

Research Article

Influence of Curing Time and Initial Moisture Content on Metakaolin-Based Geopolymer-Stabilized Soft Soil

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This study thoroughly investigated the impact of initial moisture content and curing time on metakaolin (MK)-based geopolymer stabilized soft soil. The stabilized soft soil was characterized with an unconfined compressive strength test, scanning electron microscopy, energy dispersive X-ray spectroscopy (EDS), and compaction test. The geopolymer was used at different concentrations (5%, 10%, and 15% of the dry weight of the soil), and the soft soil was prepared at its initial moisture content, which ranges from 0.75 LL to 1.25 LL. The results of the test indicate that there was an increase in yield stress at low initial moisture content. Conversely, the yield stress experienced a decrease at high initial moisture content. Furthermore, the strength of geopolymer-stabilized soil increased as the curing time increased, regardless of the binder and initial moisture contents. The microstructure analysis confirmed that the stabilized soil had a denser microstructure, the formation of homogeneous gel, and fewer microcracks and pores. As the automatic compaction test revealed, the maximum dry density increased at higher binder contents while the optimum moisture content decreased. This research demonstrates that stabilizers made with metakaolin can efficiently stabilize soft soils. It is worthwhile to conduct further studies on the durability, shear tests, and cost of using geopolymer for soil stabilization under varying environmental conditions.

1. Introduction

Soft or extremely compressible soils are present in many civil engineering project sites. These soils cannot support the loading during construction or the duration of the service life [1, 2]. Soft soil (SS) is frequently linked to a fluctuation in water content that reduces shear strength and causes swelling, shrinkage, settlement, and consolidation, which can seriously harm a civil engineering project [3, 4]. In civil engineering construction, such as foundations and pavements, soil stabilization with lime or cement is a well-established soil treatment technology widely used to increase the load-bearing capacity of soft and weak soils [5, 6]. However, due to high energy and natural resource use and cement's enormous carbon footprint, researchers have been obliged to investigate other binders [7]. Geopolymers are energy-efficient cementing agents with low toxicity, good durability, and stability at high temperatures [8].

Geopolymers (GPs) are made from industrial wastes having a high amorphous (Si and Al) content, such as fly ash (FA) and metakaolin (MK), and an alkaline activator (such as potassium/sodium silicate and potassium/sodium hydroxide) [9–11]. The chemical synthesis of geopolymer involves dissolving aluminosilicate-rich materials in strongly alkaline solutions like sodium hydroxide (NaOH) [12]. The mechanical characteristics of geopolymer materials can be enhanced by sodium metasilicate (Na_2SiO_3). This activator supplies enough silicon ions to the polymerization reaction to accelerate the activation precursor of the materials [13]. Geopolymer gelation and characteristics are strongly influenced by variables such as the Si:Al ratio, the availability of reactive alumina, water content, curing environment, and precursor purity [14].

Metakaolin (MK) is produced when pure kaolinite is heated between 500 and 550°C. The crystalline structure of kaolinite is disrupted during the calcination process, which

also drives out the chemically bonded water in its interstices [15]. As a reactive amorphous pozzolanic material with latent hydraulic capabilities and generally finer particle size than cement, metakaolin is particularly well-suited for cementing applications. Shi et al. [16] investigated the mechanical characteristics and microstructure of modified clay by metakaolin-based geopolymer. The metakaolin was used in 4%, 8%, 10%, and 12% of the total dry mass of the metakaolin and soil. The alkali activator to metakaolin ratio was constant at 0.7, and the samples were prepared at the optimum moisture content (OMC). They revealed that the unconfined compressive strength (UCS) increases with metakaolin dosage and curing time. Zhou et al. [2] studied marine SS's mechanical characteristics and micromechanisms stabilized by various calcium-based precursors. Researchers discovered that the stabilizing effects of low-calcium content geopolymers, high-calcium content geopolymers, and OPC on soft marine soil were due to the formation of an amorphous gel network structure. Miraki et al. [6] conducted a study on utilizing volcanic ash and slag that have been alkali-activated to stabilize clayey soil. They revealed that sodium aluminosilicate hydrate (N-A-S-H) and calcium silicate hydrate (C-(A)-S-H) gels were formed using the proper combination of volcanic ash and slag. Wang et al. [17] conducted a laboratory study on geopolymer-improved soil's strength performance and material ratio. The researchers utilized metakaolin (MK) as a geopolymer binder and (CaO + NaHCO₃) as an alkali activator. They discovered that the metakaolin content and the alkali activators significantly influenced the strength performance. For deep soil mixing, Abdullah and Shahin [18] investigated the geomechanical properties of fly ash-based geopolymer stabilized clay. They used a combination of fly ash and slag at a ratio of 20% as a binder and NaOH + Na₂SiO₃ as an alkali activator. The initial moisture content of the soil was prepared at the liquid limit (LL) state. The findings showed that the main contributors to the improvement in mechanical strength were the curing time and the amount of geopolymer. Numerous studies have identified variables, including geopolymer content, a precursor to activator content, temperature, and water content, as influential parameters on the strength development of stabilized soils, with the degree of influence depending on the soil's characteristics. Notably, the vast majority of prior studies focused on stabilizing soils at the optimum water content [12–14], and it is uncommon to look into the engineering characteristics of geopolymer-stabilized soils with high water contents, such as those in coastal and estuarine areas [7].

