significant cooling period of at least six hours was required before removal from the furnace. The mass measurement post-calcination showed a 55% loss, suggesting successful conversion of almost all $CaCO_3$ to CaO and significantly enhancing the engineering properties of the materials. This process highlights the potential of calcination to effectively and efficiently produce high-quality CaO from ESP, offering a promising avenue for the utilization of eggshell waste in various engineering and construction applications.

Figure 10. Calcined ESP (1,000°C)

This study meticulously explored three distinct processing methods—air drying, oven drying, and calcination—to achieve a comprehensive comparison of their effects on converting raw eggshell waste into ready-to-use ESP. Each method was carefully chosen to assess the most effective way to enhance the quality and usability of ESP for various applications. Figure 11 illustrates the entire sequence involved in each of the procedures, starting from the collection of raw eggshell waste through the final production of ESP. These procedures include initial cleaning and preparation of the eggshells, followed by grinding of the clean eggshells into a fine powder, and concluding with the drying or calcination process chosen. The detailed illustration not only serves as a guide for replicating the study's findings but also suggests the potential for these processes to be adapted and scaled up for larger field applications. By highlighting the differences in outcome for each processing method, the study provides valuable insights that can be used to optimize the conversion of eggshell waste to high-quality ESP and emphasizes the feasibility and environmental benefits of recycling eggshell waste into a useful commodity.

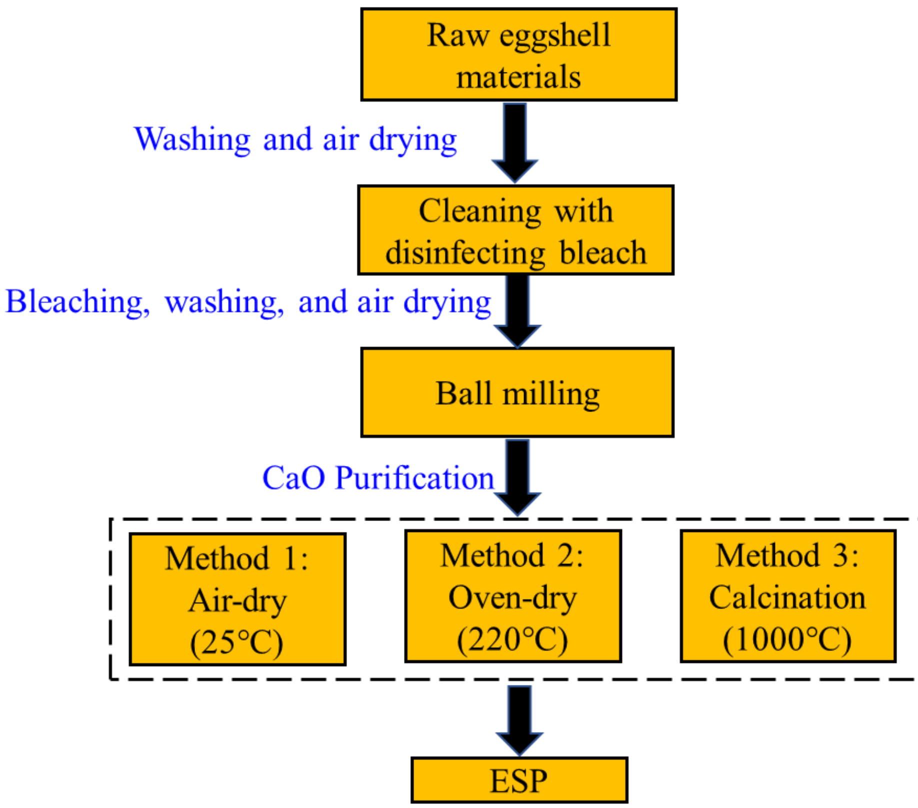


Figure 11. Production of ESP

Laboratory Testing Program

In this study, a comprehensive laboratory testing program was carried out to investigate the effects of ESP on the engineering properties of Iowa soils. To this end, ESP was added to each soil sample in proportions ranging from 6% to 12% of the soil's dry weight, with the aim of quantifying performance improvements. For comparative analysis, untreated soil specimens were also examined.

The initial phase of testing involved conducting the Atterberg limit test in accordance with ASTM D4318-17 to determine each soil's consistency through measurements of liquid limit (LL), plastic limit (PL), and PI. A standard Proctor compaction test, per ASTM D558-19, was also performed to ascertain the maximum dry unit weight (γd) and OMC.

Following these preliminary tests, Iowa soils mixed with ESP were compacted into 2 in. by 2 in. cylindrical samples for UCS testing using the method developed by O'Flaherty et al. (1963). This method is noted for its efficiency, requires less material and energy than other tests, and has been adopted in various studies (Yang et al. 2018, Yang et al. 2019) to evaluate the performance of innovative additives in soil stabilization. The compacted specimens are depicted in Figure 12, which illustrates the prepared samples for UCS testing. All specimens were wrapped with plastic film and aluminum foil for further curing conditioning.

Figure 12. ESP-treated 2 in. by 2 in. soil specimens

The study recognized that soil strength properties are highly sensitive to moisture content. As such, three moisture levels (OMC, OMC+3%, OMC+6%) were incorporated into the soil samples to evaluate the moisture resistance imparted by ESP treatment. The method of curing was also identified as a significant factor influencing the engineering properties of soil. Therefore, curing periods of 7 days and 28 days were applied to represent the early and long-term stages of strength development, respectively. Moreover, two curing temperatures were investigated—room temperature at 20°C and oven temperature at 40°C—to examine the impact of curing temperature on soil properties.

The laboratory program also evaluated the CBR of the ESP-treated specimens in accordance with ASTM D1883-21. This test, which simulates the worst-case scenario when the soil is fully saturated with water, is one of the most important assessment criteria for soil used in construction and engineering applications. The CBR specimens were compacted into 6 in. by 7 in. molds at the desired water contents with the desired ESP proportions and were then sealed and subjected to the desired curing period. This study thus investigated the CBR of soil enhanced with various types of ESP under differing curing conditions, aiming to discern the extent to which ESP could improve soil strength and under what conditions these improvements were most notable.

The microstructures of the untreated soil, ESP, and ESP-treated soil specimens were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) technologies. XRD was utilized to identify the mineral composition of the samples, providing insights into the crystalline structure. SEM generated images with magnifications ranging from 60x to 5000x, facilitating the analysis of material morphology. EDS was used for elemental analysis to examine the amounts of various elements within the samples. Through microstructural characterization, the underlying mechanisms of ESP treatment could be comprehensively understood.

Table 2 was prepared to summarize all variables investigated in the UCS testing program. The table encompasses the investigated soil types, ESP types, ESP contents, moisture levels, curing periods, and curing temperatures. To ensure reliability and minimize variance, each specimen group—with consistent variables—was replicated three times. This methodical approach aimed to provide a robust assessment of how ESP incorporation affects the mechanical properties of soil, potentially offering valuable insights into sustainable soil stabilization practices.

Table 2. Testing program of UCS

Variable	Number	Description
Soil type	2	A-4 (CL-ML), A-6 (CL)
ESP type	3	air-dried ESP, oven-dried ESP, calcined ESP
ESP content	5	0%, 6%, 8%, 10%, 12%
Moisture level	3	OMC, OMC+3%, OMC+6%
Curing period	2	7-day, 28-day
Curing temperature	2	20°C, 40°C

CHAPTER 4: LABORATORY TESTING RESULTS AND DISCUSSION

Atterberg Limits

The investigation into the effects of various types and quantities of ESP on soil consistency revealed distinct outcomes for Soil A and Soil B. As shown in Figures 13 and 14, when untreated and oven-dried ESP was introduced into both soil types, there was a moderate increase in the LL and PL, leading to a slight increase in soil plasticity. This enhancement in soil plasticity can be primarily attributed to the cation exchange activities prompted by calcium ions present in the ESP, facilitating the flocculation of clay particles. Such flocculation is likely due to the high calcium content in ESP, as suggested by studies such as Yang et al. (2019), Schwieger (1965), and Arman and Munfakh (1972). The specific characteristics of clay minerals, such as cation exchange capacity (CEC) and specific surface area (SSA), may also play a role in the differing impacts of ESP treatment across various soil types, as noted by Smith et al. (1985). Given that unheated and oven-dried ESP predominantly consists of $CaCO_3$, the availability of free exchangeable cations is relatively limited, leading to only minor changes in soil consistency.

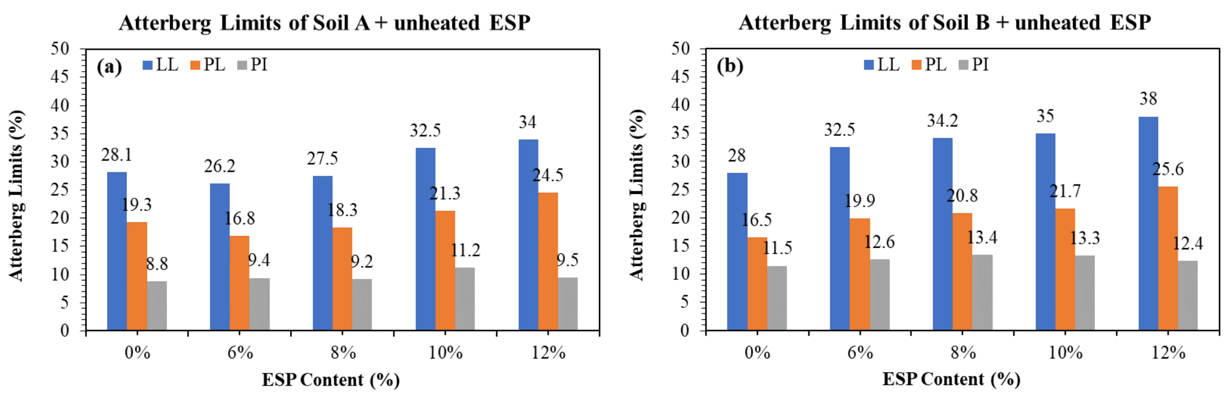


Figure 13. Results of Atterberg limits in soils treated with air-dried ESP

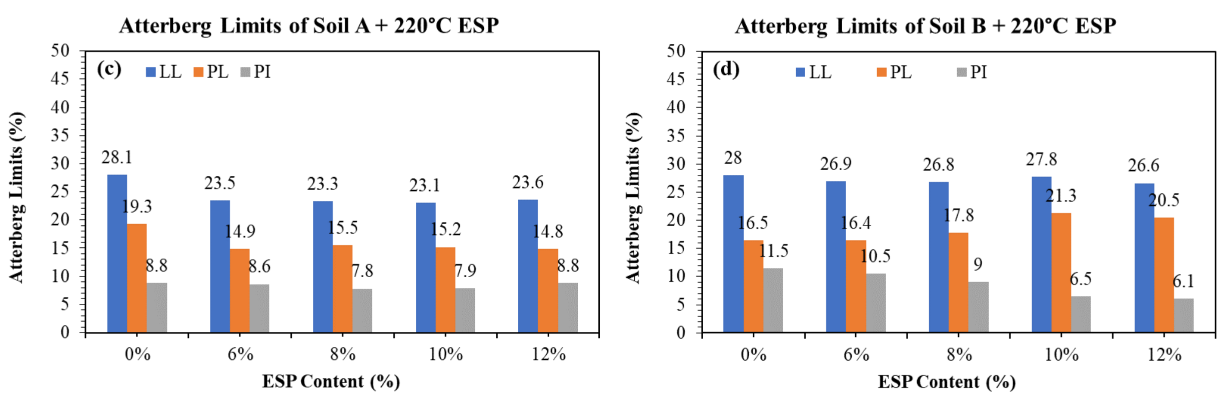


Figure 14. Results of Atterberg limits in soils treated with oven-dried ESP

In contrast, soils treated with calcined ESP demonstrated significantly different behavior compared to those treated with untreated and oven-dried ESP. The application of calcined ESP resulted in a notable decrease in both the LL and PI of the soils, although the PL values experienced an increase, as illustrated in Figure 15. This trend suggests a substantial alteration in soil plasticity and hydraulic properties, attributable to the fact that the calcined ESP is primarily composed of CaO rather than $CaCO_3$. CaO is more chemically active, capable of inducing hydration and pozzolanic reactions that generate stronger material formations and thereby reducing soil plasticity. Such changes typically correlate with decreased soil permeability, indicating that calcined ESP-treated soils exhibit significantly lower plasticity compared to untreated samples. The results from the Atterberg limits testing clearly showcase the potential of calcined ESP to serve as an effective soil stabilizer through the reduction of soil plasticity.

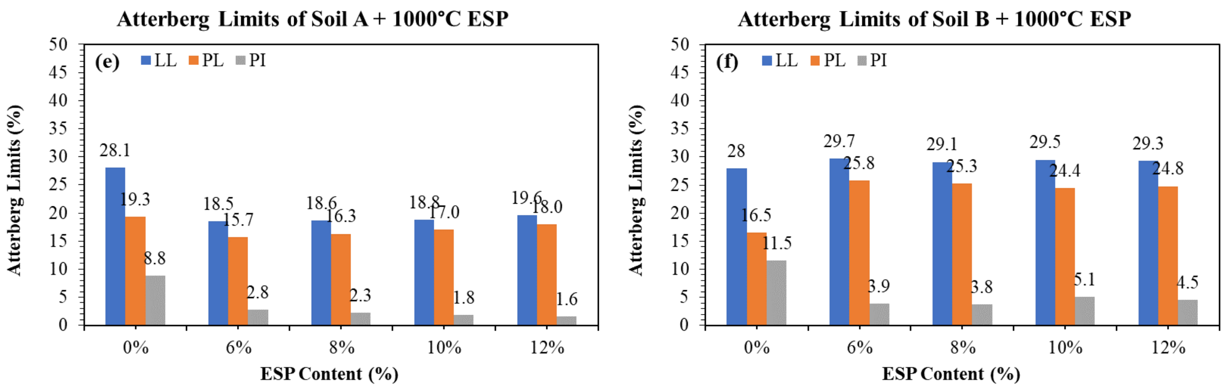


Figure 15. Results of Atterberg limits in soils treated with calcined ESP

Compaction Characteristics

The standard Proctor compaction test plays a crucial role in understanding the compaction characteristics of soil, particularly in determining the maximum dry unit weight and the OMC needed for effective compaction. The impact of ESP on these soil properties was meticulously analyzed, as depicted in Figures 16 through 18, which showcase the influence of different types of ESP on the compaction characteristics of Soil A and Soil B.

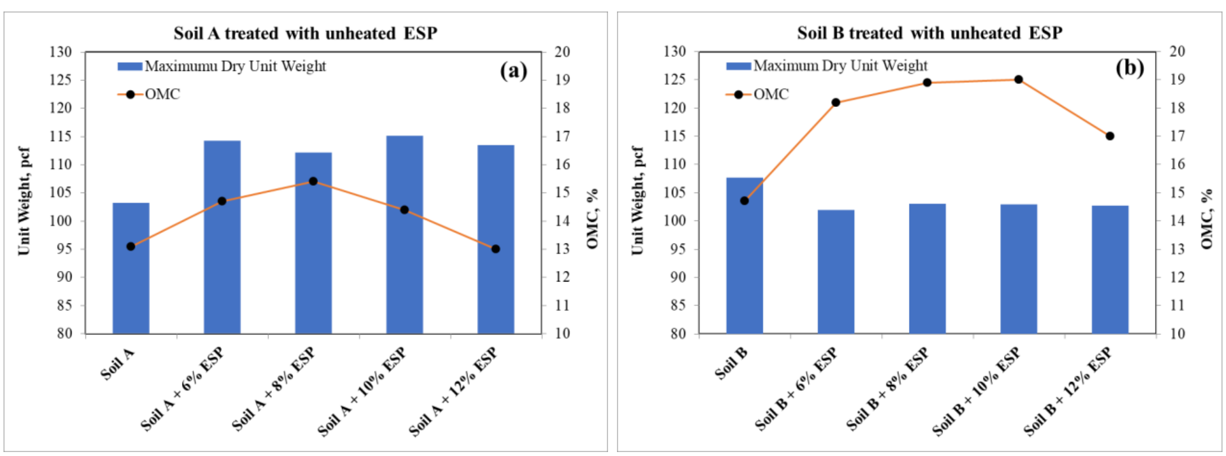


Figure 16. Results of compaction characteristics in soils treated with air-dried ESP

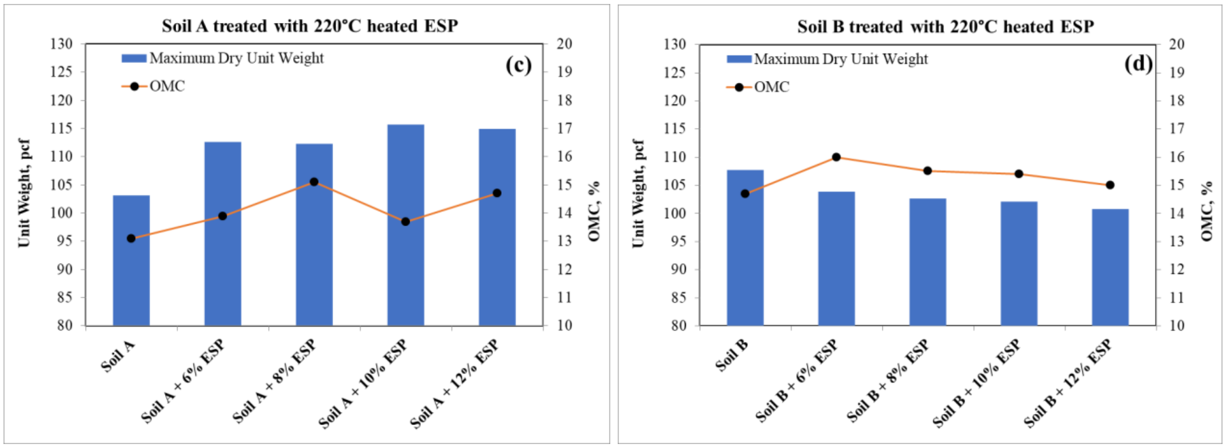


Figure 17. Results of compaction characteristics in soils treated with oven-dried ESP

