

Article

Effects of the Types and Amounts of Clay Minerals on Durability of Lime-Stabilized Clay Soils

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Featured Application: We found that in the areas with high seasonal rainfall, to stabilize clay soils, if the amount of Montmorillonite mineral is greater than a certain amount (for example, in this study, 45%), with any amount of chemical additives, they do not maintain their stability and collapse. Conversely, soils containing certain amounts of Illite and Kaolinite minerals with the smallest amount of chemical additives have the best performance.

Abstract: Although the interaction between clay minerals and lime is the most effective factor in lime stabilization techniques, it has not been deeply evaluated. This research study investigated the microstructural characteristics of lime-stabilized Bentonite and Kaolin soils using X-ray diffraction (XRD), scanning electron microscope (SEM), and energy dispersive X-ray (EDX) analyses. To consider the variation in clay mineralogy, these soils were mixed at varied ratios, stabilized, and then subjected to a durability process. The microstructural findings showed that the pozzolanic reactions with lime did not occur or occurred at a low level for Bentonite soil. However, they occurred at a very high level for Kaolin soil. The durability test confirmed the microstructural results and showed that the samples in which Bentonite soil had a share of 40 to 100% by dry weight of the soil did not last with any percent of lime. When the Kaolin soil content reached 100% by dry weight of the soil, the specimens lasted in the best possible way, even with 4% of the lime. This study concluded that the determination of optimum lime content based on the amounts and types of soil clay minerals is an important innovation for geotechnical projects and may be very cost-effective.

Keywords: clay minerals; Pozzolanic reaction; Kaolin; Bentonite; wetting-drying; SEM-EDX; durability



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1. Introduction

Expansive soils with volumetric behavior exhibit swelling by absorbing water in the wet seasons and shrinking by water evaporation in the dry seasons. This swelling–shrinkage deformation results in heave, subsidence cracks, unpredictable upward movements, and large settlements of the structure formed on these soils [1–3].

Common research studies focusing on expansive soil properties and chemical stabilization are based mainly on the plasticity index (PI) [4,5]. There are particular constraints in the stabilization scheme instructions founded on plasticity characteristics. Soils could have the same plasticity but different dominant clay minerals. When these soils are stabilized using similar chemical additives and contents, they could show different engineering behavior. Therefore, it is necessary to consider the clay mineralogy of the soil and its complex interactions with stabilizers [6–10].

In lime applications, if laboratory studies that consider few effective factors are carried out in the field conditions, the hardness and strength obtained for the soil are lower than the values required [11]. On the contrary, the strength and hardness of lime-stabilized soil samples obtained in the laboratory studies that consider many factors such as destructive

weather conditions (freezing and thawing cycles and especially wetting and drying cycles), the aggregate size of soil [12], and watering the lime stabilization surface [13] could satisfy the field condition.

For a rural road that had two types of green and brown clay, lime stabilization techniques were carried out on both field and laboratory scales. They were evaluated with CBR, soaked CBR, uniaxial, and plate loading tests. All tests, both on the field and on a laboratory scale, confirmed that the pozzolanic reactions of lime with the clay soil in the watering condition on the stabilization surface caused a significant increase in their values within 28 days of curing compared with 7 days. If the clay surface was dry, in the absence of water, due to the lack of pozzolanic reactions, the strength parameters of the tests not only did not increase but also decreased in the long term [13].

Lime-stabilization of expansive clays from three soil borrows of a major highway project in Texas, Huston, was studied by Acula et al. [14]. The result of the study showed that PI and unconfined compressive strength (UCS) tests were not sufficient to examine the long-term durability of stabilization. Therefore, mineralogical methods containing quantitative XRD, differential thermogravimetric (DTG), and PH, along with geotechnical modeling, were performed to study the durability of lime-stabilized clay soils. The geotechnical modeling established in this research study investigated the stability of four kinds of calcium–silicate–hydrates (C-S-Hs) with Ca/Si molar ratios of 2.25, 1.33, 1.25, and 0.66 against three cycles of wetting and drying by changing the PH amounts of soil–lime samples. According to this model, a minimum dosage of lime is not enough for pozzolanic reactions. The C-S-H form with the $\text{Ca/Si} > 1$ played an important role in achieving different levels of strength of clay–lime samples.

A plastic silt soil was ground in three distinct powders so that the maximum aggregate size for each was 5, 1, and 0.4 mm. The stabilization of these three types of soil powders with 2% lime at different curing times was investigated by SEM, EDX, XRD, and mercury intrusion porosimetry (MIP) analyses. The results showed that after one year of curing, the C-S-H phase in the form of tobermorite was detected in the treated soil provided with the coarse aggregates. However, the C-S-H phase was not detected in the treated soil provided with the fine aggregates by XRD analysis and was also too small to be detected using MIP [15].

Song and Hong [16] explored the effects of clay minerals and grain size distribution on suction stress in two types of unsaturated soils. The mineralogical characteristics of both soils were determined by XRD and SEM analyses. This study showed that the unsaturated properties of the soils with the same grain size distribution relied on the clay minerals.

In a study conducted by Al-Mukhtar et al. [17], the amount of lime for short-term and long-term reactions of five soils, including different dominant minerals, kaolinite, illite, smectite–kaolinite, smectite–illite, and smectite was determined by XRD and TG analysis. The mechanism of the lime–clay reactions depended on the mineralogy of clay soil. The amount of lime consumed in this study changed from nothing for kaolinite to the maximum for bentonite during short-term reactions. The pozzolanic reactions altered the structure and mineralogy of clay soils treated with lime.