The objective of this research is to investigate the effect of initial moisture content and curing time on the metakaolin-based stabilized SS. Metakaolin binder was used in proportions of 5%, 10%, and 15% of the dry weight of the soil. Sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) were used as alkali activators, as recommended by previous researchers. To achieve the stated goals, an unconfined compression strength (UCS) test, scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and compaction test were performed.

TABLE 1: Physical properties of soil.

Property	Values
Liquid limit, LL (%)	61
Plastic limit, PL (%)	34
Plasticity index, PI (%)	27
Passing sieve 75 μm (%)	86.08
Sand fraction ($\geq 75 \mu\text{m}$) (%)	13.92
Silt fraction ($75 \mu\text{m} \leq 2 \mu\text{m}$) (%)	61.69
Clay fraction ($< 2 \mu\text{m}$) (%)	24.39
Soil pH value	7.88
Specific gravity (G_s)	2.73
Maximum dry density, MDD (g/cm^3)	1.52
Optimum moisture content (%)	26
Soil classification (USCS)	MH

2. Materials and Methods

2.1. Experimental Materials

2.1.1. Soft Soil. The soil sample used in this study was collected from Samsun, Pelitköy, at the construction site, about 2–3 m deep. The disturbed soil samples were collected in plastic bags and air dried in the laboratory for several weeks. The soil was characterized by conducting several laboratory tests according to American Society for Testing Materials (ASTM) standards. The physical properties of the soil are listed in Table 1. According to the unified soil classification system [19], the soil was classified as highly plastic silt (MH). The soil under consideration has a significant proportion, ~86%, of particles smaller than 0.075 mm, which signifies that silt and clay are primary constituents of the soil. This soil type is characterized by its softness and fragility, and it cannot endure the weight of buildings and infrastructure above it [1]. Therefore, this soil type requires stabilization and special attention to minimize structural failures. The microstructure and mineralogical composition of the soil were analyzed using SEM and energy dispersive spectroscopy (EDS), as shown in Figure 1. The soil's chemical composition mainly comprises silicates and aluminum oxides, as shown by the experimental findings.

2.1.2. Metakaolin. Metakaolin (MK), a preferred binder, has low impurity concentrations and abundant reactive silica and alumina needed for geopolymerization. This binder has higher compressive strength, lower permeability, better workability, and much lower amounts of calcium oxide than fly ash and other aluminosilicate sources [20]. MK is considered environmentally friendly and cost-effective compared with other stabilizers [16]. Metakaolin is composed mainly of SiO₂ and Al₂O₃ oxides, which comprise around 95% of the compound. The sample was procured from a local supplier based in Turkey. AVS İÇ VE DIŞ TİCARET LTD. ŞTİ. company provided the chemical composition and physical characteristics, as shown in Table 2.

2.1.3. Alkali Activators. Sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) were used as alkali activators (L),

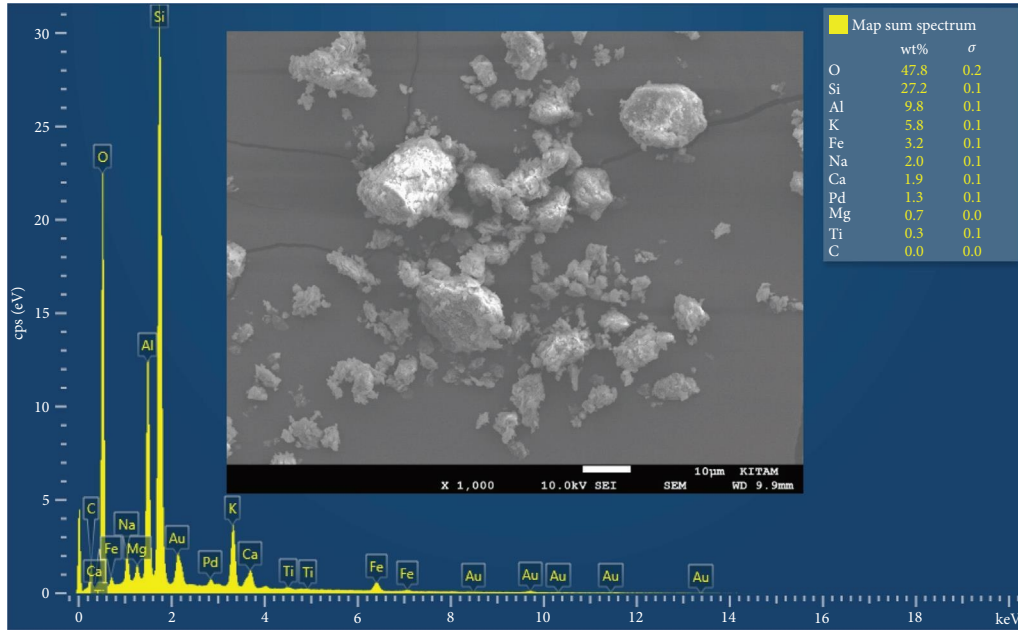


FIGURE 1: SEM and EDS results of the soil sample.