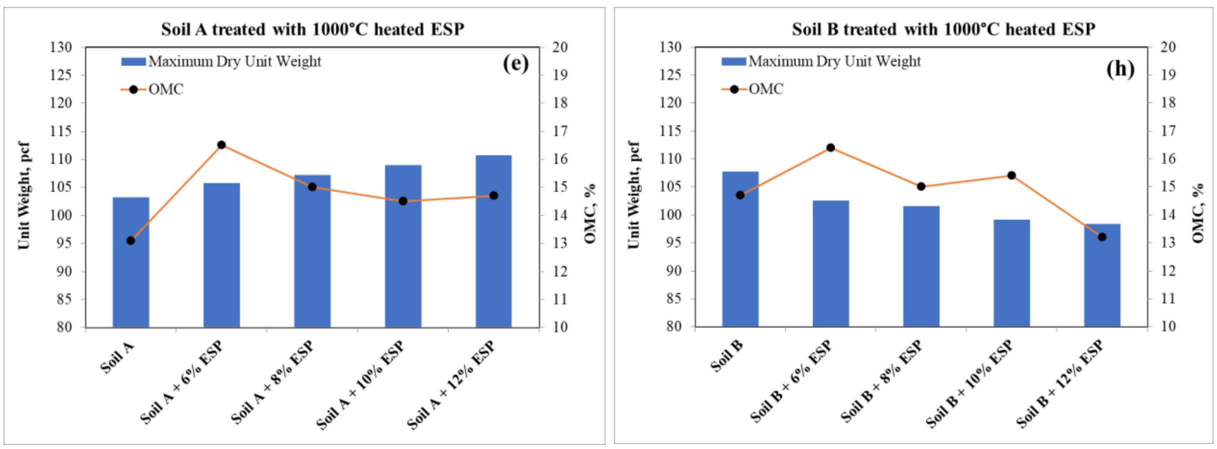


Figure 18. Results of compaction characteristics in soils treated with calcined ESP

For Soil A, a significant correlation was observed between ESP content and the soil's maximum dry unit weight. The addition of unheated and oven-dried ESP resulted in an effective increase in the soil's maximum dry density, while the impact of calcined ESP on increasing the maximum dry unit weight was relatively limited. The optimal performance in terms of OMC was noted at ESP contents of 8% for both unheated and oven-dried ESP and 6% for calcined ESP. This variation in effectiveness can be attributed to the grain size and chemical composition of the ESP used. Unheated and oven-dried ESP, primarily composed of $CaCO_3$, act more as filler materials within the soil matrix, enhancing densification during compaction without significant chemical interaction with the soil. Conversely, the CaO present in calcined ESP engages in immediate chemical reactions with the soil in the presence of moisture, leading to hydration reactions that produce flocculates. These flocculates contribute to resistance against compaction efforts, thereby limiting the increase in dry unit weight despite the filler effect provided by the calcined ESP.

The behavior of Soil B in response to ESP addition diverged significantly from that of Soil A. Being a finer soil with a high clay content, Soil B's compaction characteristics were influenced differently by ESP. The inclusion of ESP in Soil B served to replace soil grains and fill voids, enhancing the soil's compressibility. However, similarly to Soil A, the hydration reactions induced by calcined ESP generated flocculates that resisted compaction, leading to a potential decrease in the maximum dry unit weight. This difference underscores the complexity of soil-ESP interactions and highlights the need for a nuanced approach when considering ESP as a soil amendment that accounts for the specific soil type and the properties of the ESP being used.

Unconfined Compressive Strength

The UCS of soil is a critical parameter for evaluating a soil's suitability for and performance in construction and engineering applications. This study investigated the UCS properties of soil enhanced with various types of ESP—namely unheated, oven-dried, and calcined ESP—under differing moisture levels and curing conditions. The investigation aimed to discern the extent to which ESP could improve soil strength and under what conditions these improvements were most notable.

Figure 19 reveals the impact of adding unheated ESP on soil strength, highlighting that the optimum fractions of unheated ESP are 10% and 8% for Soil A and Soil B, respectively. This treatment resulted in a modest improvement in strength of less than 20%. Since this increment is attributed to the role of ESP as a filler material within the soil matrix rather than as an agent of chemical change, variations in the curing period did not substantially alter the strength outcomes. This result reinforces the notion that the primary benefit of unheated ESP in this context is its contribution to the physical structure of the soil rather than its chemical properties.

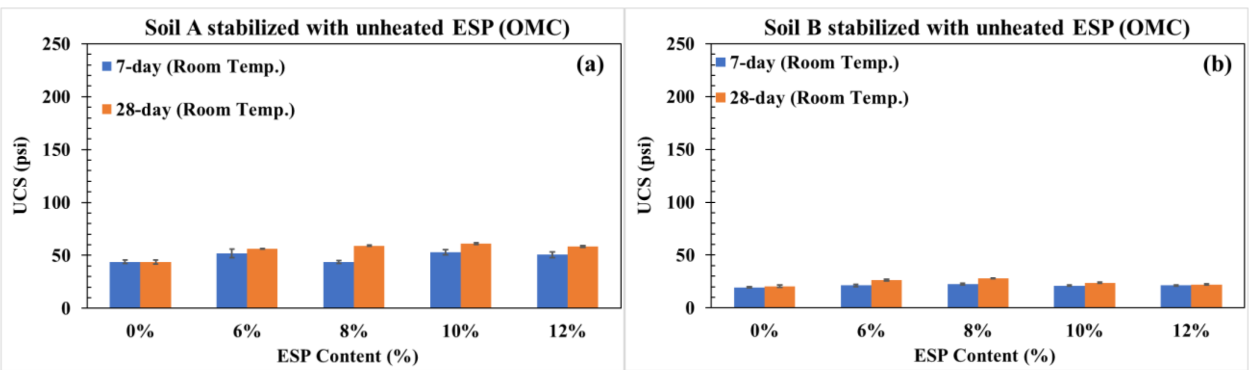


Figure 19. Results of UCS in soils treated with unheated ESP at OMC

Similarly, the results for soil specimens treated with oven-dried ESP, shown in Figure 20, align closely with those observed for unheated ESP. This similarity stems from the fact that the temperature used for oven drying ESP (220°C) is insufficient to decompose $CaCO_3$, meaning that the chemical composition of ESP remains unchanged through oven drying. Thus, both unheated and oven-dried ESP primarily serve as physical fillers, enhancing soil strength through mechanical means rather than through chemical interaction.

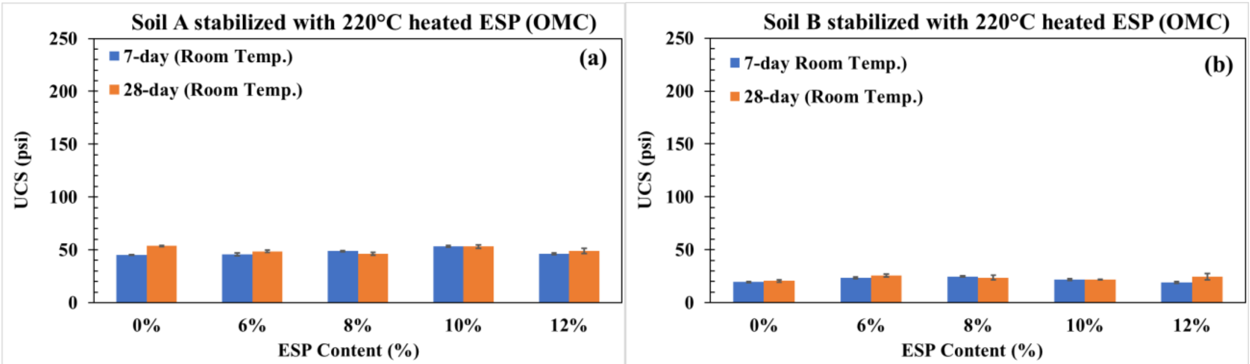


Figure 20. Results of UCS in soils treated with oven-dried ESP at OMC

The findings underscore that extending the curing period or increasing the temperature does not significantly impact the performance of unheated and oven-dried ESP because their contribution to soil strength is not influenced by chemical reactions that might be affected by these factors. Moreover, increasing the moisture level does not contribute to further strength gains for these types of ESP; rather, higher moisture levels can adversely affect the soil's strength capacity. Excessive moisture may even complicate the molding of cylindrical specimens for UCS testing, which further highlights the delicate balance between moisture content and the physical properties of soil when enhanced with ESP.

The results for soil specimens treated with calcined ESP illustrate a fundamentally different mechanism of soil stabilization compared to their counterparts treated with unheated and oven-dried ESP. The primary component of calcined ESP, CaO, engages actively with moisture to initiate hydration and pozzolanic reactions that significantly contribute to the stabilization process. Figure 21 showcases the UCS results from soil specimens treated with calcined ESP. These results reveal a remarkable strength improvement of 5 to 8 times when an additional 3% moisture above the OMC is applied and the specimens are cured at room temperature for up to 28 days.

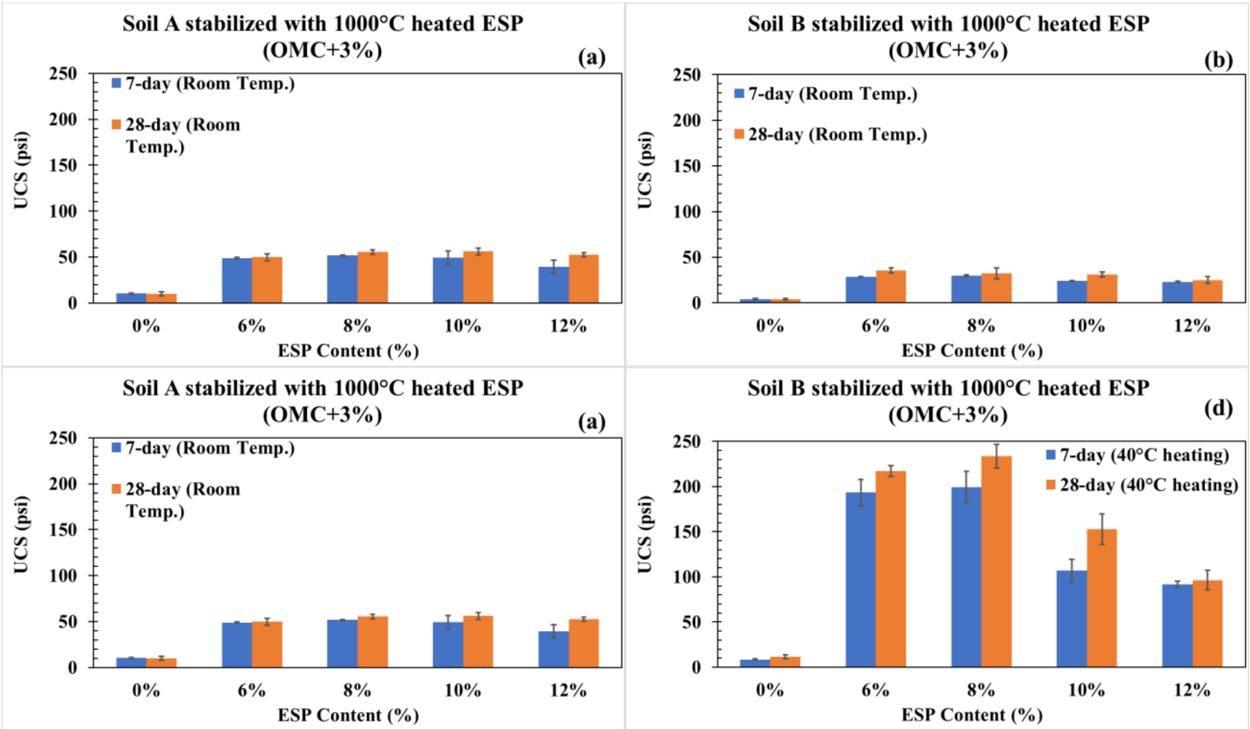


Figure 21. Results of UCS in soils treated with calcined ESP at OMC+3%

Elevating the curing temperature was found to further facilitate these hydration and pozzolanic reactions. While a 40°C curing temperature did not significantly enhance the strength capacity of Soil A, it drastically improved the strength of Soil B by over 20 times, with 8% identified as the optimum calcined ESP application rate. However, when the moisture level was increased to OMC+6%, as shown in Figure 22, a decrease in strength was observed across all treated soils compared to those at OMC+3%, with 8% remaining as the optimum ESP content for strength enhancement. Notably, at a moisture level of OMC+6%, it was impossible to manufacture untreated soil specimens due to excessive moisture, which highlights the critical role of CaO in absorbing moisture and accelerating the hydration and pozzolanic reactions.

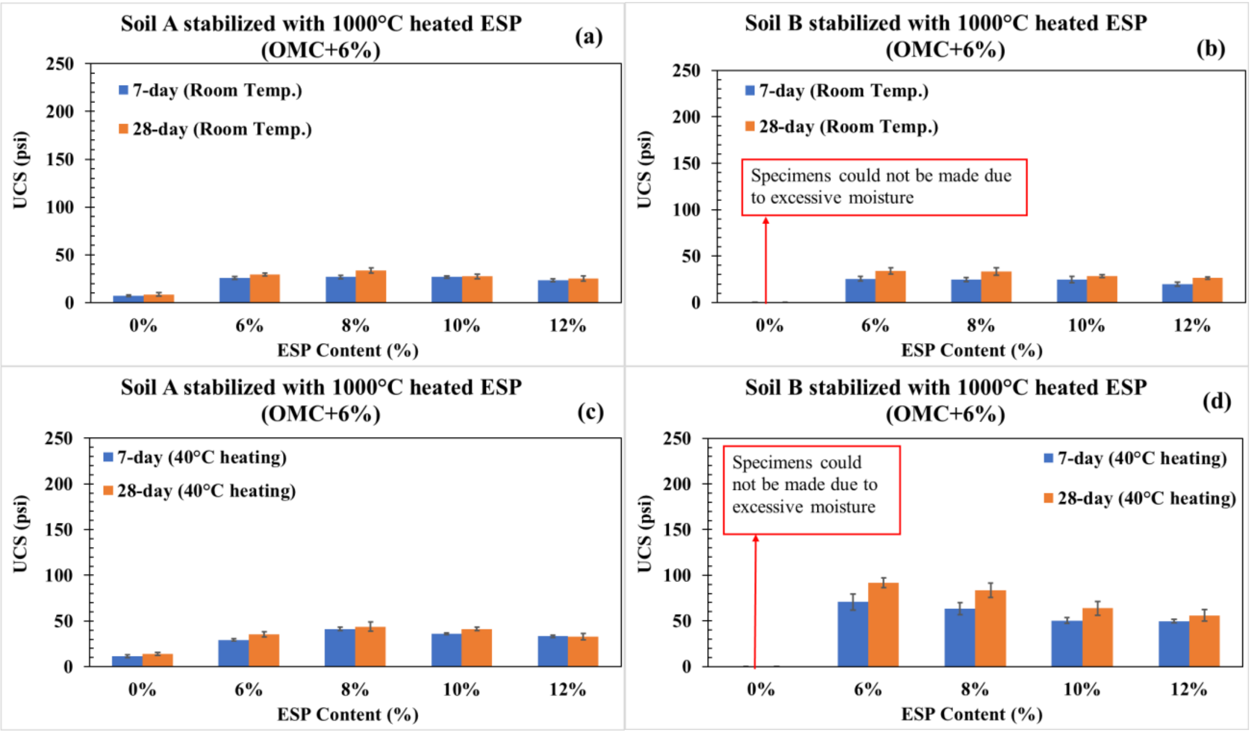


Figure 22. Results of UCS in soils treated with calcined ESP at OMC+6%

The observation regarding the impact of extended curing periods on soil strength, particularly for specimens treated with calcined ESP, also offers insights into the kinetics of strength development in stabilized soils. The finding that extending the curing period to 28 days does not confer significant additional strength gains suggests that the majority of strength development in calcined ESP-treated specimens occurs within the early stages of curing, specifically within the first 7 days.

The results also suggest that the addition of 3% moisture above the OMC is the most effective for leveraging the benefits of calcined ESP. Furthermore, a higher curing temperature is beneficial for achieving greater strength gains because it enhances the rate and efficiency of the hydration and pozzolanic reactions. This is particularly evident in the significant increase in soil strength, especially for Soil B, under these conditions. These findings underline the potential of calcined ESP as a highly effective soil stabilizer. This material offers substantial improvements in soil strength through mechanisms that capitalize on the chemical interactions between CaO and soil moisture, thereby presenting a promising avenue for soil stabilization projects.

California Bearing Ratio

Soil CBR as measured by ASTM D1883-21, which simulates the worst-case scenario when the soil is fully saturated with water, is an important parameter for evaluating a soil's strength and performance in construction and engineering applications. Therefore, the test samples were compacted at the desired water content with the desired ESP portion, then sealed and subjected to the desired curing period (i.e., the specimens were cured at room temperature for up to 28 days). This study investigated the CBR of soils enhanced with various types of ESP: unheated, oven-dried, and calcined ESP under differing curing conditions. The investigation aimed to discern the extent to which ESP could improve soil strength and under what conditions these improvements were most notable.

Figure 23 reveals the impact of adding unheated ESP on soil strength, highlighting that the optimum rate of unheated ESP is 10% for both Soil A and Soil B. ESP acts primarily as a physical filler within the soil structure rather than as an agent of significant chemical changes but nevertheless leads to improvements in the soil's properties. The duration of curing became more noticeable in the CBR test results than in other test results and was found to influence the strength outcomes. This observation supports the idea that unheated ESP's main advantage in this context is its contribution to the soil's physical composition in addition to any chemical effects it may have.