Rosen et al. [18] investigated the effect of curing time on the kinetic characteristics of pozzolanic reactions of clay soil with lime. The XRD analysis showed that the clay minerals of the soil included 40% illite, 32% kaolinite, and 28% montmorillonite. DTG analysis was performed to measure the amount of calcium carbonate before and after the stabilization of clay soil with lime. DTG results for the clay sample with 6% lime showed that the amount of calcium carbonate did not increase, but it was stable after one year of curing. In addition, its pH was still higher than 12.4 after six months. Therefore, the chemical, microstructural, and hydromechanical properties of the stabilized clay with 6% lime could change greatly in the long term.

The intragranular and intergranular distances of an untreated and lime-treated swelling clay were examined using SEM and MIP tests. Both tests confirmed that the intergranular size did not change because of clay soil modification with lime, but the intragranular size

increased. These changes caused an increase in electrical conductivity. Over time, due to the formation of cementation compounds, the electrical conductivity decreased [19].

Pedarla et al. [20] carried out a research study that aimed to determine the engineering behavior of clays with distinctive contents of montmorillonite mineral stabilized using different amounts of lime. The results indicated that the stabilization project is affected by the large content of montmorillonite, together with PI. Considering only PI will lead to unsatisfactory performance of stabilized clay soils rich in montmorillonite minerals. They found that soils containing more than 50% montmorillonite mineral could not be efficiently stabilized with 8% lime. Therefore, substitute stabilizers or the mixing of them require checking.

In addition, Bryson and Gomez-Gutierrez [21] studied the relation between shale mineralogy and engineering properties. Shales consist mainly of different contents of clay minerals such as kaolinite, illite, illite–smectite, and chlorite, in addition to quartz and some other minerals such as carbonates, pyrite, or iron oxides. Based on the results, changes in the samples' index characteristics and durability were explicated by the mineralogy.

Song et al. [22] investigated the New Orleans region with vast parts of expansive clay soils. Some parts of this region include as much as 5% montmorillonite in the soil. These expansive clay minerals caused high shear strength during dry seasons and very low shear strength during wet seasons or when water flowed into the gaps.

Chittoori et al. [23] checked the durability of chemically stabilized expansive soils exposed to wetting/drying (W/D) cycles, along with an emphasis on clay mineralogy. They stabilized eight different natural soils with varying clay mineralogy using lime and cement. Following that, they tested these samples for the durability process based on the ASTM D559 method. They examined the UCS and volumetric strain of the soil samples during the W/D procedure. Based on their results, the soils composed of high amounts of montmorillonite minerals were prone to early failings, although those with low amounts lasted all 21 W/D cycles. To implement the laboratory research practically, an interrelation formula to forecast service life in the field according to the clay mineralogy and the stabilizer dosage was expanded.

The expansive soils containing certain amounts of montmorillonite mineral were stabilized by Portland cement at low degrees, such as improving only strength parameters. A higher degree of modification was required to make the samples durable against W/D cycles. Upon adding epoxy resin in the content of optimum moisture, the stabilization performance was improved to a very high level in the range of the normal concrete so that Bentonite soil with the highest plasticity properties lasted successive W/D cycles [24].

Findings from these studies indicate that very limited knowledge is available regarding the role of clay minerals in the durability of lime-stabilized clay soils. The current stabilization instructions need to be investigated by combining clay mineralogy and durability. A major focus of this study was the examination of lime-stabilized soils exposed to W/D conditions and their clay mineralogy. Microstructural studies were performed on the untreated and lime-treated soils. Kaolin and Bentonite soils were combined at various ratios, stabilized using lime, cured, and then subjected to six cycles involving 48 h of W/D at 70 °C.

In the present research study, the results of the microstructural investigations, including SEM-EDX analysis of lime-stabilized clay soils and the durability process, are compatible. The soils containing a certain amount of illite and kaolinite minerals with the smallest amount of chemical additives have the best performance. Conversely, the stabilized clay soils containing a certain amount of montmorillonite mineral with any lime percentage do not maintain their stability against successive W/D conditions. Therefore, if no attempts are made to determine the amounts and types of clay minerals of the soils in the field projects for lime stabilization, lower performance, and more breakdowns are expected, especially in areas with heavy rainfalls.

2. Materials and Methods

This study considered utilizing two industrial soils, including Kaolin (Kao) and Bentonite (Bent). The Kao soil was in the form of a brown powder obtained from a mine located in Zarand Kerman, in the southeastern part of Iran. The Bent soil was a white powder obtained from a mineral company located in Khorasan, in the northeastern part of Iran. These two soils are used in the ceramic industry. Both soils were completely homogeneous (all the geotechnical and chemical properties were similar in their particles). The properties and classifications are given in Table 1. The soil samples were stabilized using hydrated lime (L). Some chemical and physical properties of hydrated lime are given in Table 2.

Table 1. Atterberg limits, soil gradation information, and the Unified Soil Classification System (USCS) for natural soils.

Bentonite	Kaolin	Property
396.2	30.3	Liquid limit (%)
40	19	Plastic limit (%)
356.2	11.3	Plasticity index (%)
0	36.6	Sand (%)
22	44.4	Silt (%)
78	19	Clay(%)
CH	CL	United Soil Classification
White	Brown	Color

Table 2. Chemical composition (% by weight) and physical properties of hydrated lime.

Parameter	Ca(OH) ₂	CaO	MgO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Density ($\frac{kN}{m^3}$)	Grain Specific Gravity	Loss on Ignition, LOI (%)	>75- μ m (%)
Value	90	73	1.37	1.77	0.71	0.11	4.7	2.31	24.1	1

XRD tests were performed on these soils. Approximately 10 g of each soil was air-dried for 24 h and pulverized to pass through sieve No. 400 (38 μ m diameter). The identification of expandable clay minerals included solvation with Mg. It was then exposed to glycerol to allow entry into the interlayer positions. Following that, it was heated for 1 h at 550 °C. Then, the sample was randomly mounted on a glass slide and analyzed using the XRD test.