TABLE 2: Chemical composition and physical properties of metakaolin.

Compounds	wt%
SiO ₂	50.00 ± 1.0
Al ₂ O ₃	45.00 ± 1.0
Fe ₂ O ₃	0.50 max.
TiO ₂	1.00 max.
CaO	0.50 max.
MgO	0.40 max.
K ₂ O + Na ₂ O	0.55 max.
Physical properties	Results
Whiteness (ISO), %	89 ± 1.5
Brightness, %	87 ± 1.6
pH	5.5–7.0
Specific gravity	2.6
Oil absorption (g/100 g)	53 ± 5
Grain distribution	—
Sieve balance + 45 µm, %	0.004 max.
D50 µm	4–5
D90 µm	22–23
D98 µm	39–40

as suggested by several researchers [18, 19, 21]. The NaOH pellets were dissolved in water to achieve the desired molarity and then left to cool at room temperature for 24 hr. The NaOH solution is used for its cost-effectiveness and ability to dissolve silica and alumina, creating monomers from the source material [20, 22]. The Na₂SiO₃ composed of 8.5%–9.2% Na₂O, 25%–28.5% SiO₂, and the ratio of SiO₂/Na₂O ≤ 3.3.

2.2. Sample Preparation. Before mixing, all specimens, except for the compaction test, were pulverized using the Los Angeles apparatus and oven-dried at 105°C to reduce moisture content.

The soil sample preparation is shown in Figure 2. After pulverization for the intended laboratory tests, the larger particle size was shifted with a 2 mm opening sieve.

The geopolymer slurry was prepared using metakaolin as a binder and NaOH and Na₂SiO₃ as alkali activators, as shown in Figure 3. The NaOH pellets dissolved in distilled water for a molarity of 10 and allowed to cool for 24 hr. The molarity of NaOH was prepared by dissolving 400 g of pellets in 1 L of distilled water. Researchers suggest that a molarity range of 8–14 for NaOH effectively activates geopolymers [23–25]. As previous researchers recommended, the alkali solution of Na₂SiO₃ and NaOH was mixed at a proportion of 70/30, respectively [22, 25].

2.3. Dry Density–Moisture Content Relationship. The compaction characteristics of SS were determined using an automatic soil compactor. The standard proctor mold has an internal diameter of 10.16 cm, a height of 11.64 cm, and a volume of 944 cm³. The compaction test was conducted according to the ASTM-D698 testing procedure. After applying geopolymer, the compaction test was done on treated specimens to determine maximum dry density (MDD) and OMC from compaction curves.

2.4. Unconfined Compressive Strength (UCS) Test. The UCS tests were performed on geopolymer-treated soils according to the ASTM-D2166 standard. Water up to the LL is added to the soil and held in a closed container for 24 hr to prepare the SS. The geopolymer slurry was added to the SS and thoroughly mixed using an automatic mixer for 5–10 min. Three geopolymer dosages (5%, 10%, and 15%) by the dry weight of the soil were prepared and tested after 7, 14, and 28 days of curing. The softer UCS specimens were compacted in the mold using a vibrator, whereas the stiffer specimens were statically compacted while controlling the weight of each sample. Polyvinyl chloride (PVC) plastic molds were used to prepare the



FIGURE 2: Soil sample preparation process.



FIGURE 3: Geopolymer slurry preparation using alkali activators: (a) NaOH pellets, (b) NaOH and Na_2SiO_3 solutions, and (c) geopolymer slurry.

remolded untreated and geopolymer-treated soil specimens, as shown in Figure 4. The soil specimens were statically compacted in the PVC mold and placed in the curing chamber. The UCS tests were conducted at a 1 mm/min strain rate. Duplicate specimens were prepared for each dosage and curing period to ensure data reliability. The results presented here are the average values of the identical samples, with a coefficient of variation less than 20%.

The PVC plastic mold was 46 mm in internal diameter and around 103 mm in height. The mold's inner surface was coated with oil before the placement of the soil specimens. After 2 days of curing, the UCS specimens were demolded, wrapped in plastic films, and put back in the chamber for the remaining curing times, as shown in Figure 5.

The effects of initial moisture content, curing time, and changes in the microstructure of SS were discussed briefly. In this investigation, tests for UCS and SEM were carried out on various ratios of metakaolin. The alkali activator (L) ratio to binder (MK) was maintained at 1 for all tests. The detailed mix design of the experimental work is shown in Table 3.