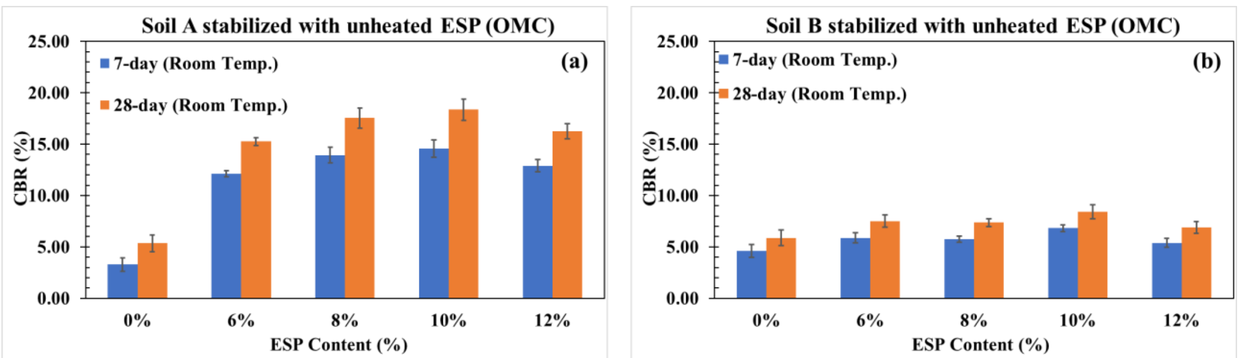


Figure 23. Results of CBR in soils treated with unheated ESP at OMC

Similarly, the results for soil specimens treated with oven-dried ESP, as shown in Figure 24, align closely with those observed for soils specimens treated with unheated ESP. This similarity stems from the fact that the temperature used for oven drying ESP (220°C) is insufficient to decompose $CaCO_3$, meaning that the chemical composition of ESP remains unchanged through oven drying. Thus, both unheated and oven-dried ESP primarily serve as physical fillers, enhancing soil strength through mechanical means rather than through chemical interaction.

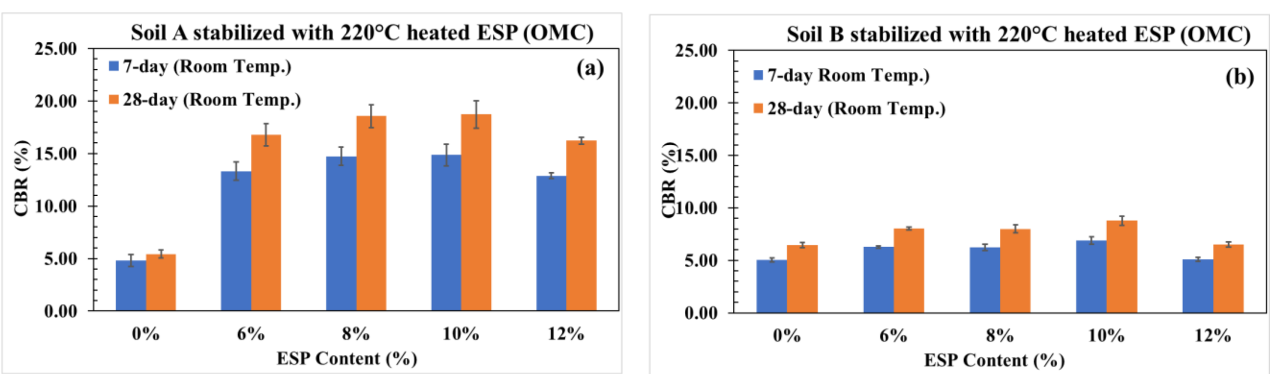


Figure 24. Results of CBR in soils treated with oven-dried ESP at OMC

Soil specimens treated with calcined ESP were made with excessive water content (OMC+3% and OMC+6%, shown in Figures 25 and 26, respectively) to illustrate the mechanism of soil stabilization for these samples compared to the mechanism exhibited by unheated and oven-dried ESP-treated samples. The primary component of calcined ESP, CaO, engages actively with moisture to initiate hydration and pozzolanic reactions that significantly contribute to the stabilization process. Figure 25 shows that the CBR results of samples without ESP and with a moisture content of OMC+3% were five times lower than the CBR results for samples prepared at OMC (depicted above in Figures 23 and 24). However, when calcined ESP was added to the soil samples, the CBR results were similar to those of the unheated and oven-dried ESP-treated soil samples, revealing remarkable strength improvement even when an additional 3% moisture above the OMC was applied. The CBR results of samples without ESP at a moisture level of OMC+3% were over four times lower than the CBR results of samples stabilized with calcined ESP, while when calcined ESP was added to the Soil A samples, the CBR results were similar to those of unheated and oven-dried ESP-treated soil samples. In contrast, Soil B treated with calcined ESP showed remarkable strength improvement compared to unheated and oven-dried ESP-treated soil samples. This indicates that the role of calcined ESP could vary with soil type. Soils with high expansive silt and clay content, like Soil B, could attain more benefits when blended with calcined ESP. Based on the results of the UCS and CBR tests, the use of 6% calcined ESP with an OMC+3% moisture content can achieve high strength similar to that achieved by 8% and 10% calcined ESP but with a lesser amount. Therefore, 6% calcined ESP with an OMC+3% moisture content could be regarded as the optimum rate for soil stabilization.

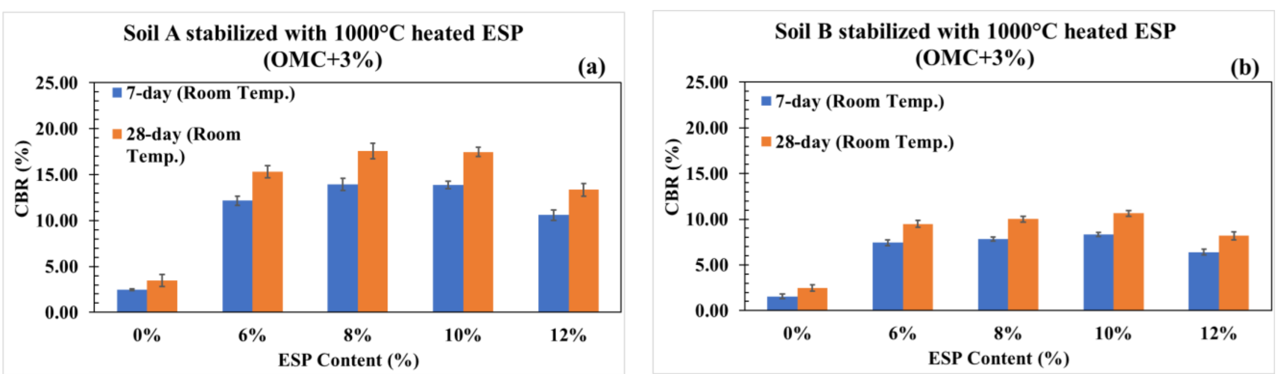


Figure 25. Results of CBR in soils treated with calcined ESP at OMC+3%

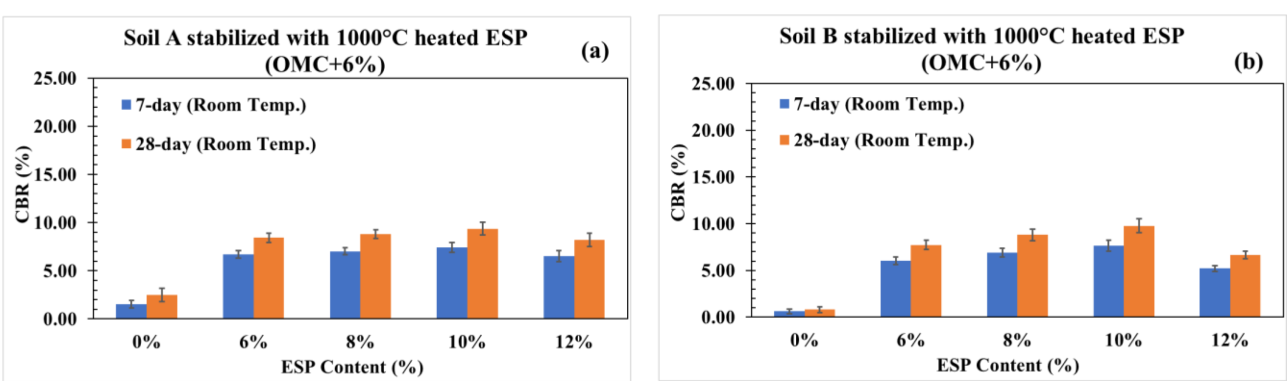


Figure 26. Results of CBR in soils treated with calcined ESP at OMC+6%

Microstructural Characterization

X-Ray Diffraction Analysis

XRD patterns were acquired using a Siemens D500 X-ray Powder Diffractometer with Cu- K radiation generated at 45 kV and 30 mA. The diffractometer is equipped with primary and secondary Soller slits, a diffracted beam monochromator (curved graphite crystal), and a point detector. Divergent and scattered beam diffractometry was conducted using 1° slits with 0.15° resolution detector slits. The samples were scanned from 5° to 60° (2θ) with a step size of 0.02° and a measuring time of 2 seconds per step.

The samples were dried at 105°C overnight. Samples were manually ground in an agate mortar and pestle and loaded into the well of a zero-background holder using the razor-tamped surface (RTS) method to minimize the preferred orientation. MDI JADE 9.5.0 software was used to plot the diffractograms and perform phase identification of the diffraction peaks within the data sets. Phase identification used the SEM-EDS elemental analysis results to find the minerals that matched the crystalline reflections based on the associations between elements.

The XRD analyses of calcined ESP (1,000°C) and of untreated and treated samples of Soil A and Soil B are discussed in this section. Figure 27 shows the XRD pattern of calcined ESP (1,000°C). The reflection peaks of calcined ESP (1,000°C) were assigned to lime (CaO) and portlandite ($Ca(OH)_2$). Apparently, the lime began to hydrate upon exposure to air.

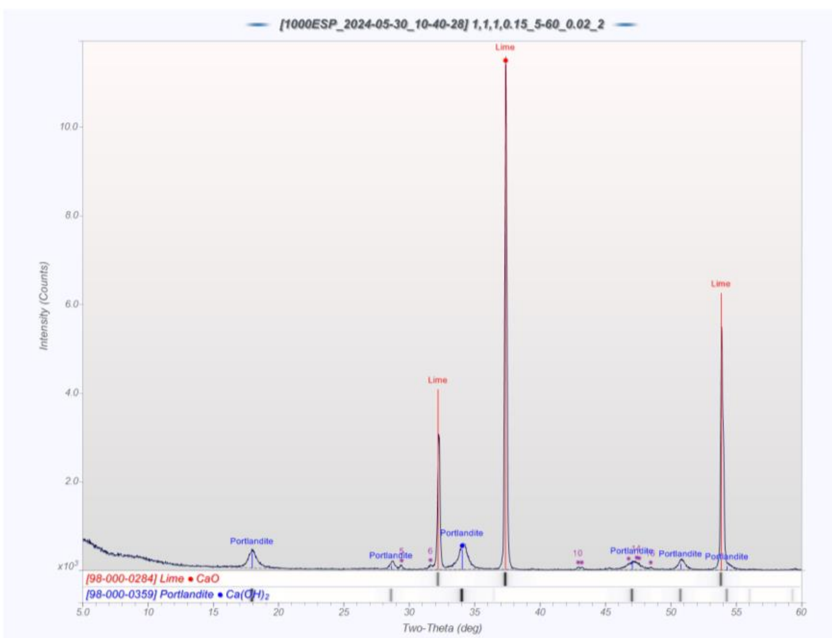


Figure 27. XRD results of calcined ESP (1,000°C)

Figure 28 shows the XRD results of untreated Soil A (silty soil). According to the results, Soil A is mainly comprised of the following major crystalline phases: quartz (SiO_2), albite ($NaAlSi_3O_8$), calcite ($CaCO_3$), and dolomite ($MgCa(CO_3)_2$). Note that lime is included in the chart but is not present in the sample. Its main peak at 37.3° is explained by a dolomite reflection.

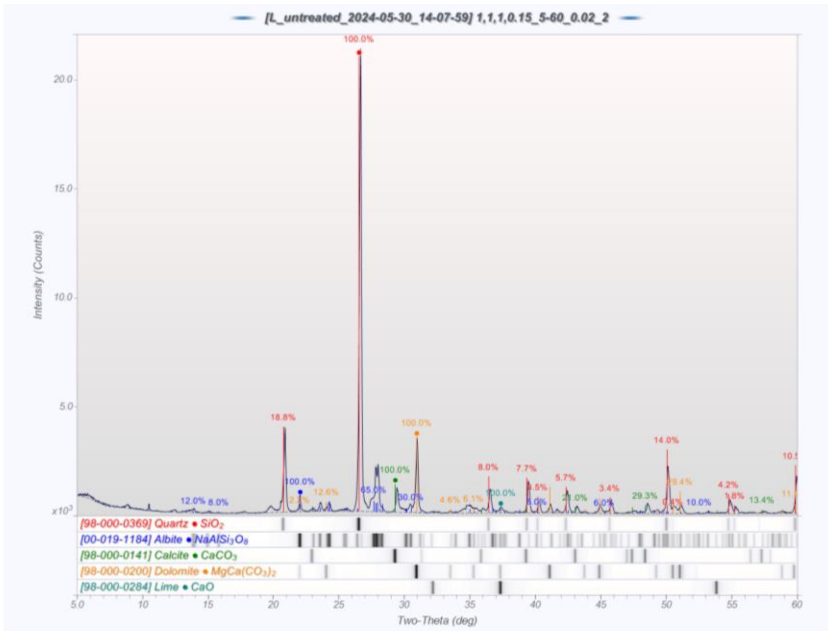


Figure 28. XRD results of untreated Soil A (silty soil)

Figure 29 shows the XRD results of treated Soil A (silty soil) with 8% calcined ESP (1,000°C) and 6% moisture content. The pattern is basically that of untreated Soil A plus lime (CaO) and portlandite ($Ca(OH)_2$) from the calcined ESP.

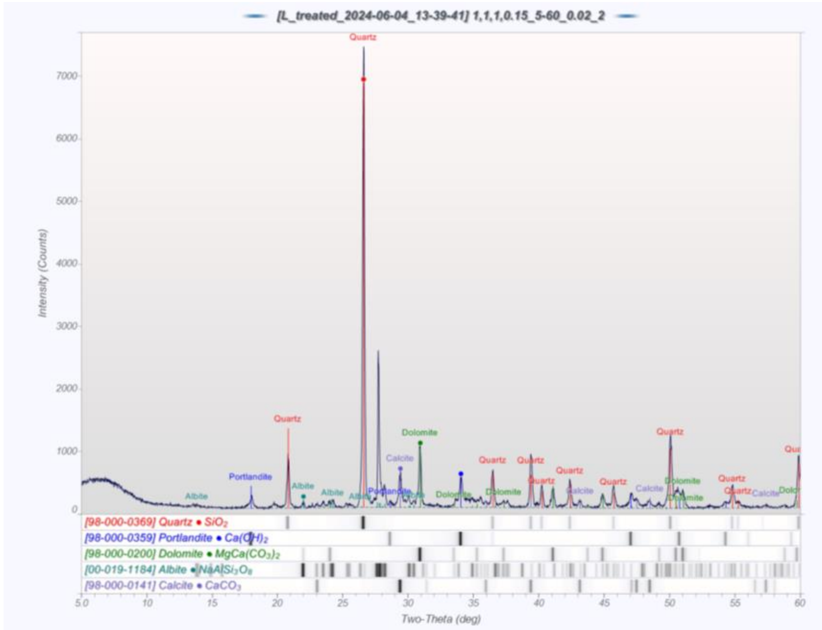


Figure 29. XRD results of treated Soil A (silty soil) with 8% calcined ESP (1,000°C) and 6% moisture content

Figure 30 shows a comparison of the XRD results of untreated Soil A (silty soil) and treated Soil A (silty soil) with 8% calcined ESP (1,000°C) and 6% moisture content. The figure highlights that the only difference between the samples is the addition of portlandite to the pattern.

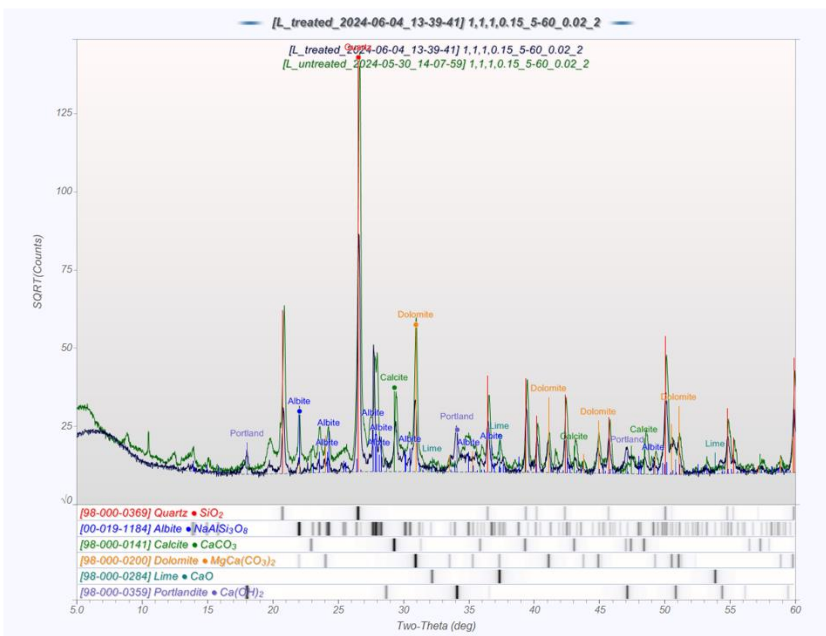


Figure 30. Comparison of XRD results of untreated Soil A (silty soil) and treated Soil A (silty soil) with 8% calcined ESP (1,000°C) and 6% moisture content

Figure 31 shows the XRD results of untreated Soil B (clayey soil). According to the results, Soil B is mainly comprised of quartz (SiO_2), calcite ($CaCO_3$), and albite ($NaAlSi_3O_8$), similar to Soil A, which is a typical mineralogy of soils. However, according to AASHTO, Soil A is classified as A-4 and contains a small amount of clay. A-4 soils are characterized as having a higher percentage of silt compared to clay.