The XRD test was conducted by employing the powder XRD method, a PANalytical X'pert APD diffractometer (with a Philips pw3830 goniometer), and a graphite monochromator (PHILIPS company in Netherlands). It was operated with a 40 kV/30 mA tube power and a Cu anode X-ray tube ($\lambda = 1.5418 \text{ \AA}$). Qualitative and quantitative analysis of the XRD patterns was performed using the Philips XPert Highscore plus version 3.0. software based on the International Centre for Diffraction Database (ICDD).

The changes in the microstructure of treated and untreated clay soils were studied using SEM and EDX analysis. Morphological data derived from SEM micrographs and chemical compositions were evaluated using the EDX technique. To prepare the samples for the SEM images and EDX spectra, the fractured fragments of untreated and lime-treated specimens were collected from uniaxial tests. These fragments were then oven-dried, pulverized, and passed through sieve No. 400. The powdered sample was coated with silver. Scanning electron micrographs of the silver-coated samples were obtained using a Jeol-Jsm 840A Scanning Electron microscope (Jeol company in Japan). EDX analysis was obtained at an acceleration voltage of 20 kV and a working distance of 10 nm.

2.1. Sample Preparation

Kao and Bent soils were mixed at different ratios of 0, 20, 40, 60, 80, and 100% for the sample preparation in this study. Each soil combination was completely mixed with 4, 8, 12, 16, and 20% lime in the dry state separately until the mixture reached a uniform and homogeneous appearance. All materials were added by employing the complementary substitution method [25].

For preparing the 60% Kao + 40% Bent + 20% L sample, 80% of the soil was mixed with 20% of the lime by dry weight of the soil. The soil was a mixture of 60% Kao soil and 40% Bent soil. The optimum moisture content was then added, and it was remixed thoroughly. Mixing continued until the final mixture reached a uniform moisture distribution. Then, the wet mixture was allowed to mellow. The untreated soil was put in plastic bags and left for 36 h, and the lime-treated soil for 1 h. Before compaction, the mixtures in plastic bags were passed through a No. 10 sieve. It led to the uniform distribution of lime and the soil and prevented the formation of large particles.

The soil samples were compacted at the optimum moisture content and maximum dry density obtained using the standard Proctor compaction conditions. In order not to affect the preparation method and sample compaction regarding the results of tests and also to investigate the clay minerals' impact on the compressibility of lime-stabilized kaolin and bentonite soil, the soil samples were compacted using the standard Proctor compaction conditions (constant energy equal to 0.055 (kgm)/cm^3). After compaction, each sample was wrapped in cellophane and placed in a polyethylene bag to prevent moisture loss. They were then cured at room temperature for 7 and 28 days.

2.2. Durability Testing

In this research study, it was attempted to follow the ASTM D559 standard [26] requirements to perform the durability process. This standard is about checking the durability of the soil–cement samples. Some experimental studies adapted this standard in a way that was compatible with the type of chemical stabilizer, soil, and the purpose of their research study [25].

After curing for 7 and 28 days, the soil–lime samples were subjected to six W/D cycles. Each cycle involved 48 h of wetting by immersing the soil samples in the water at room temperature. Then, they were dried at 70°C for 48 h. The wetting period was increased to 48 h compared with the 5 h recommended by ASTM D559 [26]. A prolonged wetting period was applied, causing the weak samples to disintegrate. Figure 1 shows how the cycles were applied.

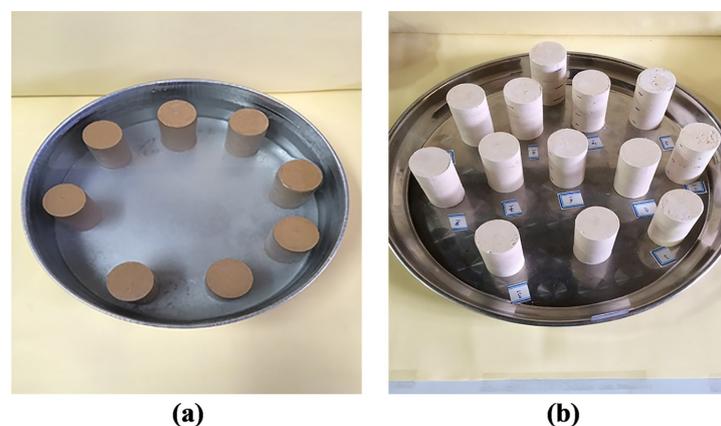


Figure 1. Kao + 12% L—28-day samples at the end of 48 h of (a) soaking (b) drying for the first cycle.

The Uniaxial Compressive Strength test was conducted based on ASTM D2166 [27], in which loads were applied to soil samples at a strain rate of 1 mm/min. It was measured for the soil–lime samples after 7 and 28 days of curing and after wetting in the third and

sixth cycles. Volumetric changes during wetting and drying in each cycle and the amount of weight loss due to brushing were measured for the samples that lasted up to 6 cycles following the ASTM D559 standard.

3. Results and Discussion

3.1. Microstructural Assessment of Lime-Stabilized Kao and Bent Soil Samples

This section first estimated the types and amounts of clay minerals of untreated Kao and Bent soils by mineralogical analysis of the XRD test. Then, the morphological surface changes in Kao and Bent soils' clay minerals and their effects on the quality of lime stabilization were examined by SEM images. They were taken at the magnification capacity of 30 KX and the scale bar of 1 μm . The EDX spectrum was conducted along with SEM imaging to control the changes in the chemical formation of clay soils before and after treatment with lime. The EDX analysis was performed at three points specified on each SEM image.