3. Results and Discussion

3.1. Dry Density–Moisture Content Relationship. The compaction characteristics curves for SS and geopolymer-treated

soil are presented in Figure 6. The SS's MDD and OMC were 1.52 g/cm^3 and 26%, respectively. The soil treated with geopolymer (GP) shows an increase in dry density and a decrease in moisture content. Previous studies have also reported a reduction in OMC for soil stabilized with geopolymer [27, 28]. Adding a viscous alkali solution likely caused the decline in OMC, as it improves lubrication for soil particles. The 15% geopolymer treatment has been found to achieve a greater MDD than that of the SS. Furthermore, a higher dosage of metakaolin-based geopolymer has been shown to increase dry density while reducing moisture content [29].

3.2. Stress–Strain Relationship. The SS was prepared to a LL value of 61%, and the alkali activator to metakaolin ratio was kept at 1.0. The stress–strain curves for geopolymer-treated soil with 5%, 10%, and 15% binder contents and SS are illustrated in Figure 7(a)–7(c). The UCS test shows that adding geopolymer enhanced the yield stress of the treated soil compared to the untreated soil [18]. The 15% geopolymer-treated SS has the lowest yield (peak) stress compared to the 5% and 10% geopolymer-treated SSs, as shown in Figure 7(c). The geopolymer becomes too liquid and less conducive to connecting soil when the alkali activator to metakaolin ash ratio increases [16]. The geopolymer samples with 5%, 10%, and 15% binder content and the 28 days curing time resulted



FIGURE 4: UCS specimen preparation using PVC plastic mold.



FIGURE 5: Specimens curing chamber.

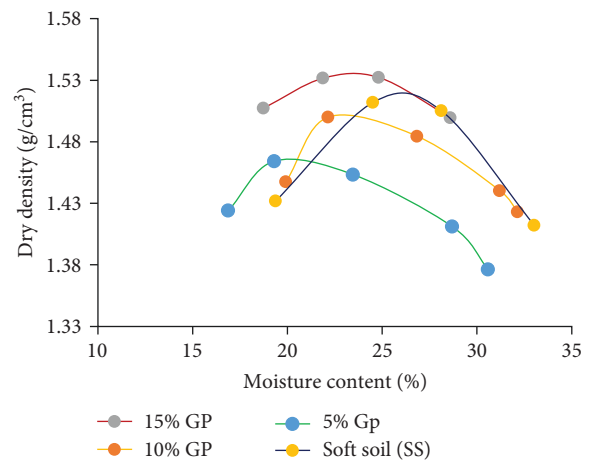


FIGURE 6: Moisture content–dry density relationship of soft soil and geopolymer-treated soil [26].

TABLE 3: Details of mix design for the experiment.

Mixture	MK content (%)	L	Na ₂ SiO ₃ /NaOH	L/MK	Curing time (days)
Soil + 5% MK	5	Na ₂ SiO ₃ /NaOH	70/30	1	7, 14, and 28
Soil + 10% MK	10	Na ₂ SiO ₃ /NaOH	70/30	1	7, 14, and 28
Soil + 15% MK	15	Na ₂ SiO ₃ /NaOH	70/30	1	7, 14, and 28