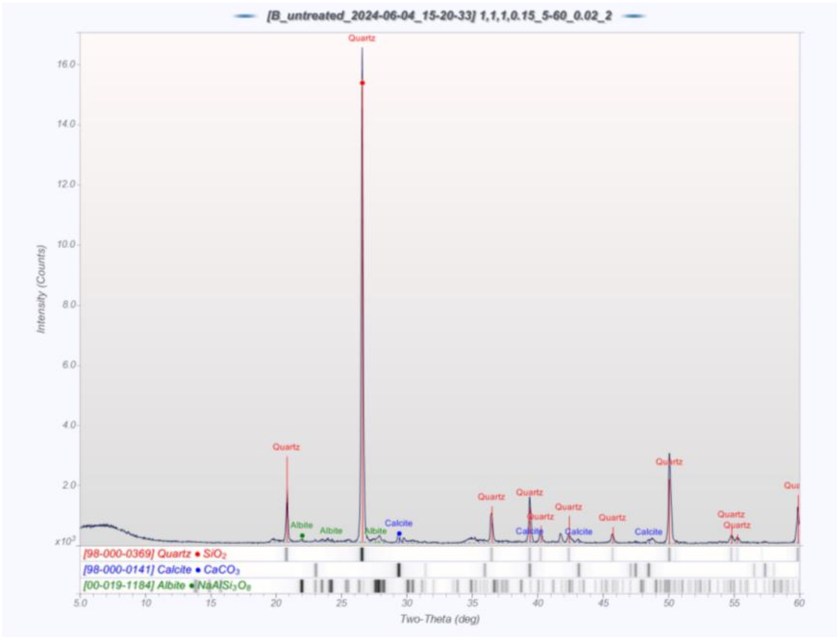


Figure 31. XRD results of untreated Soil B (clayey soil)

Figure 32 shows the XRD results of treated Soil B (clayey soil) with 8% calcined ESP (1,000°C) and 6% moisture content. Quartz (SiO_2), albite ($NaAlSi_3O_8$), and portlandite ($Ca(OH)_2$) are the major crystalline phases present in this sample. Lime (CaO) from the calcined ESP (1,000°C) is evident in the structure of treated Soil B.

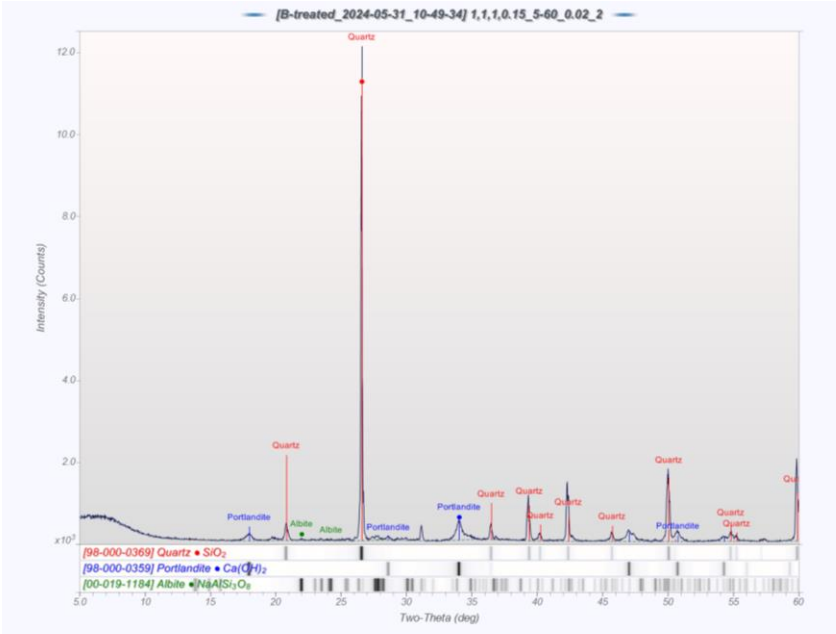


Figure 32. XRD results of treated Soil B (clayey soil) with 8% calcined ESP (1,000°C) and 6% moisture content

Figure 33 shows a comparison of the XRD results of untreated Soil B (clayey soil) and treated Soil B (clayey soil) with 8% calcined ESP (1,000°C) and 6% moisture content. The findings indicate that lime from the calcined ESP reacts with moisture in the soil mixture, resulting in the formation of hydration products, specifically portlandite ($Ca(OH)_2$). The XRD results confirm that the improved soil strength resulting from calcined ESP treatment is mainly attributed to chemical reactions like hydration.

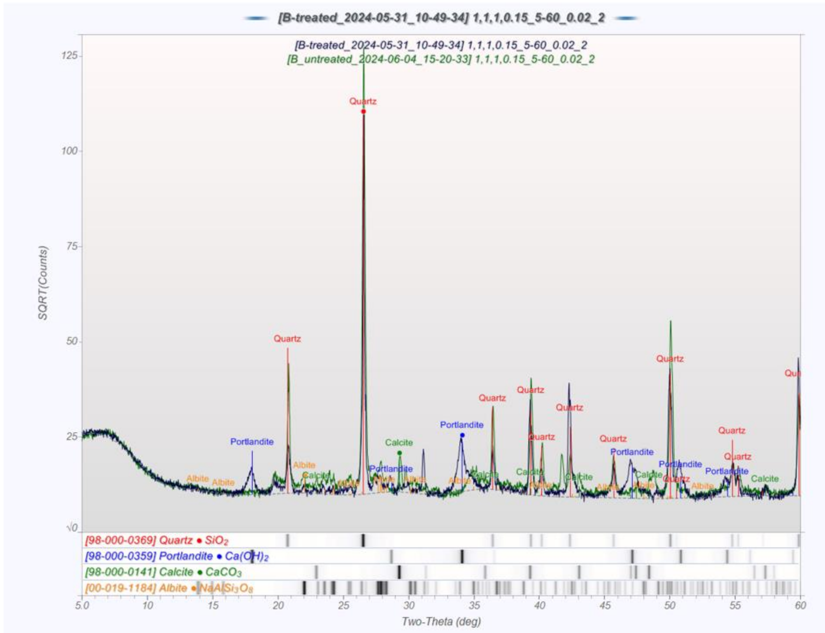


Figure 33. Comparison of XRD results of untreated Soil B (clayey soil) and treated Soil B (clayey soil) with 8% calcined ESP (1,000°C) and 6% moisture content

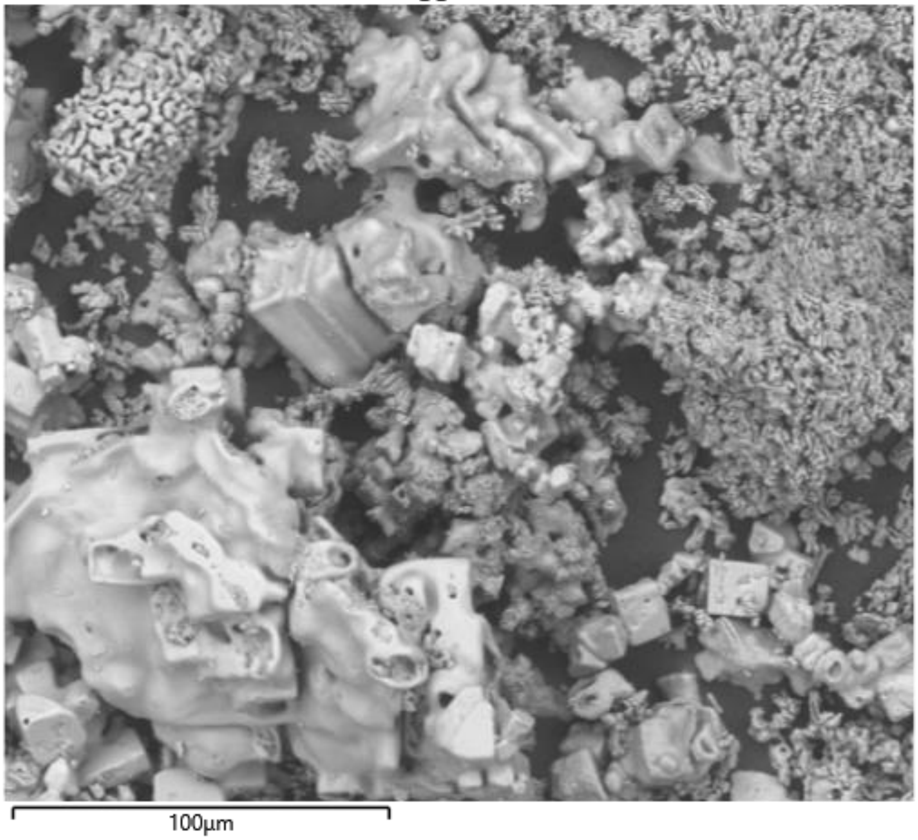
Scanning Electron Microscopy and Energy-Dispersive X-Ray Spectrometer

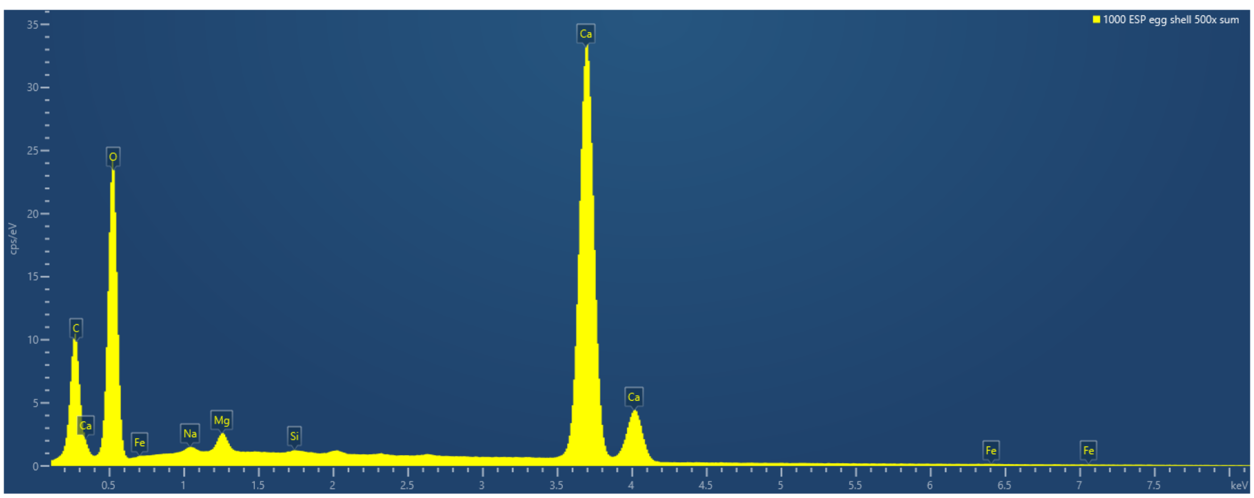
Samples were examined using a FEI Quanta-FEG 250 scanning electron microscope equipped with an Oxford Instruments Aztec energy-dispersive spectrometer for elemental analysis. Its X-Max 80 detector can analyze carbon and heavier elements.

Images were collected at magnifications from 60x to 5000x. Selected areas were mapped to determine elemental distribution. Conditions were set to collect x-rays at 10 to 15 kcps. Maps were collected for 10 minutes at a resolution of 256 pixels across the map.

The SEM was used to document the morphology of the two soils both before and after stabilization with calcined ESP (1,000°C). In this analysis, the changes at 8% calcined ESP (1,000°C) and 6% moisture content, where the highest strength values were obtained, were considered. Figures 34 through 38 show SEM images of calcined ESP (1,000°C), untreated Soil A, treated Soil A with 8% calcined ESP (1,000°C) and 6% moisture content, untreated Soil B, and treated Soil B with 8% calcined ESP (1,000°C) and 6% moisture content, respectively.

1000 ESP egg shell 500x bse





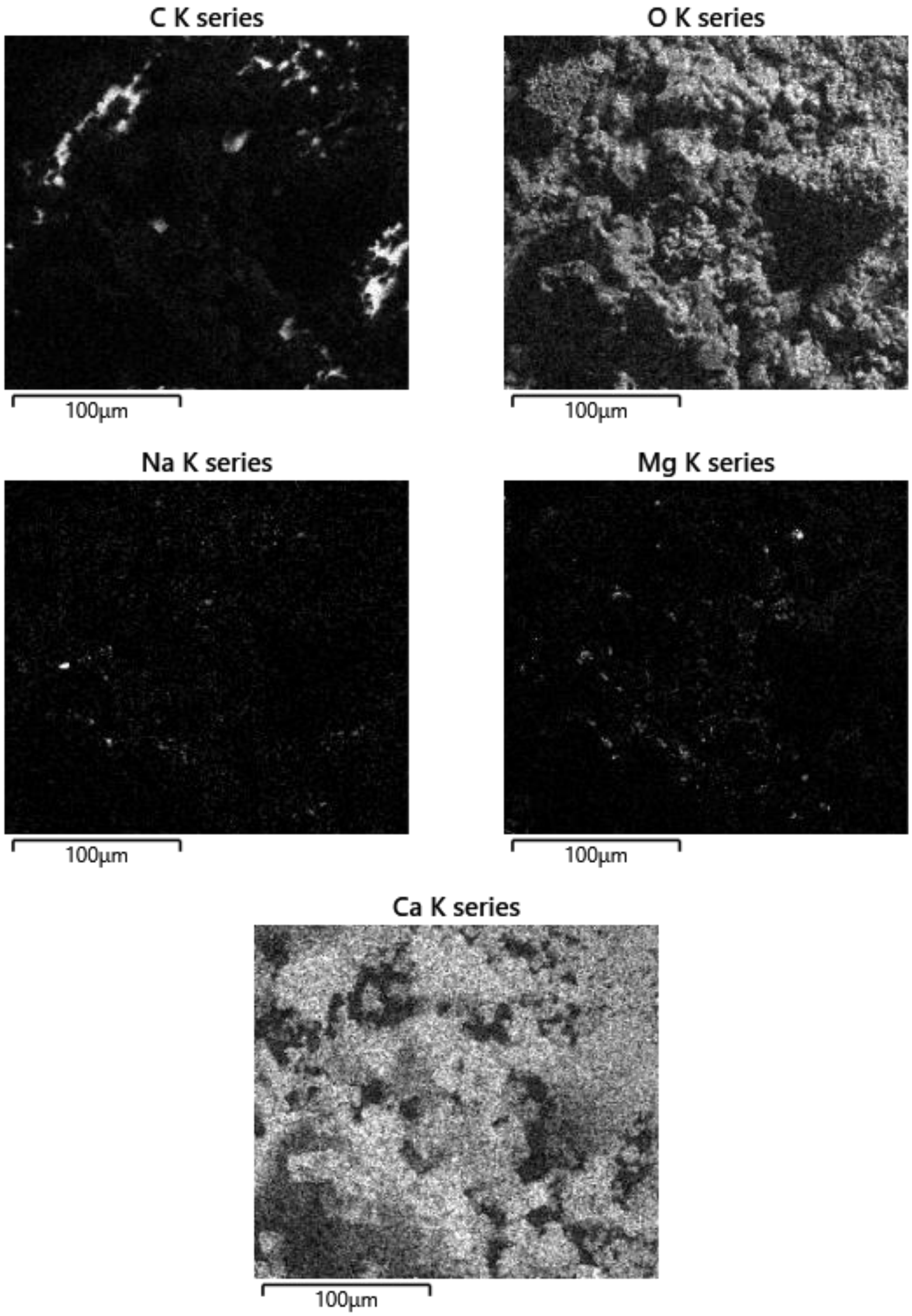
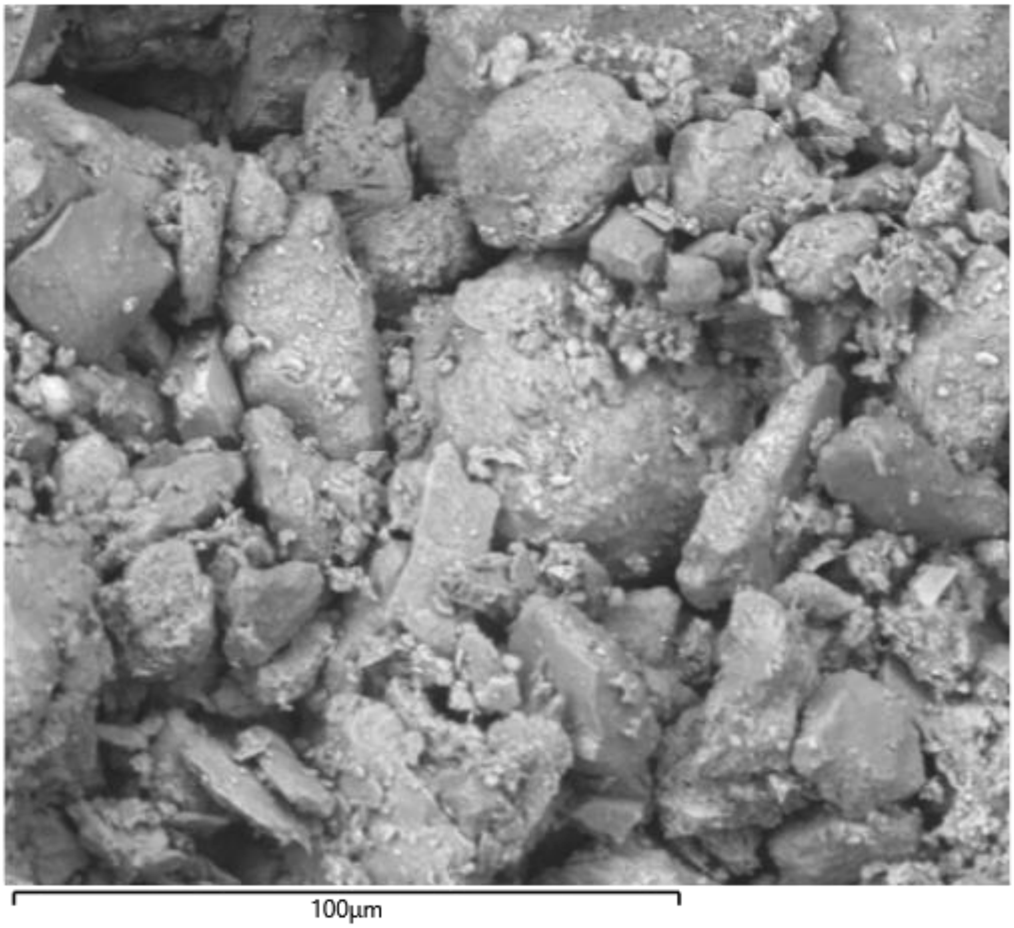
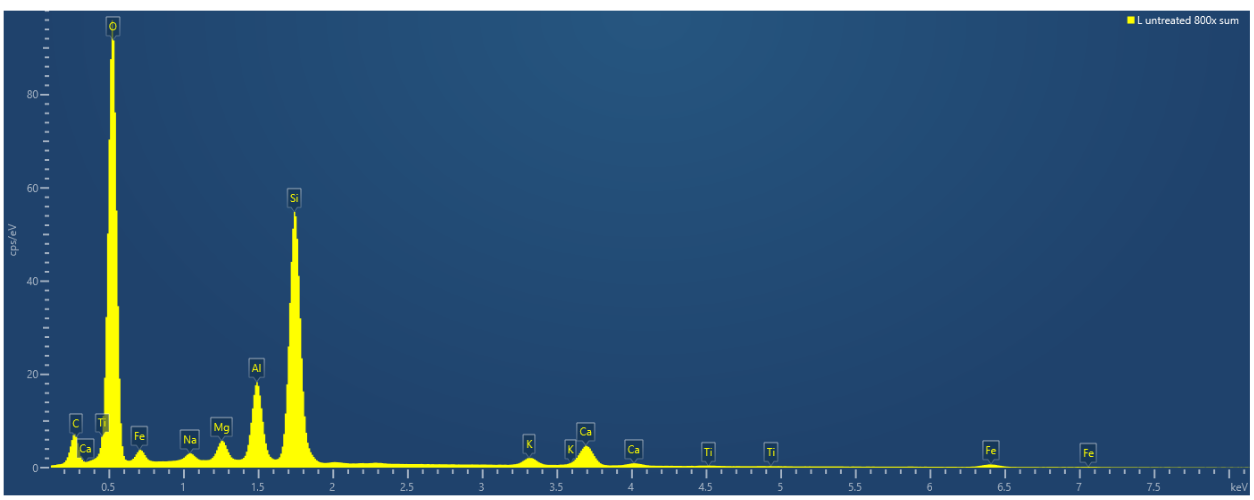
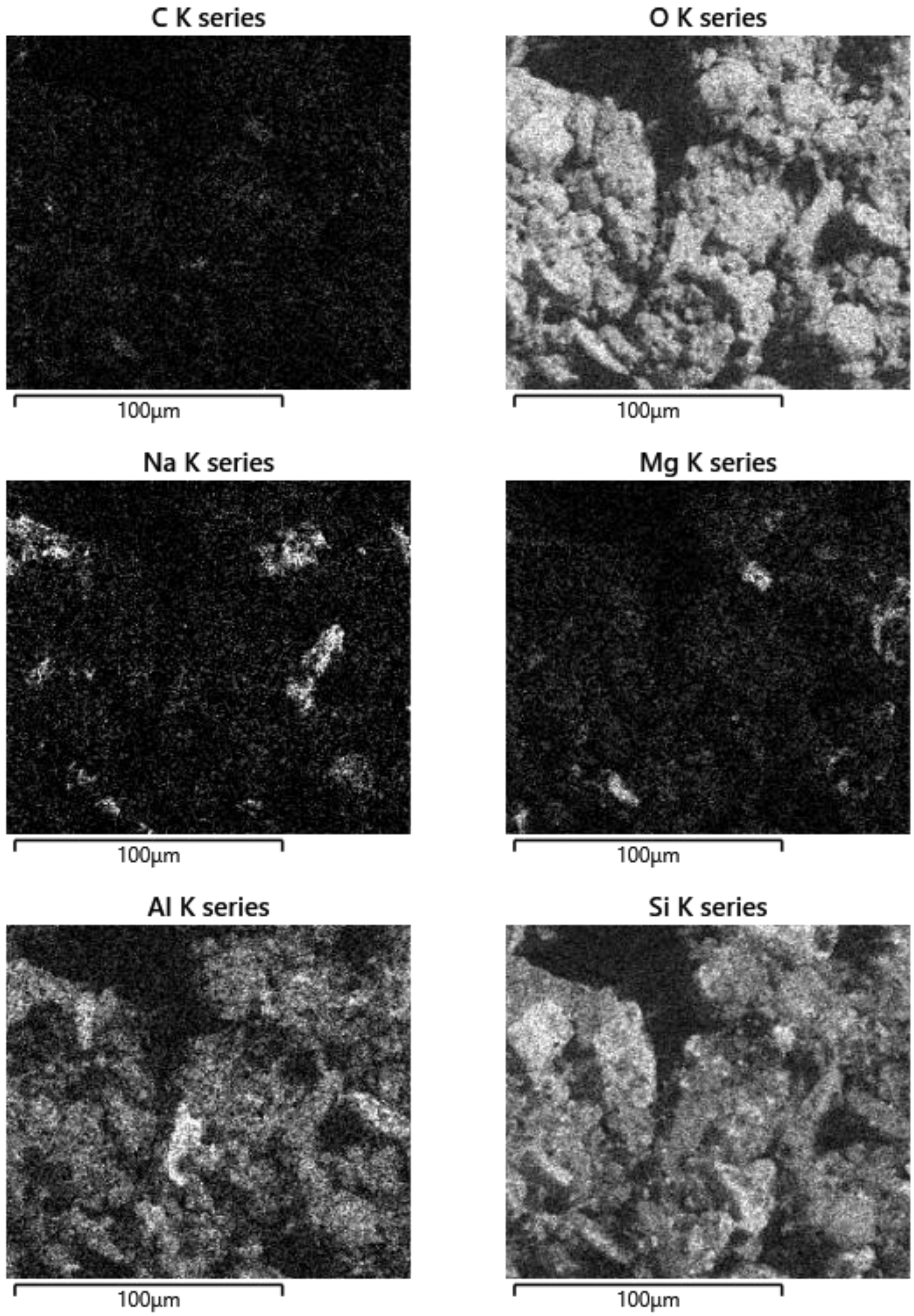