3.1.1. Quantitative Mineralogy Analysis of Kao and Bent Soils with XRD Test

Glycerol solvation transfers the 001-peak of Mg-saturated smectite from 12–15 \AA to 17–18 \AA [28]. Heating the two vermiculite and smectite to 300 $^{\circ}\text{C}$ and greater temperatures lead to water evaporation and move the 001-peak to 10 \AA [28]. According to Figures 2a and 3a, glycerol solvation shifted the 001-peak of Mg-saturated Bent soil from 12.88 \AA to 17.73 \AA , and heating to 550 $^{\circ}\text{C}$ moved the 001-peak to 9.89 \AA . Therefore, the clay mineral of the Bent soil was montmorillonite.

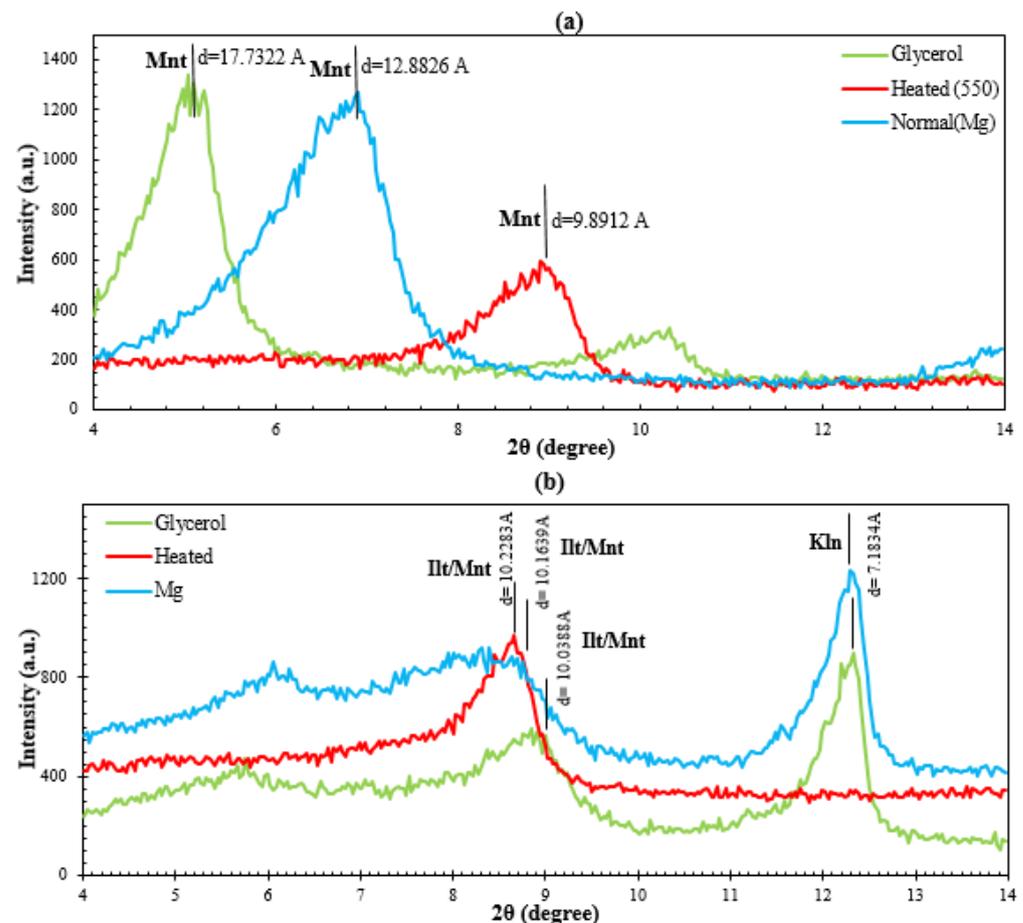


Figure 2. X-ray diffraction of Mg-solvation, Glycerol-solvation, and Heated clay fractions of the selected samples: (a) Bent soil; (b) Kao soil.