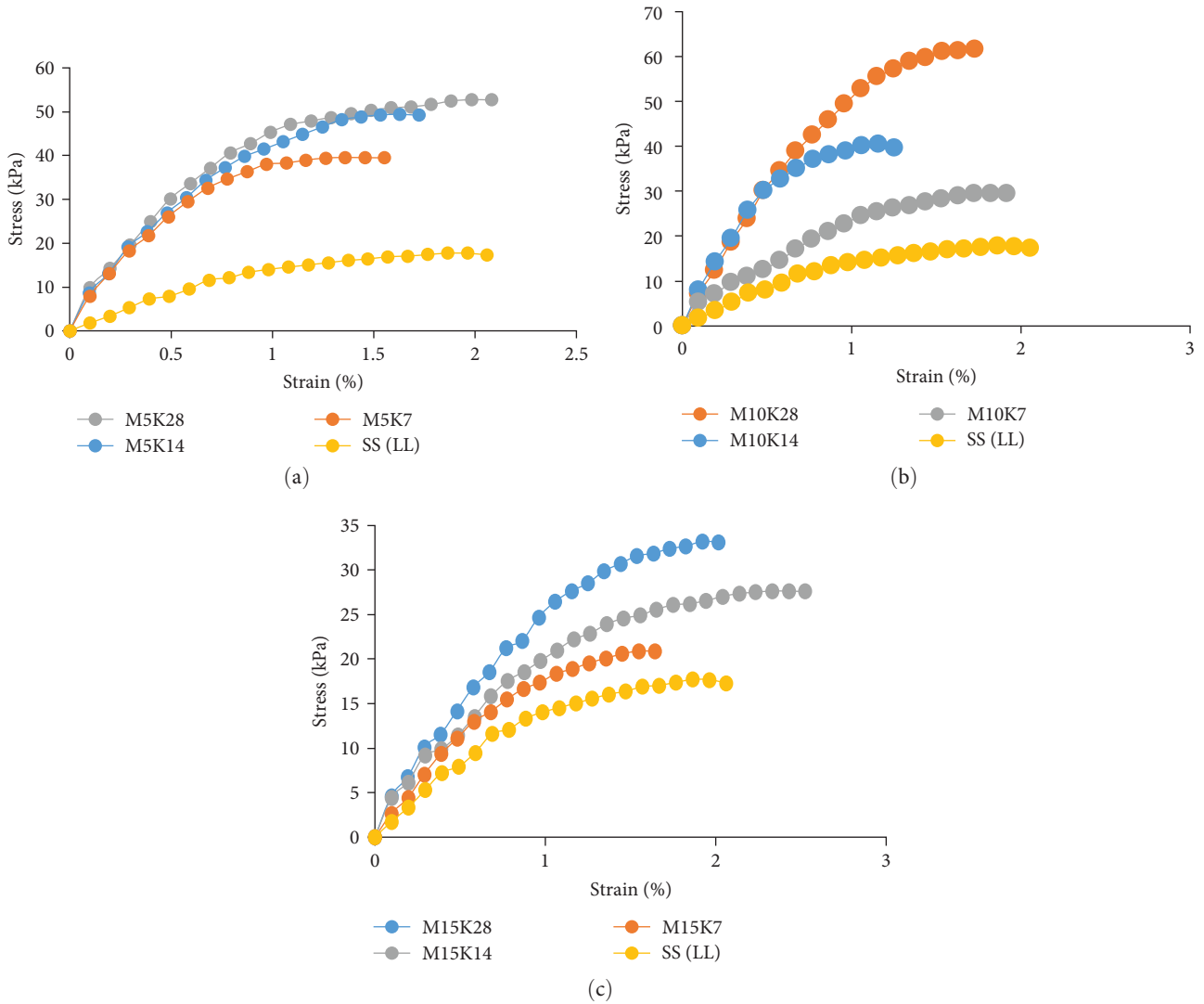


FIGURE 7: Stress–strain curves: (a) 5% binder, (b) 10% binder, and (c) 15% binder contents.

in yield stresses of 52.74, 61.34, and 33.2 kPa, respectively. The geopolymer with a 10% binder content showed the highest peak stress. It is worth noting that the yield stress for each binder content increases as the curing time increases. As anticipated, the UCS values of all MK-stabilized soils were significantly higher than their untreated counterparts [1].

3.3. Effect of Binder Contents on UCS Values. The UCS values of the geopolymer-treated specimens were analyzed after 7, 14, and 28 days of curing periods. Figure 8(a) shows that the UCS values increase as the binder content and curing time increase when the initial moisture content of the SS is 0.75 LL. Researchers found that adding metakaolin to SS increases the UCS values [1, 14, 30, 31]. The increase in silicon and calcium levels in the matrix resulted in higher values of UCS. At low initial moisture contents, UCS values increase with binder content, while in Figure 8(b), they decrease with increasing binder content. After conducting tests on UCS, it was found that the 10% MK geopolymer produced higher compressive strength when the initial moisture content was 1 LL.

3.4. Effect of Initial Water Content on UCS. Figure 9 presents the impact of different initial moisture contents on UCS values for a 10% geopolymer binder. The UCS values are observed to decrease as the initial water content increases. Evidently, the water content exceeded the required amount for the given binder content. The high water content filled gaps in the soil structure, reducing the molarity of alkali activators (L). This made fewer silicon and aluminum ions available for geopolymerization [22, 32]. The UCS values increased as the curing period increased, regardless of the initial water content. However, a significant difference in strength was observed when the initial water content changed from 0.75 LL (45.75%) to 1.25 LL (76.25%). This study compared two soil samples' UCS values. The first sample had an initial moisture content of 0.75 LL and a UCS value of 184.07 kPa at 28 days of curing.

On the other hand, the second sample had an initial moisture content of 1.25 LL and a UCS value of 8.6 kPa at the same curing time. No strength development was observed at 7 days curing periods when the initial water content was 1.25 LL