Figure 34. SEM images of calcined ESP (1,000°C)

L untreated 800x bse



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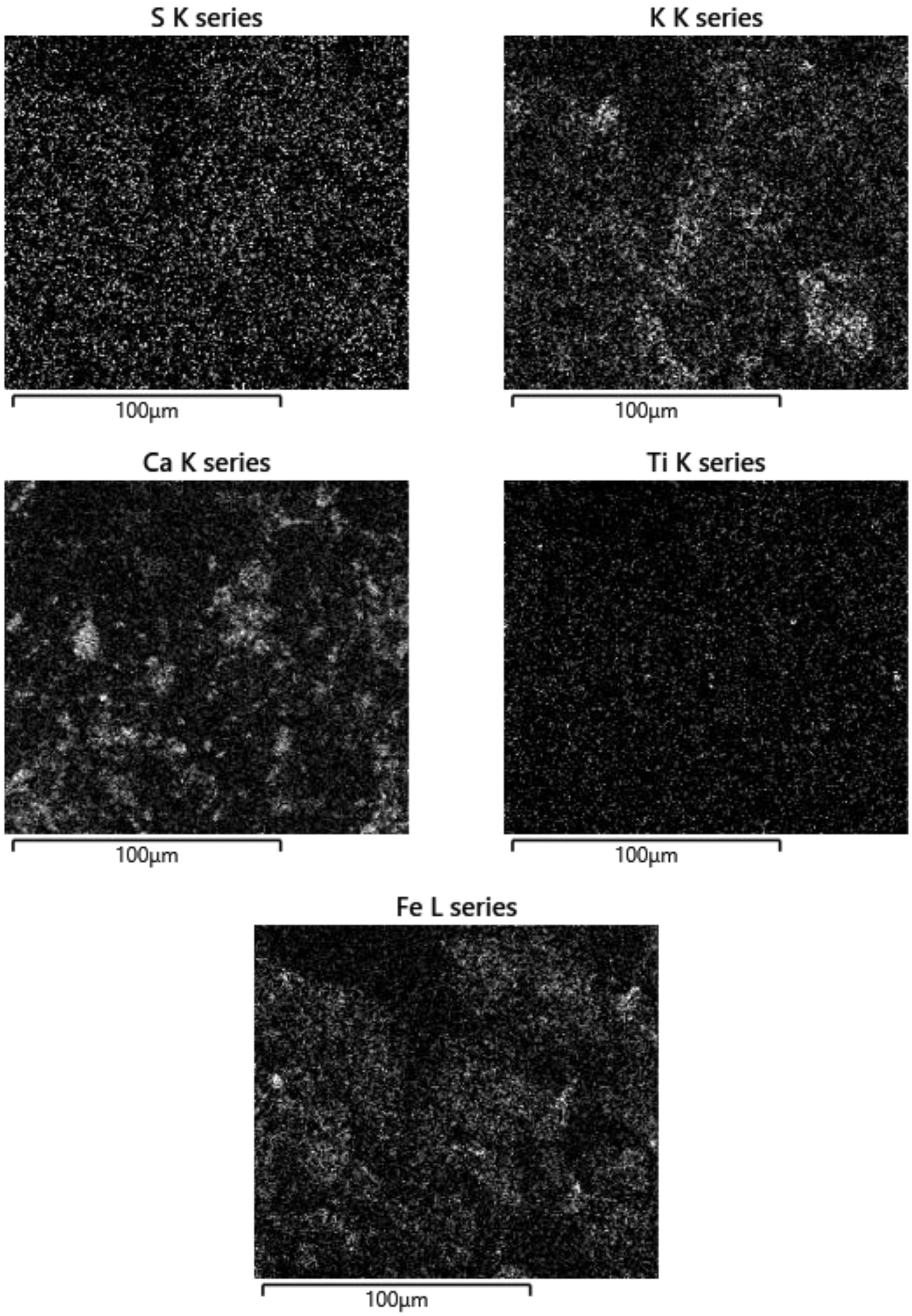
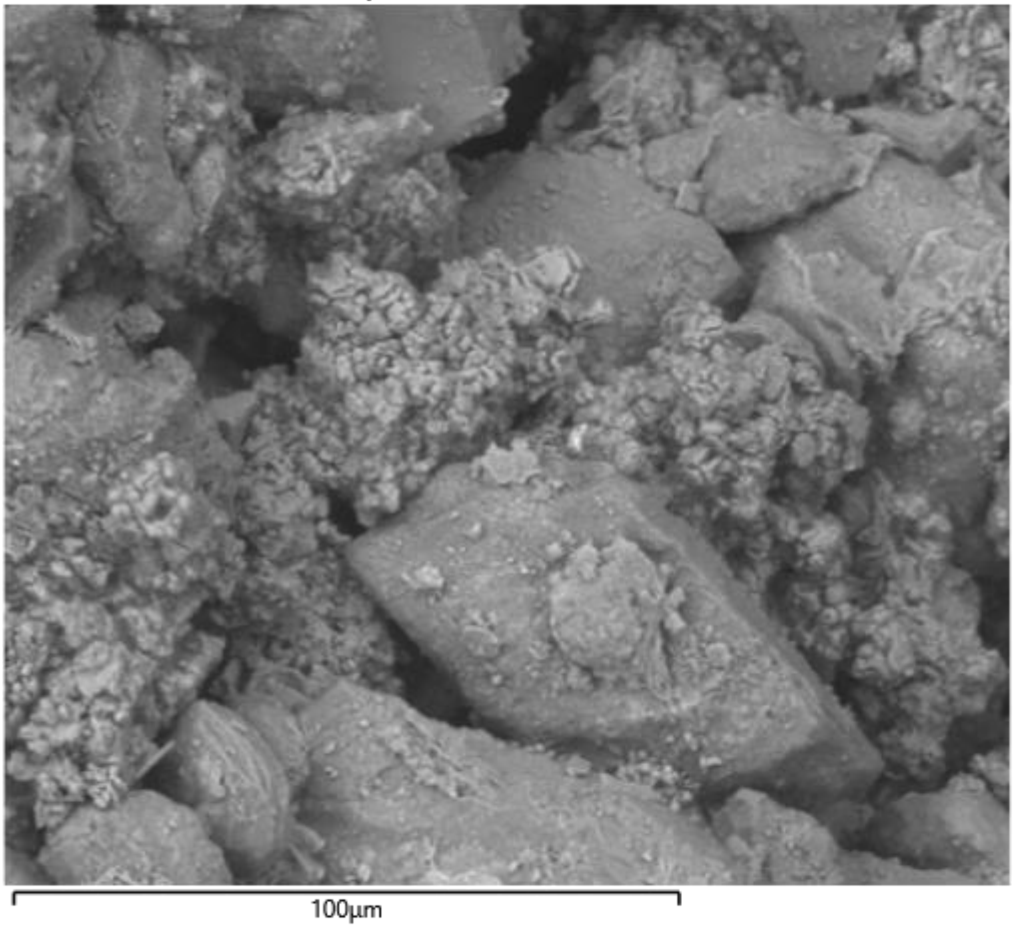
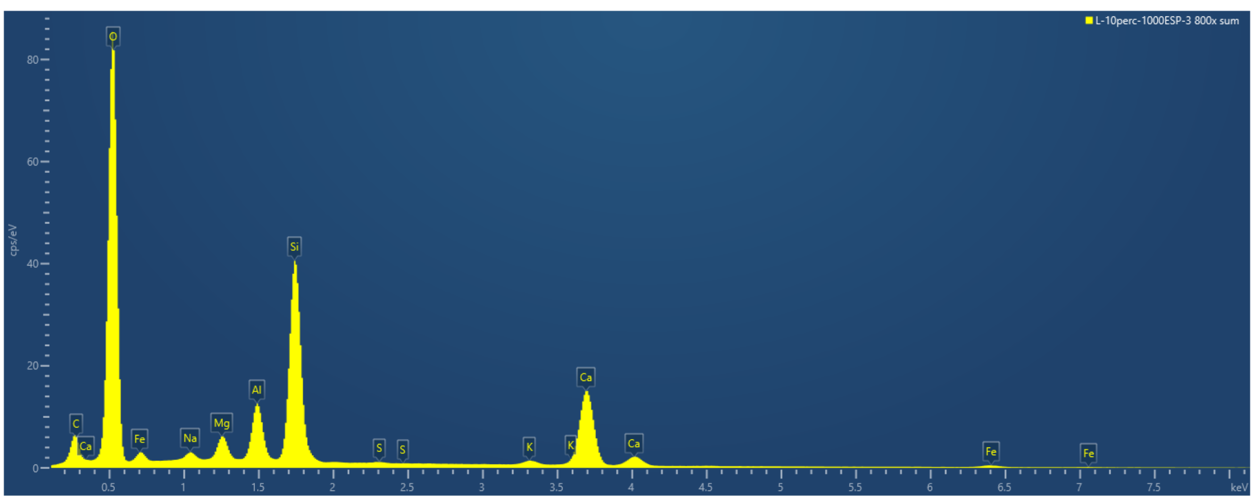
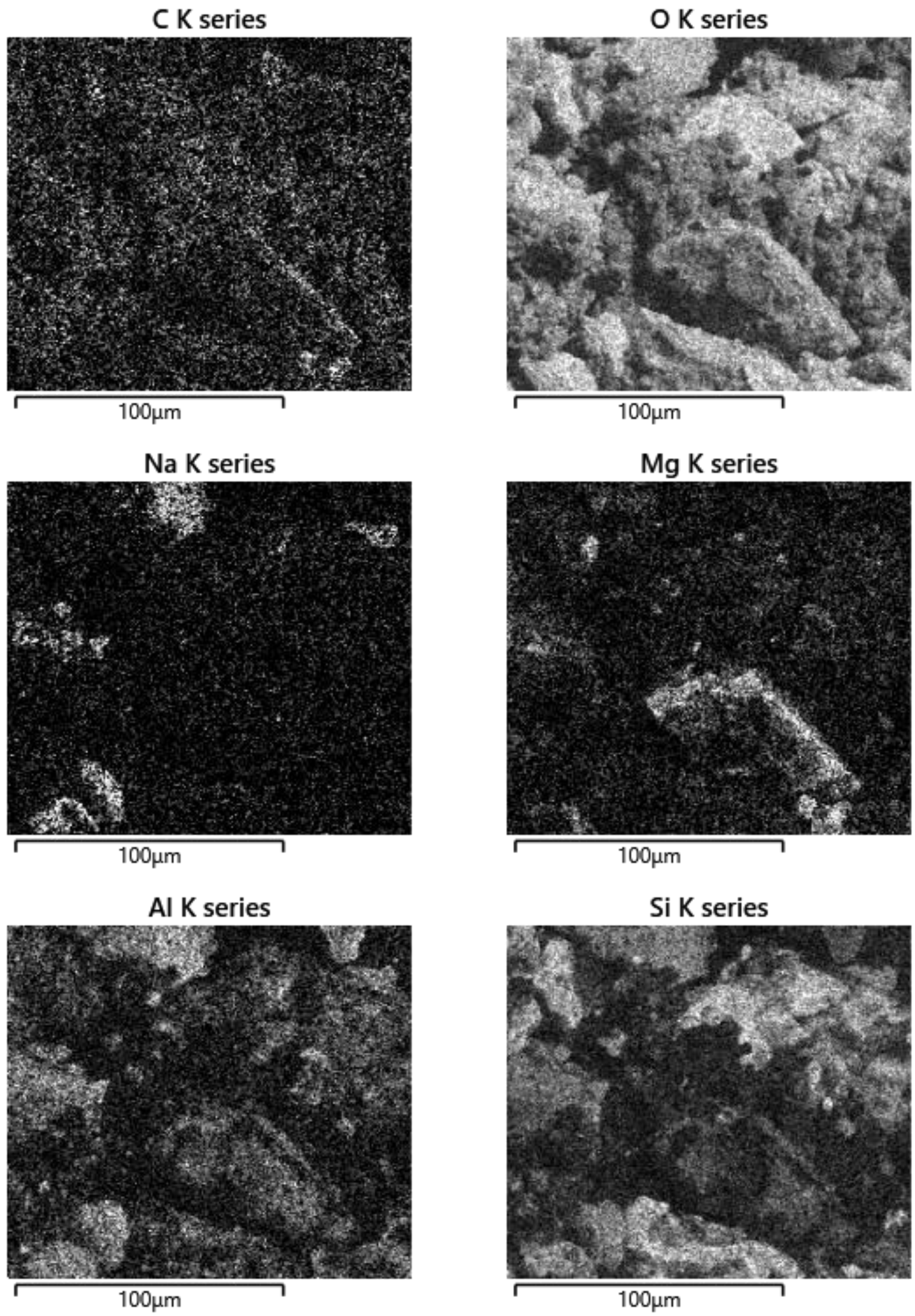