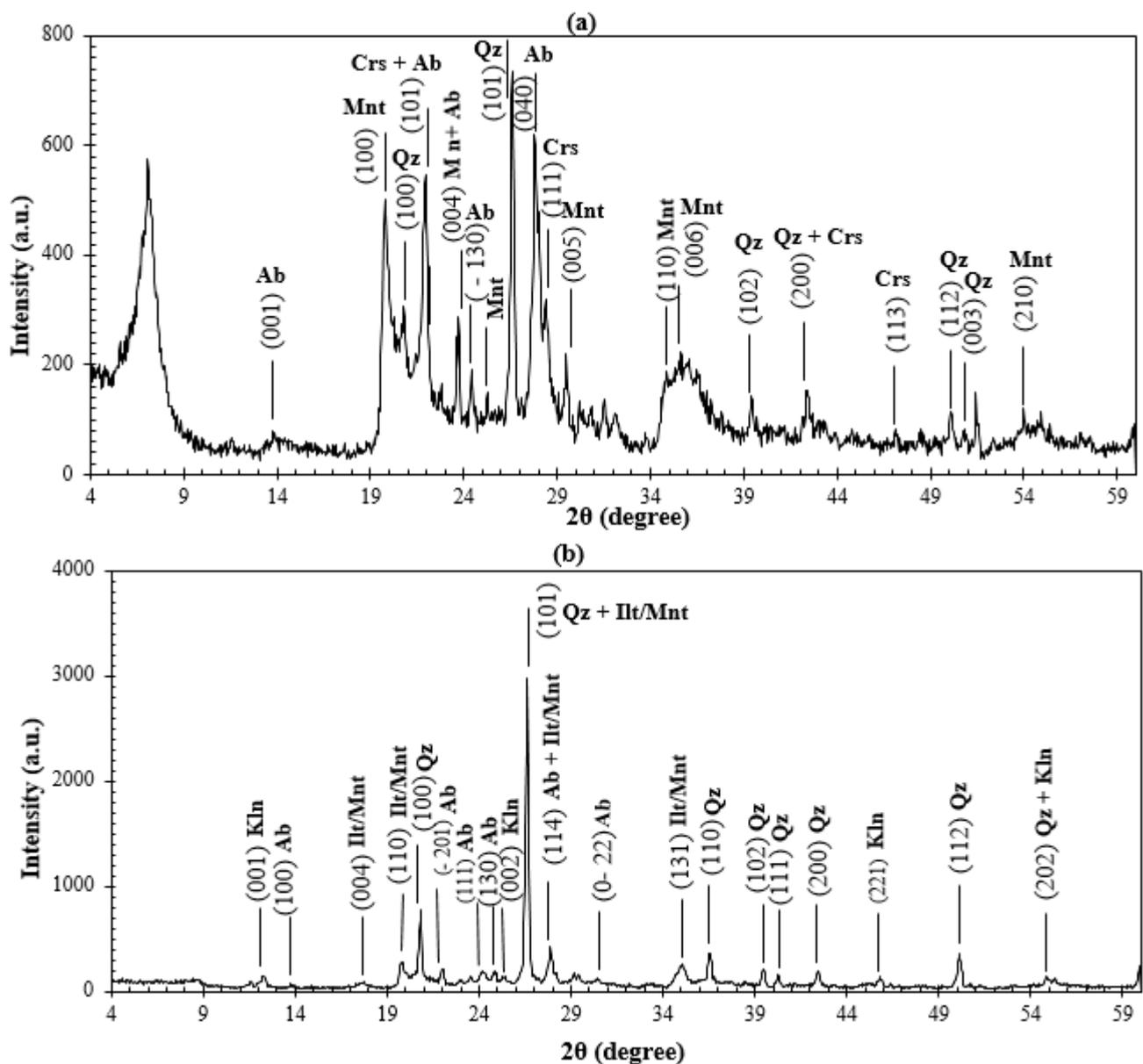


Figure 3. X-ray diffraction of (a) Bent soil; (b) Kao soil containing Montmorillonite (Mnt), Kaolinite (Kln), Cristobalite (Crs), Illite–Montmorillonite (Illt/Mnt), Quartz (Qz), Albite (Ab), and d-spacing in Angstrom (d).

The peak reflections fail to recognize kaolinite at 7.15 Å and 3.57 Å, which stays constant when exposed to solvation with glycerol (Mg-saturated) and disappear upon heating to 550 °C [29,30]. Two distinctive types in some clay soils are formed by the mixed layer minerals, including one smectite-rich, S/I, with a peak near 15.5 Å and the other illite-rich, I/S, with a peak nearer to 10 Å [31]. Based on these studies and Figures 2b and 3b, the clay minerals of Kao soil were kaolinite and the mixed layer of illite–montmorillonite. Adopting these methods [32,33], and according to Figures 2b and 3b, the mixing percentages of illite and montmorillonite in the mixed-layer mineral of illite–montmorillonite were calculated at approximately 95% and 5%, respectively. The quantitative analysis results of the XRD patterns performed on Kao and Bent soil samples are presented in Table 3.

Table 3. Mineralogical composition of Bentonite soil and Kaolin soil (wt% of the total amount).

Phase	Bentonite		Kaolin	
	Reference Code	(%)	Reference Code	(%)
Kaolinite, $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ (%)	-	-	00-029-1488	15
Montmorillonite, $\text{Ca}_{0.2}(\text{Al,Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot x\text{H}_2\text{O}$ (%)	00-013-0135	45	-	-
Quartz, SiO_2 (%)	00-033-1161	14	00-033-1161	35
Albite, $(\text{Na,Ca})(\text{Si,Al})_4\text{O}_8$ (%)	00-009-0457	26	00-009-0466	12
Cristobalite, SiO_2 (%)	00-039-1425	12	00-001-0424	3
Illite–Montmorillonite, $(\text{K,H}_3\text{O})\text{Al}_2\text{Si}_3\text{AlO}_{10}(\text{OH})_2$ (%)	-	-	00-026-0911	25
Gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (%)	-	-	00-033-0311	2
Hematite, Fe_2O_3 (%)	-	-	00-033-0664	2
Calcite, CaCO_3 (%)	00-005-0586	3	00-005-0586	2
Orthoclase (%)	-	-	00-031-0966	4

3.1.2. Scanning Electron Microscopic Analysis

The structure of illite and kaolinite clay minerals mainly consists of particular stacks of tetrahedral and pseudo-hexagonal sheets [34]. The texture of illite contains the kind of layer stacks that are thinner than kaolinite. The most important factor that affects the structure of illite clay minerals is the potassium interlayer cation (K^+). It makes the basal spacing structure wider than kaolinite [35].

Figure 4a is the SEM image of the untreated Kao soil, which indicates two types of minerals, including kaolinite and illite. According to the SEM image of Figure 4e, upon adding 4% lime to Kao soil and after 28 days of curing, significant changes occurred in the microstructure of the Kao soil. The pozzolanic reactions of Kao soil with lime have resulted in the formation of cementation compounds.