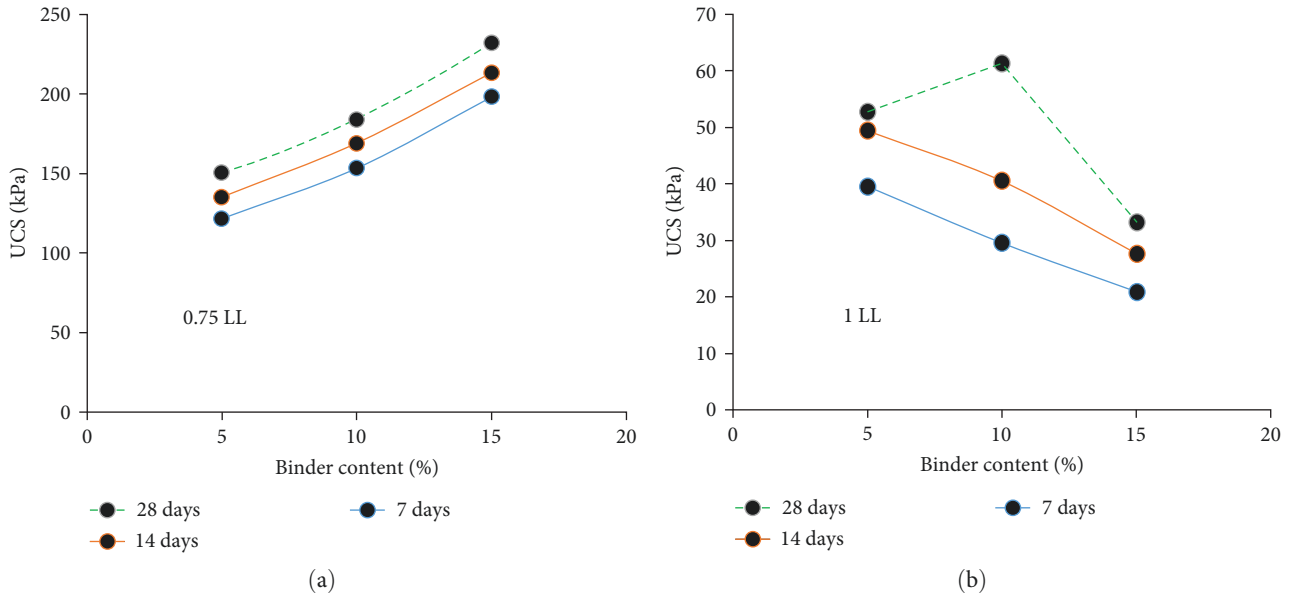


FIGURE 8: UCS values at various binder and initial water contents: (a) initial moisture content (0.75 LL) and (b) initial moisture content (1 LL).

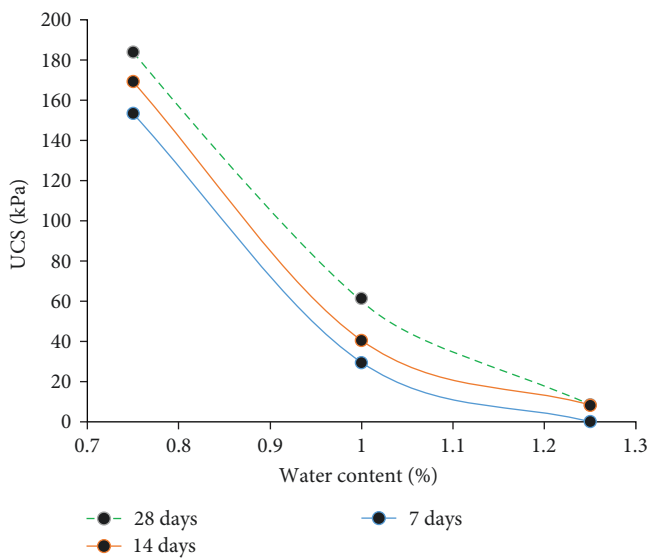


FIGURE 9: UCS values at different initial water contents for 10% MK binder.

(76.25%). It was observed that the initial water content significantly influences the strength of UCS specimens.

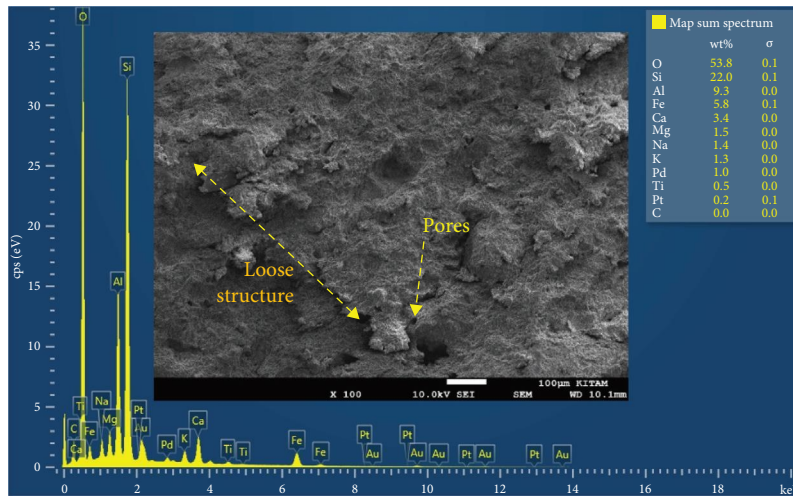
3.5. Microstructure Characterization. The scanning electron microscope uses a high voltage to accelerate electrons, which are then focused on the sample in a vacuum. As the electron beam scans the model, various interactions occur. Detectors capture signals from interactions, amplify them, and display them on a screen. The scanning electron microscope enables microstructure observation with increased resolution, depth of focus, and the ability to combine image and analysis utilizing various microscopy methods. When viewed at multiple length scales, the microstructural characteristics of a particular