Figure 35. SEM images of untreated Soil A

L-10perc-1000ESP-3 800x bse



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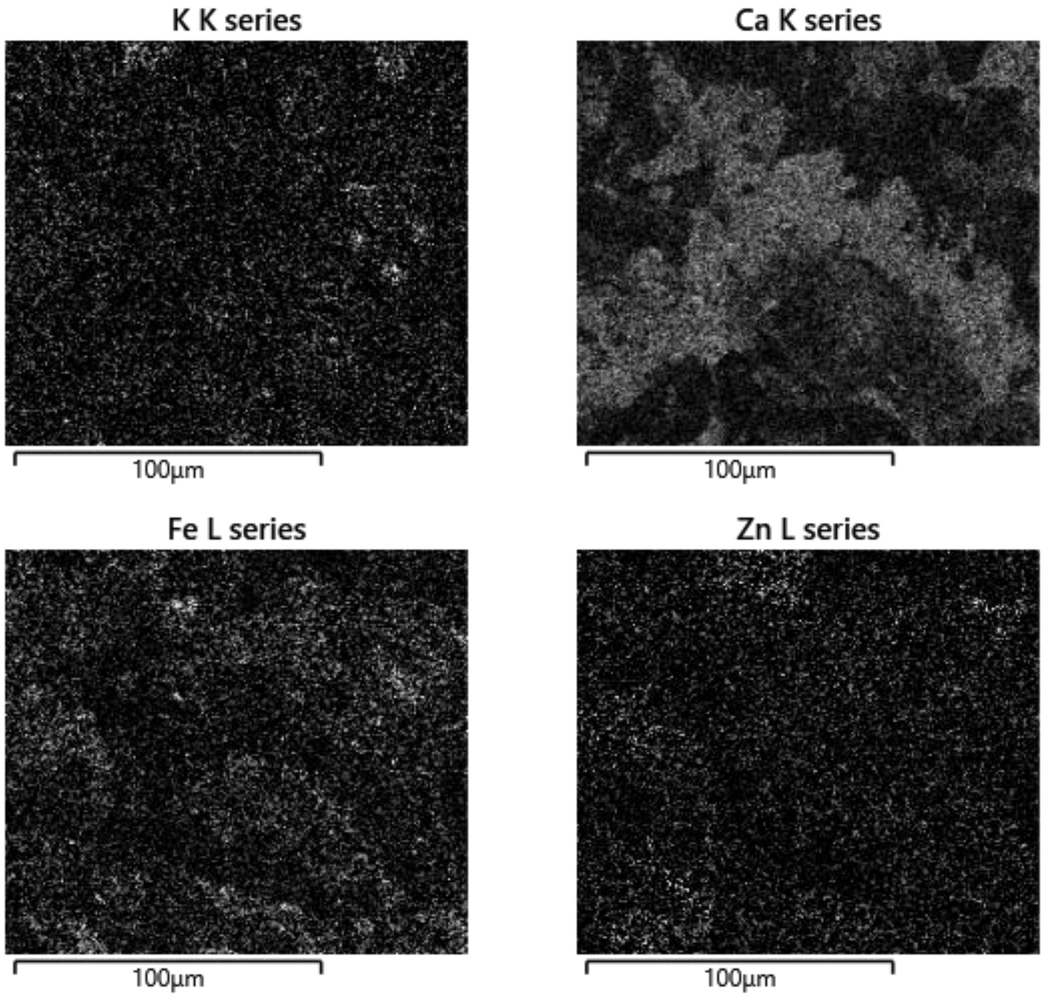
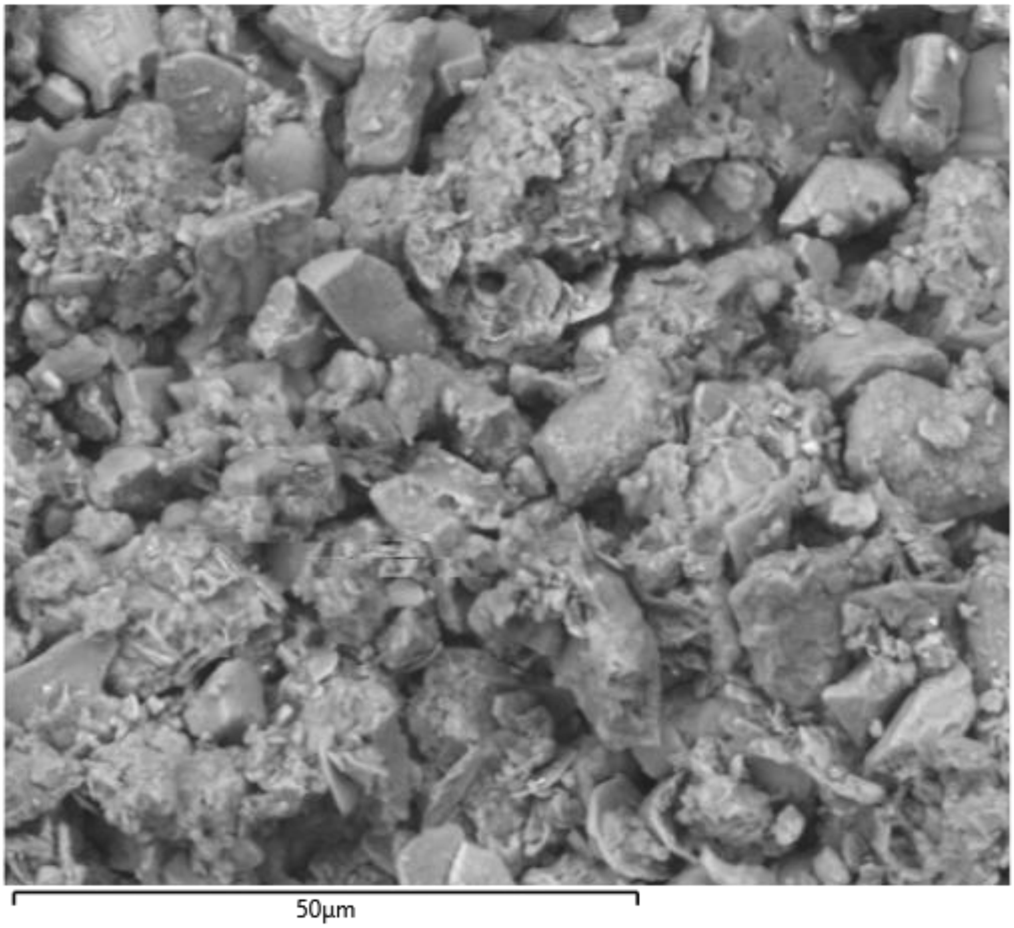
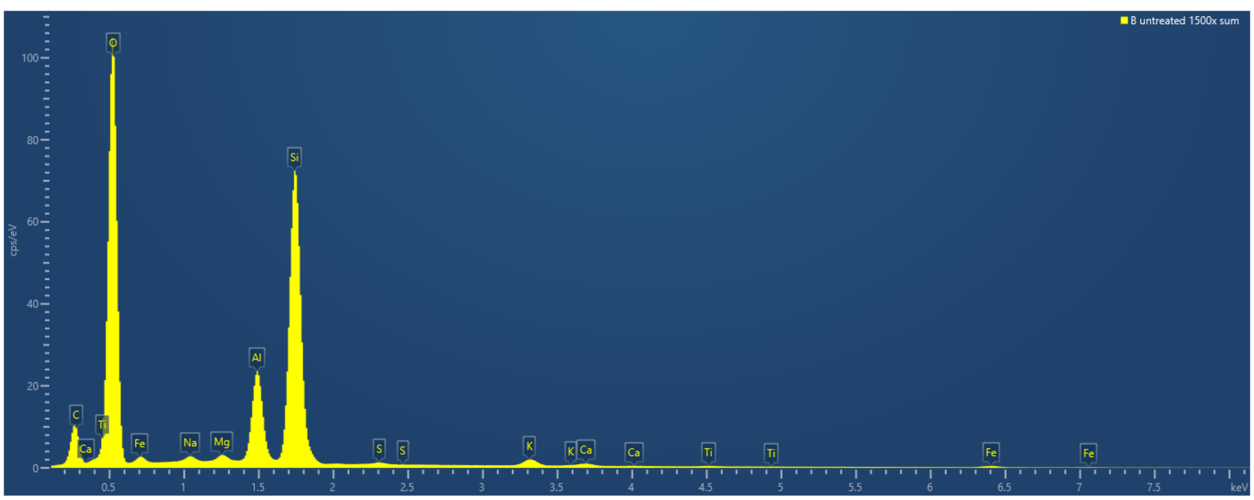
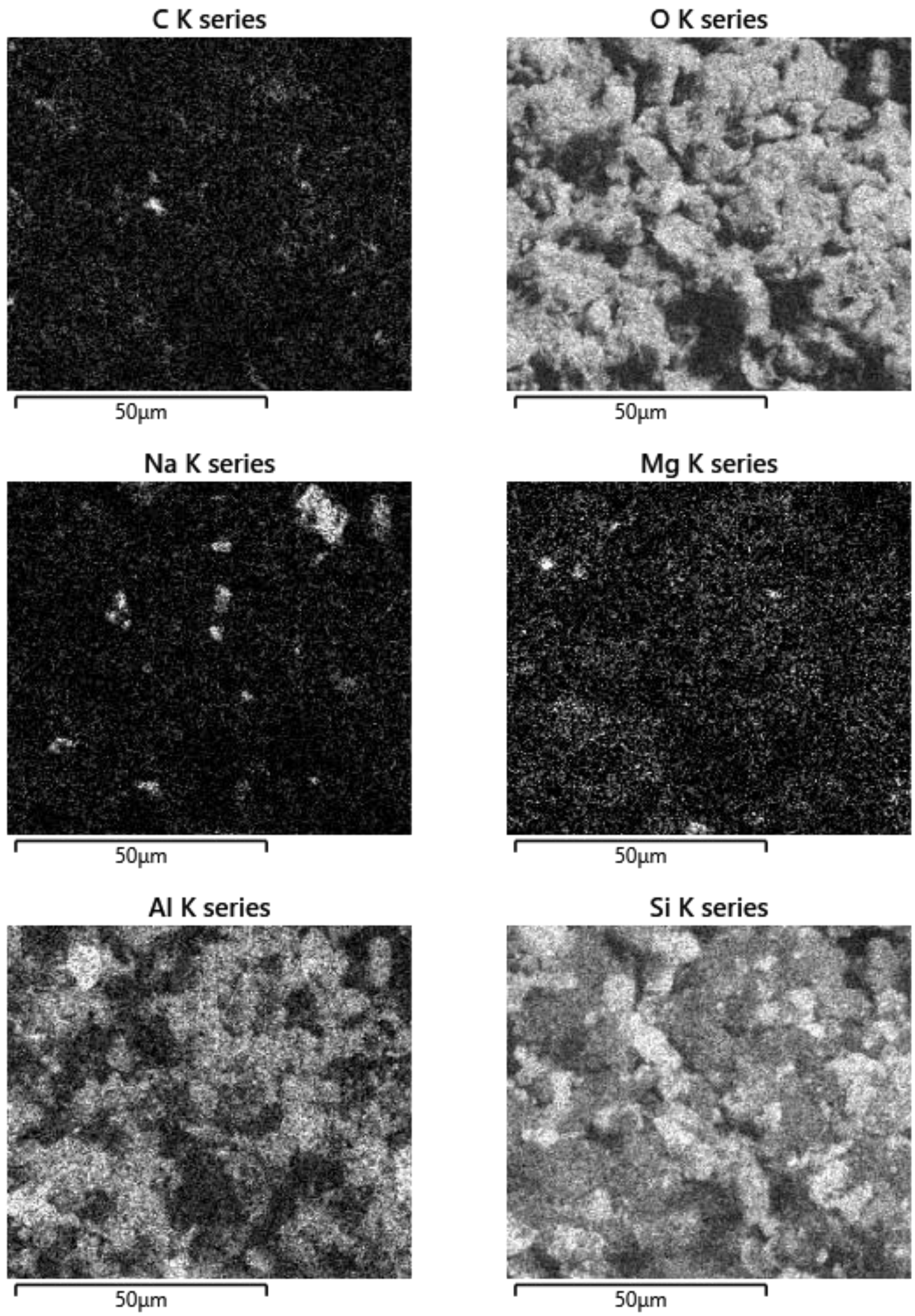


Figure 36. SEM images of treated Soil A with 8% calcined ESP (1,000°C) and 6% moisture content