These adhesive materials bonded the clay mineral particles together, resulting in the creation of more agglomerated and denser particles. When some of the soil particles were substituted with lime particles, the lime provided adequate bonding material to create agglomerations. The intrinsic pozzolanic characteristic of clay minerals in Kao soil, which were kaolinite and illite in the reaction with lime, caused stiff texture and cementitious matrix formation. These cement materials covered and bound the separate Kao soil particles. It could be noted that even the types of clay minerals in Kao soil changed.

Based on the SEM image of Figure 4i, upon adding 20% lime to Kao soil, similar to the sample with 4% lime, because of the formation of structures called flocculated, significant morphological changes occurred compared with untreated Kao soil. However, the surface texture of Kao soil with 20% lime did not change much compared with the sample with 4% lime in terms of density and agglomeration.

The changes in the surface texture of untreated Kao soil with the addition of 20% lime compared with 4% lime were small and negligible. Therefore, according to the SEM images, adding 4% lime to Kao soil created cementation compounds with sufficient strength and durability.

Figure 5a is the SEM image of the untreated Bent soil that shows the montmorillonite mineral's texture. Irregular ridges and inhomogeneities are mainly observed in its surface morphology. According to Figure 5a,e,i, upon adding 4 and 20% lime to the Bent soil, slight changes occurred in the Bent soil's microstructure after 28 days of curing. These slight changes occurred particularly at the boundaries of some ridges on the surface texture of the soil. The surface texture of bent soil upon adding lime up to 20% has not flocculated and agglomerated. This indicated that no cementation compounds were formed to alter the surface texture and morphology of the Bent soil's clay minerals. Therefore, the pozzolanic reactions of lime with montmorillonite mineral of Bent soil were either performed poorly or not performed.

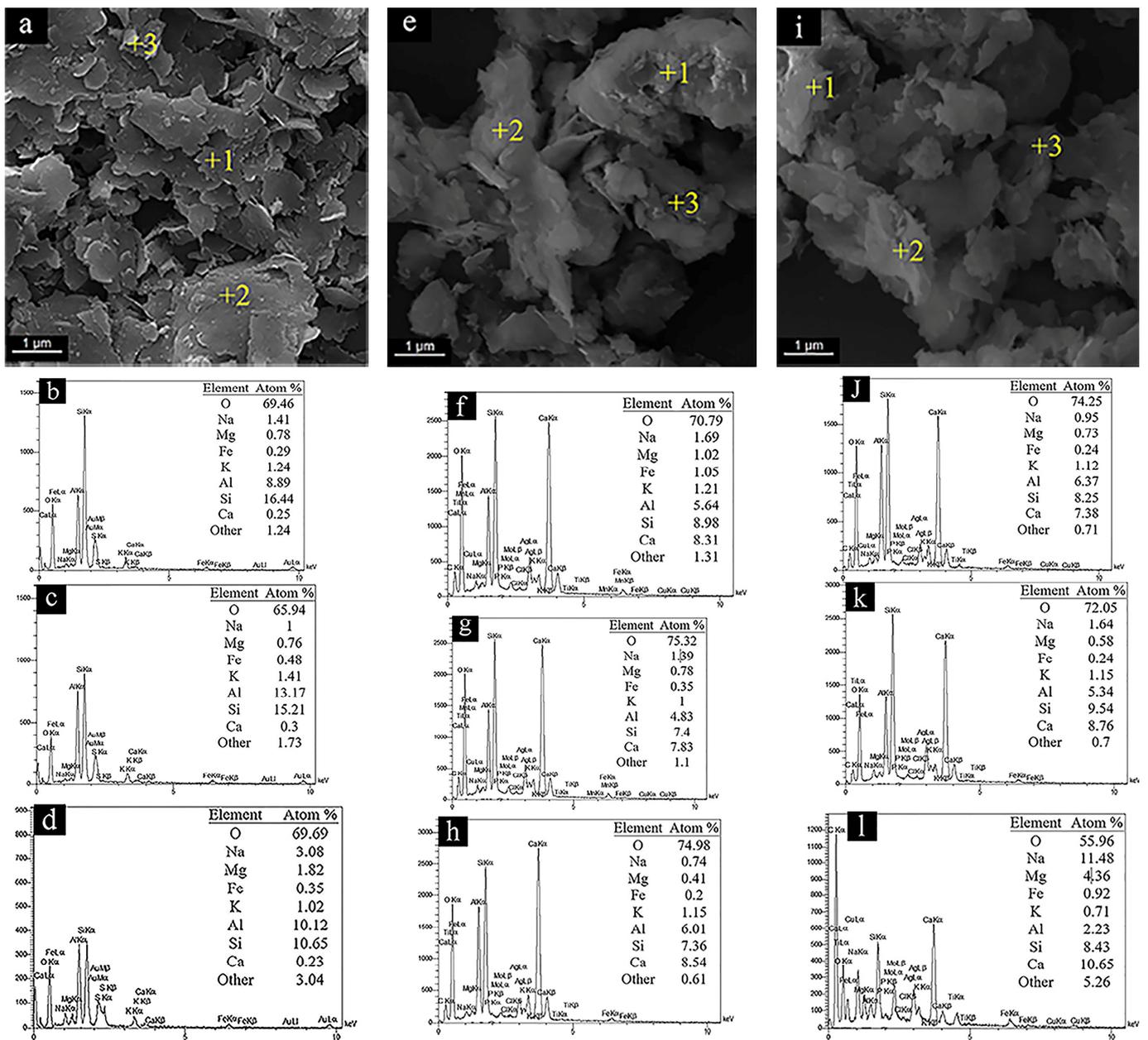


Figure 4. (a) SEM image of Kao soil and its EDX analysis at the specified point (b) 1, (c) 2, (d) 3; (e) SEM image of Kao + 4% L after 28 days of curing and its EDX analysis at the specified point (f) 1, (g) 2, (h) 3; (i) SEM image of Kao + 20% L after 28 days of curing and its EDX analysis at the specified point (j) 1, (k) 2, (l) 3.