material might alter significantly [33]. SEM and EDS tests have been conducted for 5%, 10%, and 15% MK geopolymer-treated soil at 7 and 28 days of curing periods. In this study, the treated soil micrographs were magnified 100x and 10,000 times. The SEM images at 100x amplification enable us to see pores and cracks, while higher amplification makes it possible to see microcracks, micropores, and hydration products [34]. After 7 days of curing, the pores, cracks, and loose structures in the 5% and 10% MK-based geopolymer-treated soil were observed, as shown in Figure 10(a)–10(c). However, the 15% MK-treated soil exhibited a more compacted structure. Shi et al. [16] found that treated soil had a denser and more compact structure than untreated soil. Figure 11(a)–11(f) illustrate that the 10% MK-based geopolymer-treated soil has more porosity than the 5% and 15% MK-based geopolymers.

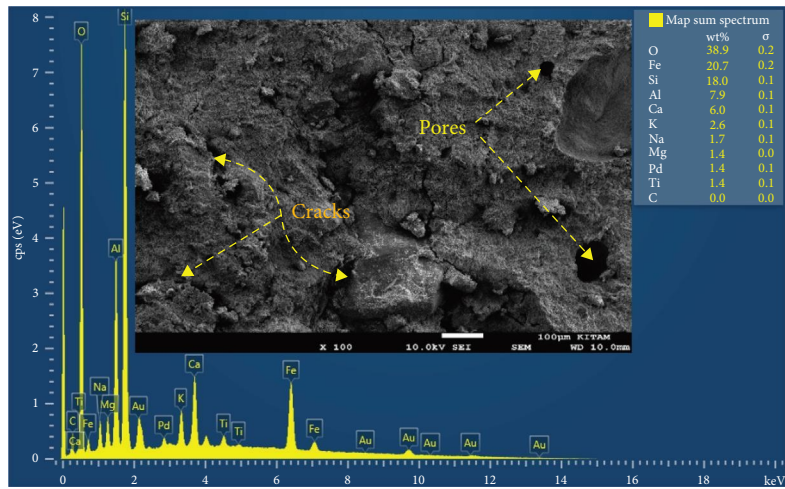
4. Conclusions

This study examined the effect of initial moisture content and curing periods for SSs stabilized with metakaolin-based geopolymer. UCS test, SEM, energy dispersive X-ray spectroscopy (EDS), and compaction test were performed to evaluate the mechanical strength and microstructure of the stabilized soil. Based on the experimental findings, the following conclusions can be drawn:

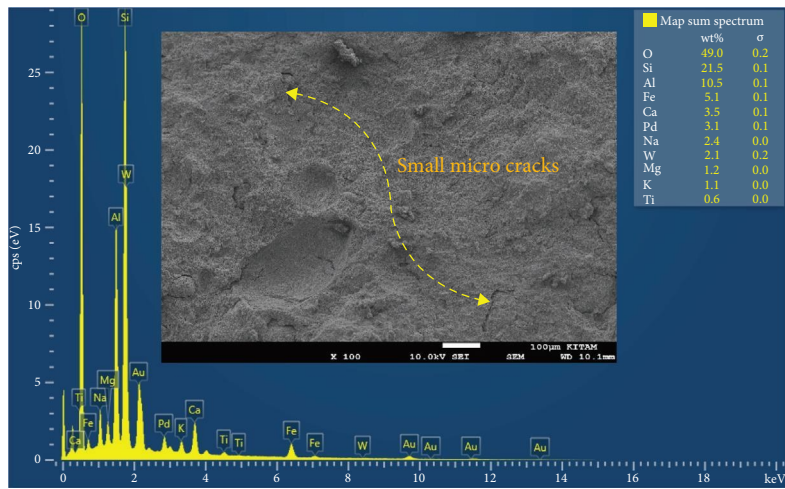
- (1) UCS values increased with increased binder content and curing periods at 0.75 LL initial moisture content. The soil's UCS values at 7 and 28 days of curing were 198.17 and 232.21 kPa, respectively, with a 15% MK binder. Moreover, increasing the binder content from 5% to 15% resulted in a remarkable increase in UCS values of 150.43 and 232.21 kPa, respectively.
- (2) The impact of the initial moisture content on the UCS values of SS treated with 10% MK has been thoroughly analyzed in this study. The UCS values



(a)



(b)



(c)

FIGURE 10: SEM and EDS results at 7 days curing time: (a) 5% MK, (b) 10% MK, and (c) 15% MK.