B untreated 1500x bse



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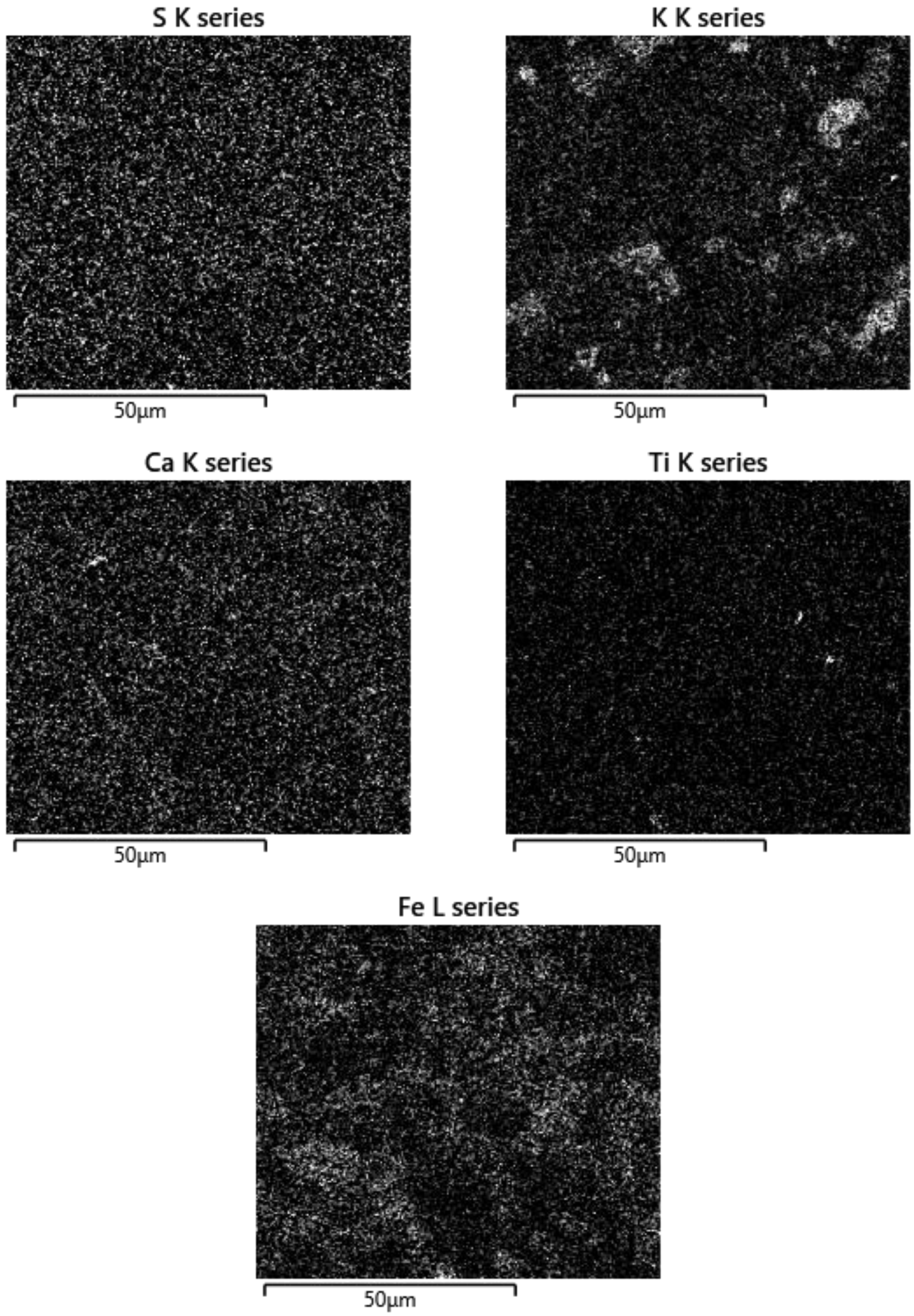
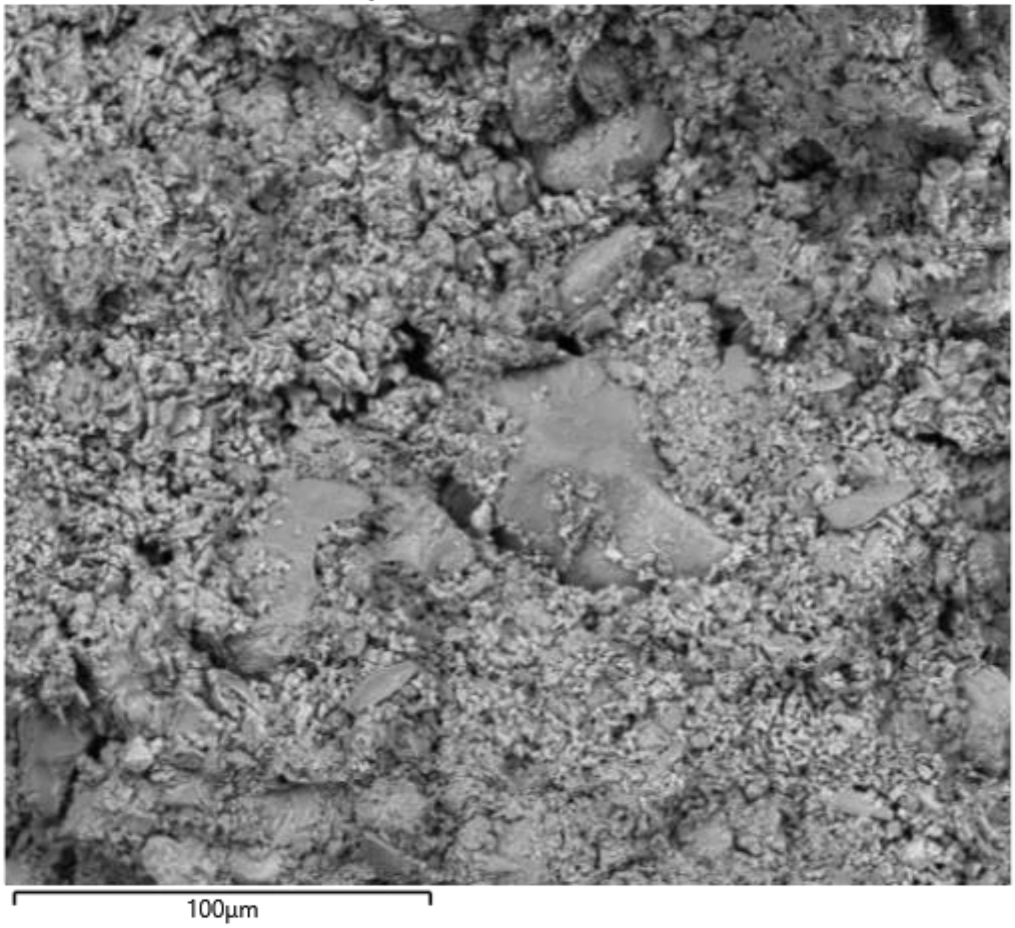
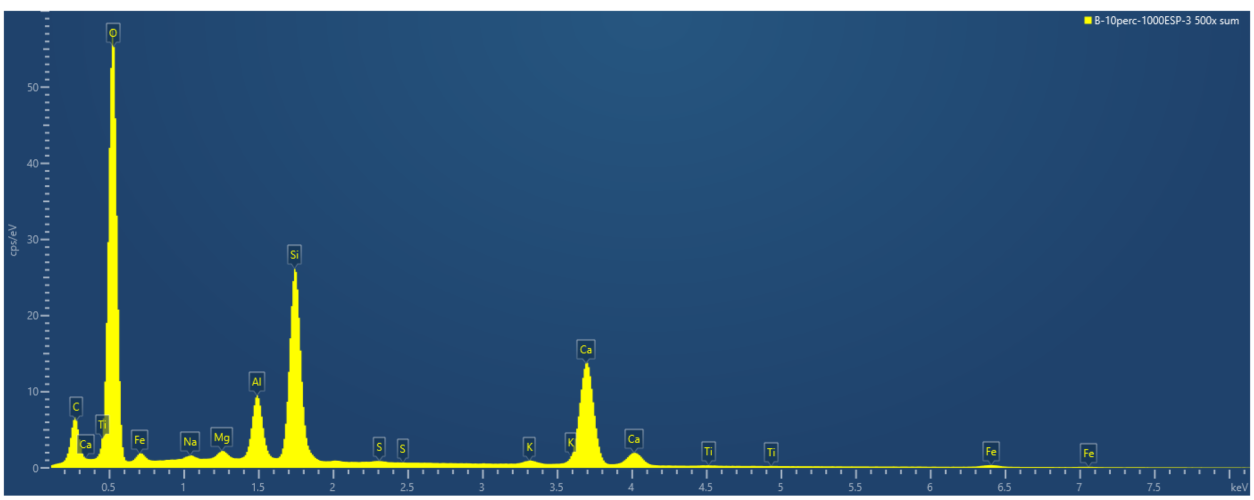
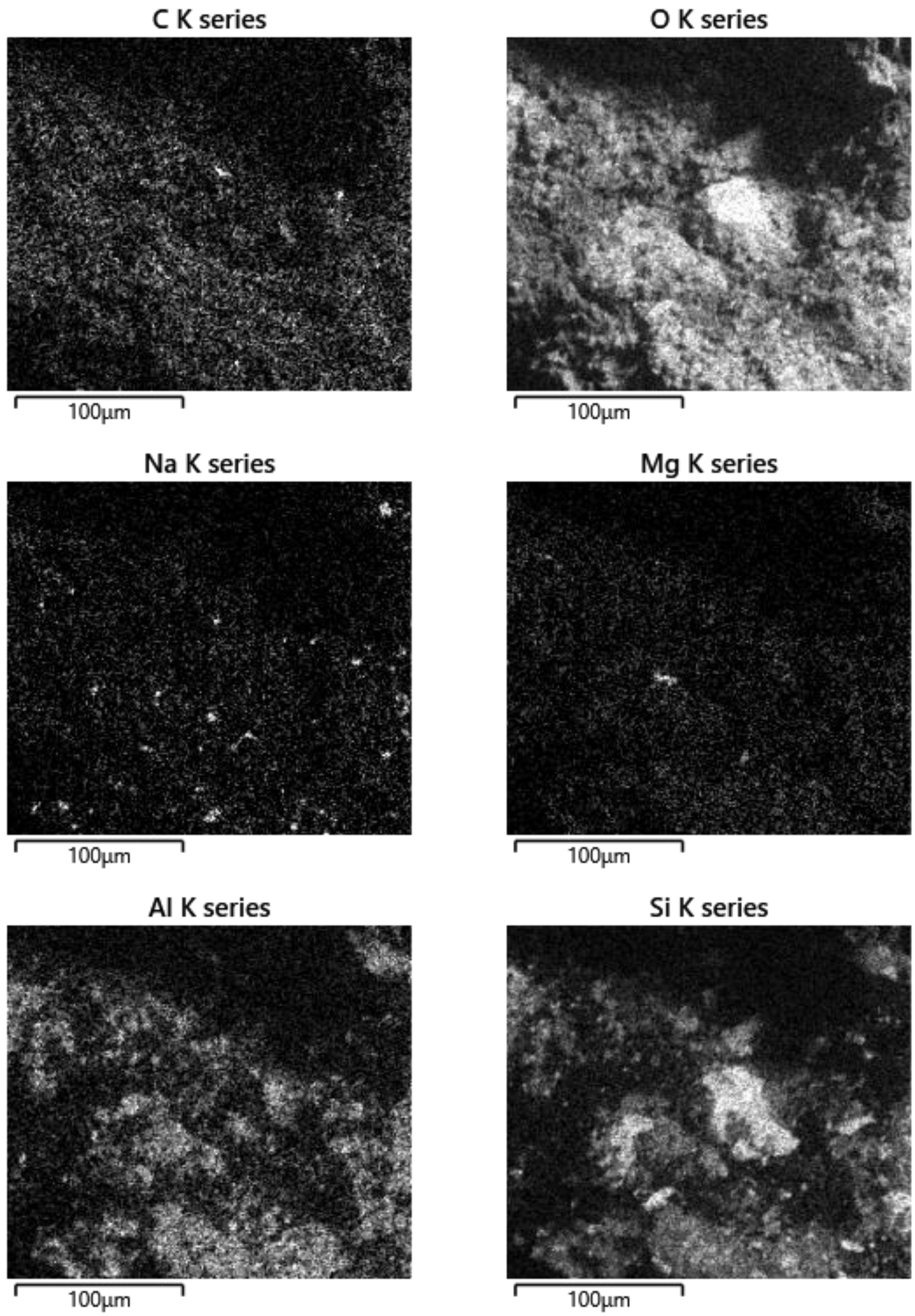


Figure 37. SEM images of untreated Soil B

B-10perc-1000ESP-3 500x bse



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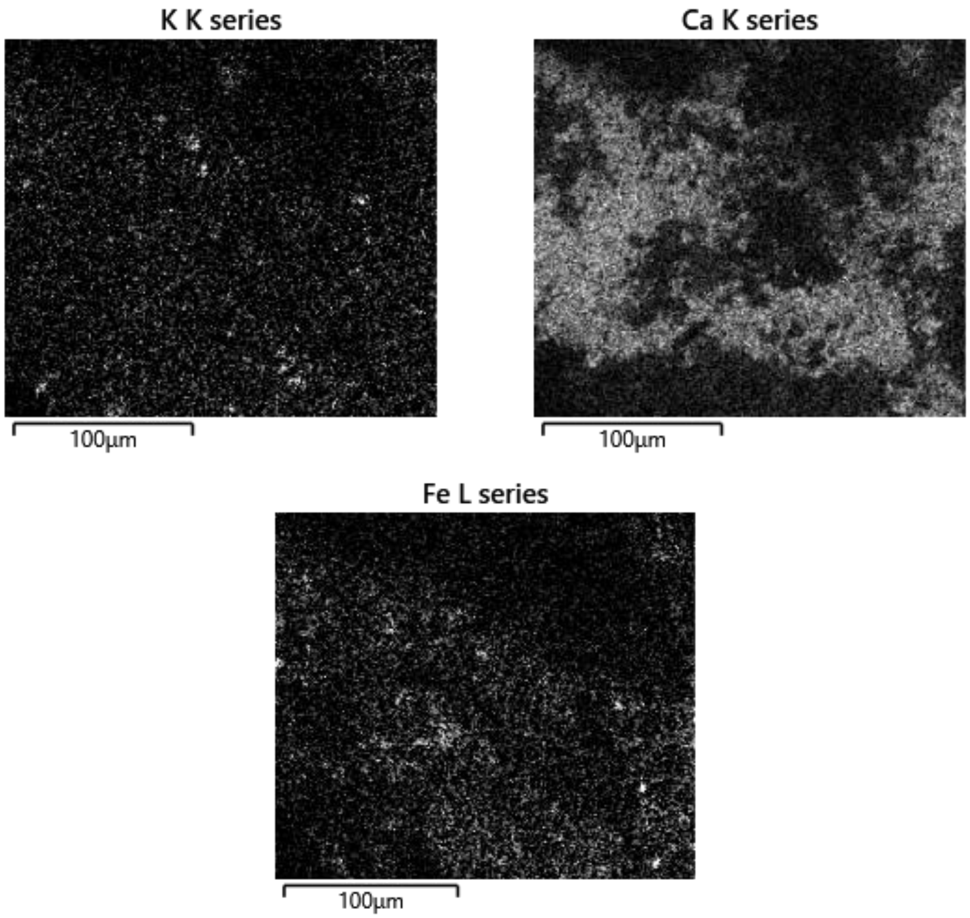


Figure 38. SEM images of treated Soil B with 8% calcined ESP (1,000°C) and 6% moisture content

Figure 34 shows that the microstructure of the calcined ESP (1,000°C) is mainly comprised of Ca and O. This is consistent with the XRD analysis. The spectra indicate that lime and portlandite have different levels of O, suggesting that they represent two distinct phases due to their differing morphologies. The Na and Mg contents are from trace phases. The C content is from exposed carbon tape. The calcined ESP (1,000°C) particles appear to have two different morphologies, one markedly finer than the other.

From the SEM images of untreated Soils A and B in Figures 35 and 37, it is evident that the structures of both soils feature a large amount of space between particles. As in the XRD analysis results, it can be observed that Si, O, Al, Mg, Na, and Ca are the main components of the soils. According to the XRD and SEM analyses, quartz (high in Si), albite (with Na), K-feldspar (with K, not identified by XRD), calcite (Ca and O), and dolomite (Ca+Mg and O) are observed in untreated Soils A and B, although the particle sizes of these components are somewhat different. Soil B has a larger number of fine particles than Soil A.

The SEM images of treated Soil A (silty soil) with 8% calcined ESP (1,000°C) exhibit a dispersed structure with different sizes of particles (Figure 36). Treated Soil A has similar phases to untreated Soil A except for a significantly higher amount of Ca. The SEM images of treated Soil B exhibit the same structure as the images of untreated Soil B except for significantly widespread areas of Ca between the grains (Figures 36 and 38).

According to the results, when calcined ESP (1,000°C) is added to the soils, the effects of calcined ESP (1,000°C) are visible in the microstructure of the treated soil samples (Figure 38), as evidenced by the presence of portlandite in the untreated soil structure. Portlandite fills the intergranular spaces of the untreated soil structure. Figures 39 and 40 show a comparison of the four Ca and Si maps (as dominant elements in the SEM analysis) in untreated Soils A and B and treated Soils A and B with 8% calcined ESP (1,000°C) and 6% moisture content, respectively.

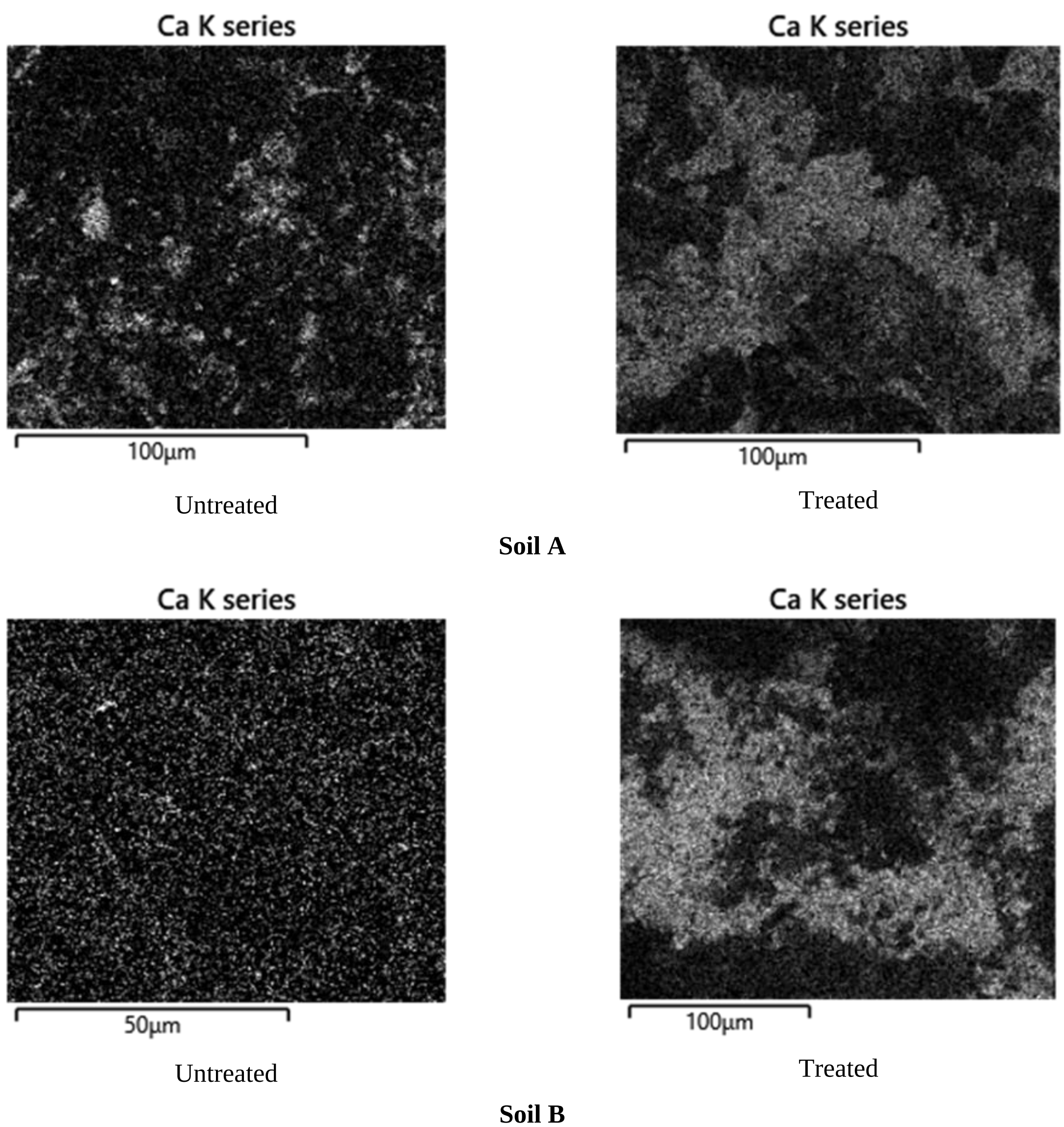
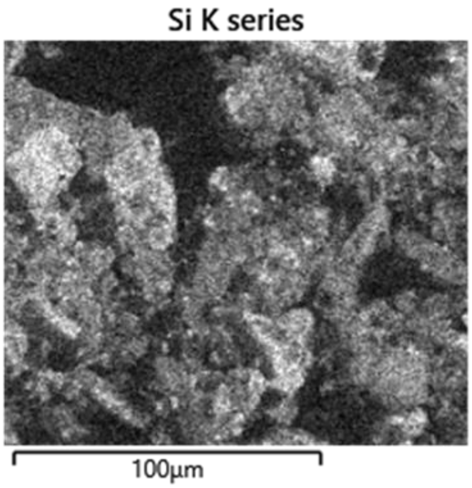
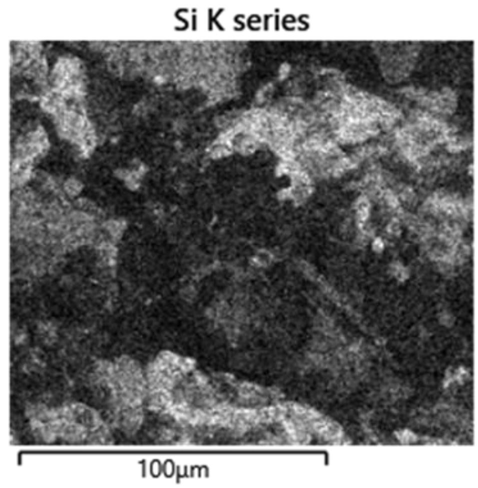


Figure 39. Comparison of the four Ca maps for untreated Soils A and B and treated Soils A and B with 8% calcined ESP (1,000°C) and 6% moisture content

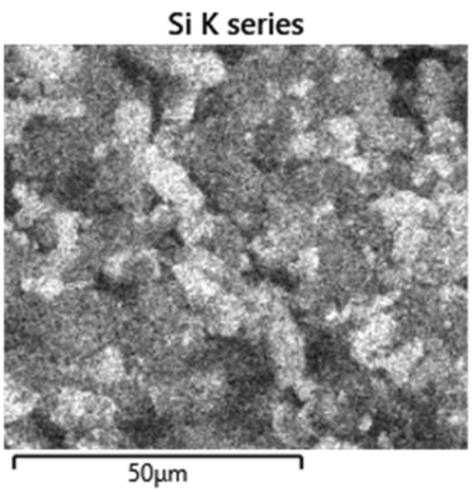


Untreated

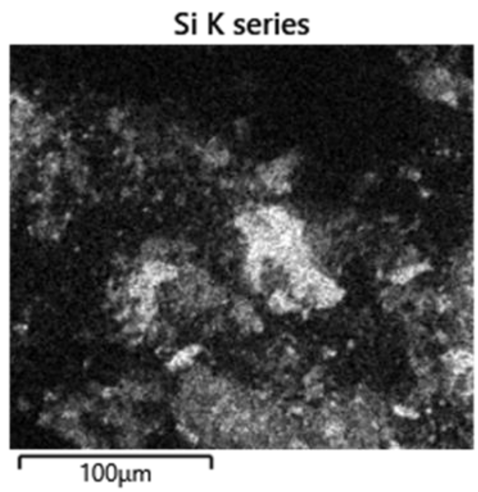


Treated

Soil A



Untreated



Treated

Soil B

Figure 40. Comparison of the four Si maps for untreated Soils A and B and treated Soils A and B with 8% calcined ESP (1,000°C) and 6% moisture content

In summary, the SEM images demonstrate that the voids evident in the untreated soils are filled by newly formed chemical reaction products in the treated soils, resulting in a denser and more solid soil structure. The SEM images reveal that the treated soils exhibit reduced particle spacing and increased calcium distribution, confirming the stabilizing effects of calcined ESP. These findings indicate that calcined ESP is an effective and sustainable soil stabilizer that can enhance the strength and structural integrity of soil.