3.1.3. Quantitative EDX Spectrum Analysis

The untreated Kao soil has a discontinuous texture. Its surface morphology contains considerable numbers of pores and cavities since no hydration materials exist (Figure 4a). According to EDX analysis results of Figure 4b–d, the strongest peaks are related to Si and Al, and the weakest peaks are related to Ca, Fe, K, and Mg. From Figure 4e, upon adding 4% lime and after 28 days of curing, the Kao surface soil was filled with cementitious materials such as calcium silicate hydrates (CSH), calcium aluminum silicate hydrates (CASH), and calcium aluminum hydrates (CAH). The EDX results of Figure 4f–h show that the values of Ca, Si, and Al peaks became stronger. This could be the result of the cementitious materials produced by the slow mechanism of the pozzolanic reaction.

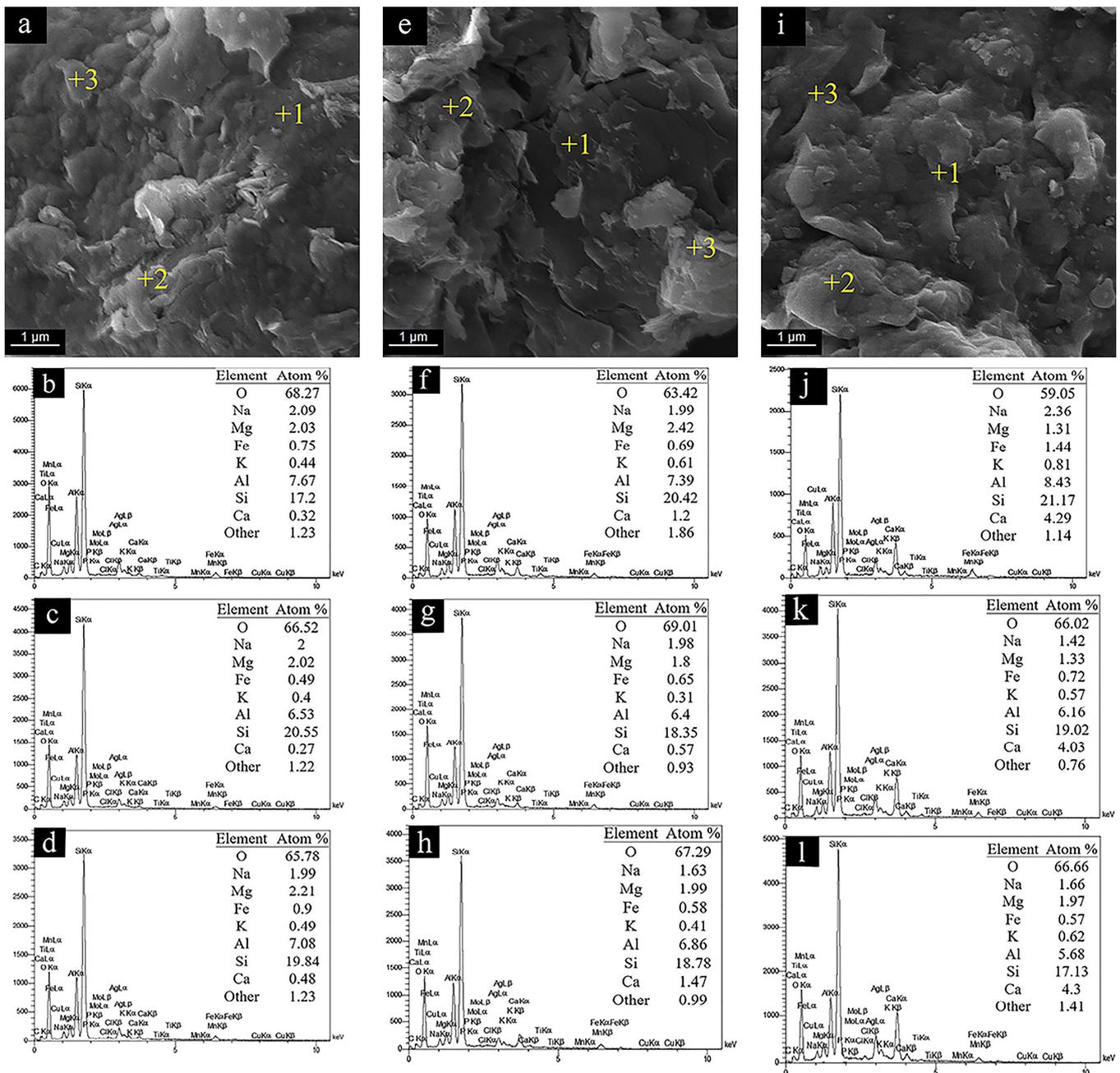


Figure 5. (a) SEM image of Bent soil and its EDX analysis at the specified point (b) 1, (c) 2, (d) 3; (e) SEM image of Bent + 4% L after 28 days of curing and its EDX analysis at the specified point (f) 1, (g) 2, (h) 3; (i) SEM image of Bent + 20% L after 28 days of curing and its EDX analysis at the specified point (j) 1, (k) 2, (l) 3.

In addition, upon adding 4% lime to the Kao soil and after 28 days of curing, the average molar ratio of Ca/Si at the three points shown on the SEM image of Figure 4a was increased from 0.02 to 1.05 at the three points marked on the SEM image of Figure 4e. Therefore, upon adding 4% lime, the average molar ratio of Ca/Si became 52.5 times that of the untreated Kao soil sample. This significant increase in the average molar ratio of Ca/Si upon adding 4% lime and its value, which was more than one, indicated that the pozzolanic reactions and the formation of cementation compounds had occurred.