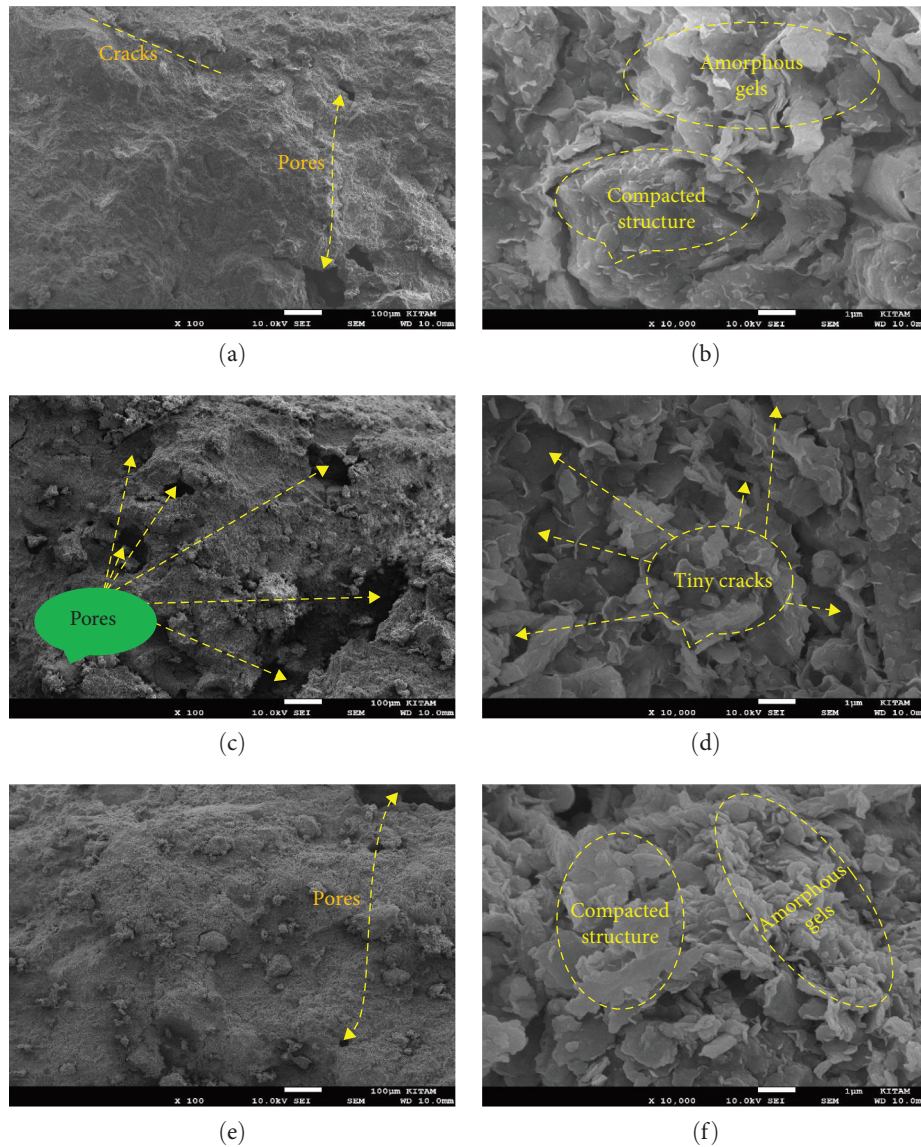


FIGURE 11: SEM results for 5%, 10%, and 15% MK at 28 days curing time: (a) 5% MK at 28 days ($\times 100$), (b) 5% MK at 28 days ($\times 10k$), (c) 10% MK at 28 days ($\times 100$), (d) 10% MK at 28 days ($\times 10k$), (e) 15% MK at 28 days ($\times 100$), and (f) 15% MK at 28 days ($\times 10k$).

significantly decreased when the initial moisture content varied between 0.75 LL and 1.25 LL. Soil specimens with 0.75 LL initial moisture content had a UCS value of 184.07 kPa, while those with 1.25 LL initial moisture content had a UCS value of 8.36 kPa.

- (3) A higher dosage of metakaolin-based geopolymer effectively reduced the OMC and improved the MDD. The soil treated with 15% MK showed a dry density of 1.53 g/cm^3 and an OMC of 23%, whereas the untreated soil had a dry density of 1.52 g/cm^3 and an OMC of 26%.
- (4) The SEM results showed that the treated soil samples had a more compact microstructure with fewer microcracks and pores, and a formation of homogeneous gel was observed.
- (5) Using metakaolin-based geopolymer to stabilize SS is an effective and eco-friendly alternative to

traditional stabilizers that emit high levels of CO_2 . The soil was effectively enhanced, resulting in increased strength and reduced compressibility. This remarkable improvement significantly minimized deformation and settlement in the construction of buildings and roads. In this research, a limited number of laboratory tests were carried out, and soil stabilization was done for high-plasticity silty soil (MH). The durability test, shear tests, and the cost of using metakaolin-based stabilizers are recommended for further studies.

Data Availability

The datasets generated and analyzed during the current study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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