CHAPTER 5: PROOF-OF-CONCEPT DEMONSTRATION

Proposed Eggshell Processing Method for Field Application

This study took a significant step forward from traditional laboratory evaluations of the use of eggshells for soil stabilization by conducting an innovative field simulation; this proof-of-concept demonstration aimed to translate the laboratory findings from this study into practical, scalable applications. Recognizing the environmental and health concerns as well as the high costs associated with the use of bleaching solutions in eggshell processing, this study proposed a revised methodology tailored for large-scale field applications:

1. **Washing and drying**. Initially, raw eggshell materials are thoroughly washed and dried to remove any contaminants and excess moisture.
2. **Calcination**. The eggshells are then subjected to calcination at 1,000°C for a minimum of five hours in a large-scale furnace. This step is crucial for burning off organic matter, resulting in purified eggshell flakes predominantly composed of calcium oxide (CaO).
3. **Grinding**. The calcined eggshell materials are subsequently ground using a large ball mill machine to produce calcined ESP.

By omitting the bleaching step typically employed in laboratory investigations, this approach not only reduces potential environmental and health risks but also cuts costs significantly. The calcination process efficiently eliminates eggshell residues and membranes, yielding purified eggshell flakes ready for grinding into calcined ESP.

Method for Proof-of-Concept Demonstration

The practical applicability of this method was tested through a field simulation involving the following:

- **Soil-ESP mixture preparation**. A 6 in. by 10 in. steel mold, as shown in Figure 41, was filled with a mixture of soil and calcined ESP, then compacted using standard compaction effort to ensure uniform density and structure.
- **Strength testing**. The compacted soil-ESP mixture was then subjected to dynamic cone penetration (DCP) testing to measure its CBR, a key indicator of the material's bearing capacity and strength.

Figure 41. Mold for simulating field conditions

This simulation exclusively used calcined ESP, which was identified in the laboratory study as providing superior strength enhancement properties, at an optimal inclusion rate of 6% and a moisture content of OMC+3%, conditions that were determined in the laboratory study to yield the highest strength gains. Unlike the 40°C and 7-day curing protocol typical in laboratory settings, the field simulation adopted a 30°C heating temperature and a 1-day curing period to more accurately reflect real-world construction conditions.

This innovative field simulation aimed to demonstrate the feasibility of using calcined ESP for soil stabilization in actual construction scenarios and to provide a practical framework for future applications and further research into sustainable construction materials.

Testing Results

The laboratory-controlled DCP testing results shown in Figure 42 demonstrate that calcined ESP significantly enhanced the CBR values of the treated soils, which experienced up to a 3.5-fold increase after just one day of curing. The results for penetration depth per blow shown in Figure 43 indicate that ESP-treated soils are stronger than untreated soils, evident in the lower penetration depth in the ESP-treated soils after one drop of the hammer. These findings align closely with the results obtained from UCS testing, which further validates the effectiveness of using eggshells as a bio-cement material for soil stabilization purposes.

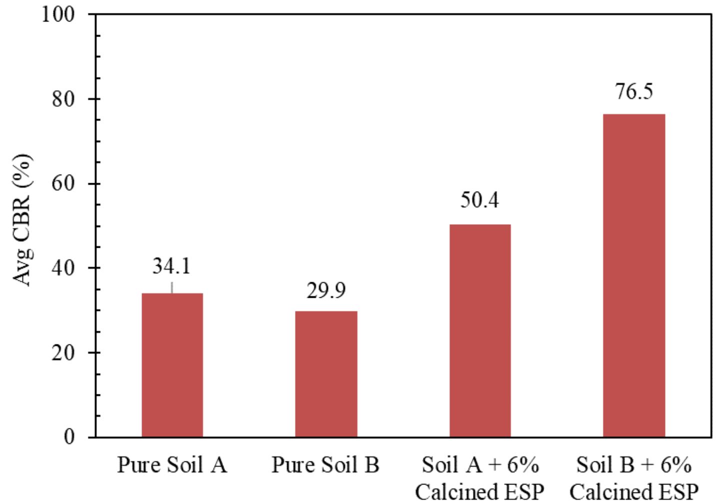


Figure 42. CBR values from laboratory-controlled DCP test

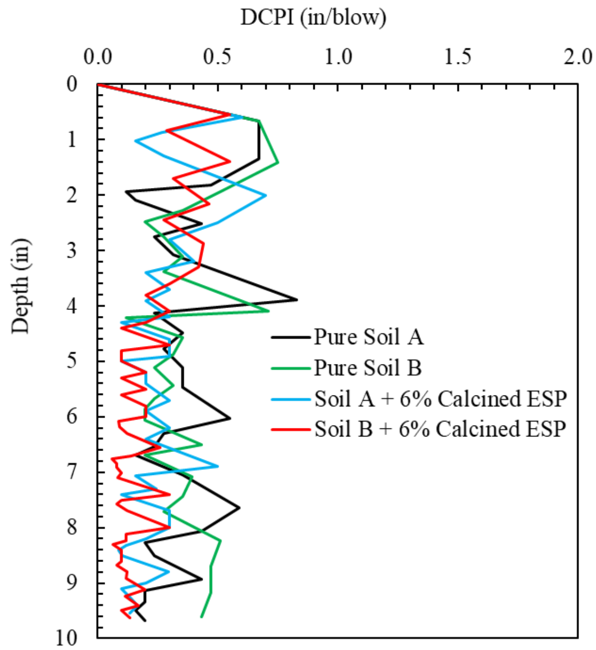


Figure 43. Penetration depth per DCP blow

This correlation between DCP and UCS testing outcomes underscores the potential for eggshell materials to improve the structural integrity and load-bearing capacity of soils and to make soils more suitable for construction and infrastructure projects. The observed effectiveness of calcined ESP in enhancing soil properties highlights its potential as a sustainable and environmentally friendly alternative to traditional cementitious materials.

Given the positive impact of higher temperatures on the hydration and pozzolanic reactions essential for the stabilization process, it is anticipated that the application of eggshell materials in soil stabilization projects would be most advantageous during the summer months. The higher temperatures characteristic of this season are likely to further facilitate these chemical reactions and thereby maximize the strength and durability of the stabilized soil. This insight suggests a strategic approach to scheduling construction and stabilization projects that takes advantage of seasonal variations to optimize the performance of eggshell-based bio-cement materials.

The laboratory-based DCP field simulation test successfully demonstrated the significant benefits of using calcined ESP to enhance the engineering properties of soil. When used similarly to quicklime, ESP reacts with soil particles to foster the hydration of lime to hydroxide. This transformation suggests that with appropriate processing and construction techniques, ESP could serve as an effective stabilizing additive, promising substantial improvements in soil stability.

Furthermore, a potential application of eggshell materials is in cement production. Demonstrations have already established that eggshell waste can be efficiently converted into CaO at a low cost. This breakthrough offers a valuable opportunity to produce cement using ESP-derived CaO, which presents an innovative approach to utilizing waste materials in construction. This not only highlights the versatility of eggshell waste as a resource but also underscores the environmental benefits of recycling waste into valuable construction materials.

CHAPTER 6: CONCLUSIONS

Eggshells are composed mainly of calcium carbonate, making them a valuable source of calcium, an essential nutrient for plants and soil. This natural composition, coupled with the abundant availability and low cost of eggs, has sparked interest in investigating their suitability for soil stabilization applications. The use of eggshells in soil stabilization offers several benefits. The calcium carbonate present in raw eggshell materials acts as a binder and filler, enhancing the mechanical properties of soil such as strength and stability. Calcium carbonate can be decomposed through calcination into CaO, an active compound that causes the hydration of lime to hydroxide, thereby dramatically altering the engineering properties of soils. This improvement in soil quality can lead to reduced settlement, increased load-bearing capacity, and improved resistance to erosion and deformation. Eggshells can also serve as a sustainable alternative to traditional stabilizers, reducing reliance on synthetic or chemical additives.

This study evaluated the performance of ESP-treated soil matrixes, and the key findings can be summarized as follows:

- Eggshell waste should be properly cleaned and ground into ESP before use in geo-material stabilization.
- The use of untreated and oven-dried ESP could slightly increase the LL and the PL of soils, leading to a minor increase in soil PI.
- The use of calcined ESP led to a significant decrease in LL and PI and an increase in PL, indicating a reduction in soil plasticity due to active chemical reactions involving the CaO present in calcined ESP.
- All ESP types showed a strong correlation between ESP content and maximum dry unit weight for Soil A, reflecting densification effects.
- Untreated and oven-dried ESP acted as filler material, improving maximum dry density without significant chemical interaction.
- The use of calcined ESP led to a limited increase in maximum dry unit weight because the hydration reactions resisted the compaction efforts, especially at higher moisture levels.
- The use of untreated and oven-dried ESP led to a limited strength improvement of less than 20%; extending the curing period or raising the temperature did not significantly affect performance because the ESP acted as a physical filler.
- The use of calcined ESP led to a strength improvement of 5 to 8 times when an additional 3% moisture above the OMC was applied and when the soil was cured at room temperature for up to 28 days.
- Elevated curing temperatures significantly increased the strength of Soil B by over 20 times, indicating enhanced hydration of lime to hydroxide.
- The optimum calcined ESP application rate was identified as 8%, with higher moisture levels (OMC+6%) leading to decreased strength compared to lower moisture levels (OMC+3%). However, since a 6% fraction of calcined ESP reflected the second-highest strength gain and was close to the performance of 8% ESP, 6% calcined ESP is recommended for field application.
- Based on the laboratory CBR testing, the optimal content of unheated ESP for improving soil strength was found to be 10% for both Soil A and Soil B. OMC also played a crucial role in

the compaction and strength outcomes. The samples for CBR testing were compacted at the desired OMC before being sealed and cured. Variations in the curing period significantly impacted the CBR results, indicating that both the OMC and the curing duration are critical factors in the effectiveness of ESP in enhancing soil strength.

- The laboratory-based DCP simulations demonstrated that calcined soil can triple CBR values compared to untreated soil. This finding aligns with UCS test results from this study.
- The XRD and SEM analyses revealed that adding calcined ESP (1,830°F or 1,000°C) to soils results in a denser and more solid soil structure due to the formation of hydration products. This was validated by XRD analysis and was visible in the microstructure of the treated soil samples.

CHAPTER 7: RECOMMENDATIONS FOR IMPLEMENTATION AND FUTURE RESEARCH

Implementation Recommendations for Using Eggshells as a Stabilizing Additive in Iowa

Based on the key findings, recommendations for using eggshells as bio-cement materials in the stabilization of Iowa geo-materials can be summarized as follows:

- Before field application, it is essential to thoroughly wash and calcine Iowa eggshell materials at 1,000°C for a minimum of five hours to remove any residual matter from the eggshells before they are finely ground into powder for use.
- The detailed illustration of ESP production in this study not only serves as a guide for replicating the study's findings but also suggests the potential for these processes to be adapted and scaled up for larger field applications. By highlighting the differences in outcome for each processing method, the study provides valuable insights that can be used to optimize the conversion of eggshell waste to high-quality ESP and emphasizes the feasibility and environmental benefits of recycling eggshell waste into a useful commodity.
- ESP is highly recommended for stabilizing expansive soils such as loess, and it has proven particularly advantageous for soils that exhibit high moisture contents or acidity levels.
- The optimal ESP application rate for field use is between 6% and 8%. This range allows for adjustments based on soil characteristics, budget constraints, and local environmental conditions to achieve the best outcomes.
- To enhance the hydration of lime to hydroxide, which is critical for soil stabilization, the addition of moisture to reach OMC+3% during construction is advisable.
- The most effective time to employ calcined ESP in construction projects is during the hot summer months, preferably around noon. The elevated temperatures at this time can significantly boost the stabilizing effects of ESP, leading to greater strength gains in the treated soil.

Future Research Recommendations for Using Eggshells as a Stabilizing Additive in Iowa

Future research recommendations can be summarized as follows:

- Initiate pilot projects focusing on the use of ESP in pavement foundations and granular roads to provide tangible evidence of its performance in real-world conditions. These projects should aim to replicate laboratory conditions as closely as possible to validate the effectiveness of ESP in soil stabilization and pavement improvement.
- Explore the synergistic effects of combining calcined ESP with other industrial byproducts such as fly ash and slag in concrete and mortar mixes. To reduce the carbon footprint of construction materials, the potential for ESP to act as a partial replacement for portland cement should also be investigated.
- Conduct environmental impact assessments to evaluate the life-cycle benefits and potential drawbacks of using ESP in construction. These assessments would include investigating the

carbon sequestration potential of ESP, the anticipated reduction in landfill waste, and the overall sustainability of utilizing eggshell waste.

- Conduct durability tests on ESP-treated materials against various environmental stressors such as acid rain, high-salinity environments, and extreme temperature fluctuations. Assessing the freeze-thaw durability and shear resistance of ESP-treated materials in more depth will provide valuable data on their resilience.
- Perform a cost-benefit analysis comparing the use of ESP to the use of traditional materials like lime and cement. This analysis should consider not only material costs but also long-term savings due to improved durability and reduced maintenance needs.
- Collaborate with regulatory bodies to develop standards and guidelines for the use of ESP in construction, including benchmarks for quality, safety, and environmental impact, in order to facilitate broader adoption.
- Develop educational programs and materials for industry professionals about the benefits and applications of ESP. Workshops, seminars, and online resources can help disseminate knowledge and encourage the adoption of ESP in construction projects.
- Investigate the nano-engineering of ESP to enhance its reactive properties and effectiveness as a supplementary cementitious material. This could open new avenues for high-performance composites and specialized construction applications.
- Foster international collaboration to share knowledge, research findings, and best practices related to the use of ESP and other sustainable materials in construction. This can accelerate innovation and the global uptake of green construction technologies.
- Using ESP for larger-scale field applications requires a significant amount of eggshell waste to be collected and processed. This process involves initial cleaning and preparation of the eggshells, followed by grinding them into a fine powder, and finally subjecting them to a chosen drying or calcination process. The Iowa State University research team began exploring the feasibility of establishing a mobile plant facility to produce ESP on a large scale or partnering with a cement plant company to use clinker to dry and process the raw eggshells.

Figure 44 illustrates how the method of ESP production described in this study could be extended to a larger-scale field application procedure. Figure 45 demonstrates a real-world ESP application.

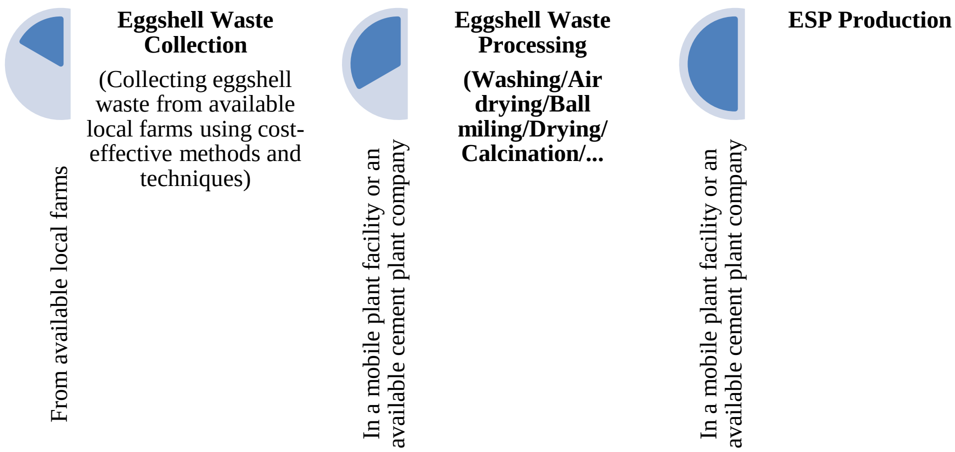


Figure 44. ESP production procedure for larger-scale field applications

Figure 45. Demonstration of a real-world ESP application

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