Based on the EDX analysis results of Figure 4j–l performed at the three specified points of Figure 4i upon adding 20% lime to the Kao soil, and after 28 days of curing, its Ca/Si

molar ratios ranged from 0.89 to 1.26. Its average molar ratio of Ca/Si changed slightly compared with the sample with 4% lime. Therefore, the pozzolanic reactions with the addition of 20% lime to the Kao soil did not result in more durable cementation compounds than the sample with 4% lime. As could be seen from the SEM image of Figure 4e, upon adding 4% lime to the Kao soil, almost no clay mineral was found. With the addition of lime of greater than 4%, the necessary elements provided by the clay minerals of Kao soil were not sufficient for the pozzolanic reactions with the total content of the added lime. Therefore, at the microstructural scale, the optimum lime content was 4%.

The Ca/Si molar ratio for both untreated Kao and Bent soils was about 0.02 (see Figures 4b–d and 5b–d). From the results of the EDX analysis of Figure 5f–l upon adding 4 and 20% lime to the Bent soil and after 28 days of curing, the Ca/Si molar ratios at the three points marked on the SEM images of Figure 5e,i had little difference from each other, and their average molar ratios of Ca/Si were 0.06 and 0.22, respectively. Upon adding lime to the Bent soil, the molar ratio of Ca/Si must reach at least 0.55 for the pozzolanic reactions to start [36,37]. These small values in the Ca/Si molar ratios with the addition of 4 and 20% lime to Bent soil indicated that the pozzolanic reactions resulting in strong and durable cementation compounds were not performed. The average molar ratio of Ca/Si of Kao soil with the addition of 4 and 20% lime to Bent soil indicated that the pozzolanic reactions resulting in strong and durable cementation compounds were not performed. The average molar ratio of Ca/Si of Kao soil with the addition of 4% lime was 4.77 times that of the Bent soil sample with the addition of 20% lime. Therefore, the Kao sample, whose predominant clay minerals were kaolinite and illite, had sufficient elements for the pozzolanic reactions. It reacted with lime in the best possible way. However, the Bent sample, whose predominant mineral was montmorillonite, lacked sufficient elements for the pozzolanic reactions with lime.

3.2. Durability Assessment of Lime-Stabilized Kao and Bent Soil Samples against Wetting and Drying Cycles

3.2.1. Bent Soil Sample Pattern

When the untreated Bent soil sample was soaked in water, it began to absorb water. Its moisture content increased from 40 to 191% and swelled after 48 h. As shown in Figure 6, it had a severe jelly state because of the high plasticity and adhesion of its intrinsic clay minerals. According to Table 3, the swollen Bent soil sample contained 45 wt% montmorillonite mineral of the total amount. Despite the high swelling, it did not collapse and retained its relative stability. As soon as it was oven-dried, because of a large number of deep cracks of high shrinkage, it collapsed before being submerged in the water for the next cycle.

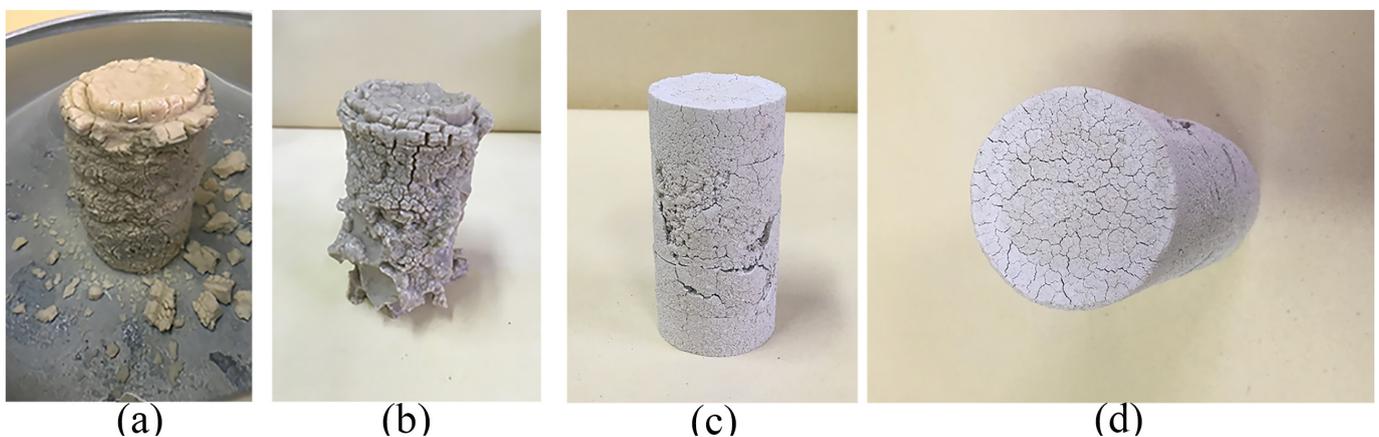


Figure 6. Bent soil sample: (a) By immersion in the water; (b) After 48 h of wetting in the water; Bent + 8% L—28-day sample after drying for the first cycle from the: (c) Front; (d) Top.

Figure 7 shows the UCS versus lime content for 60, 80, and 100% inclusion of Bent soil. Upon adding 4% lime, the UCS of Bent soil samples cured for 7 and 28 days became 3.8 and 4.5 times that of the unmodified samples, respectively. Additionally, upon submerging the samples in water about half an hour later, they collapsed much earlier than the unmodified samples.

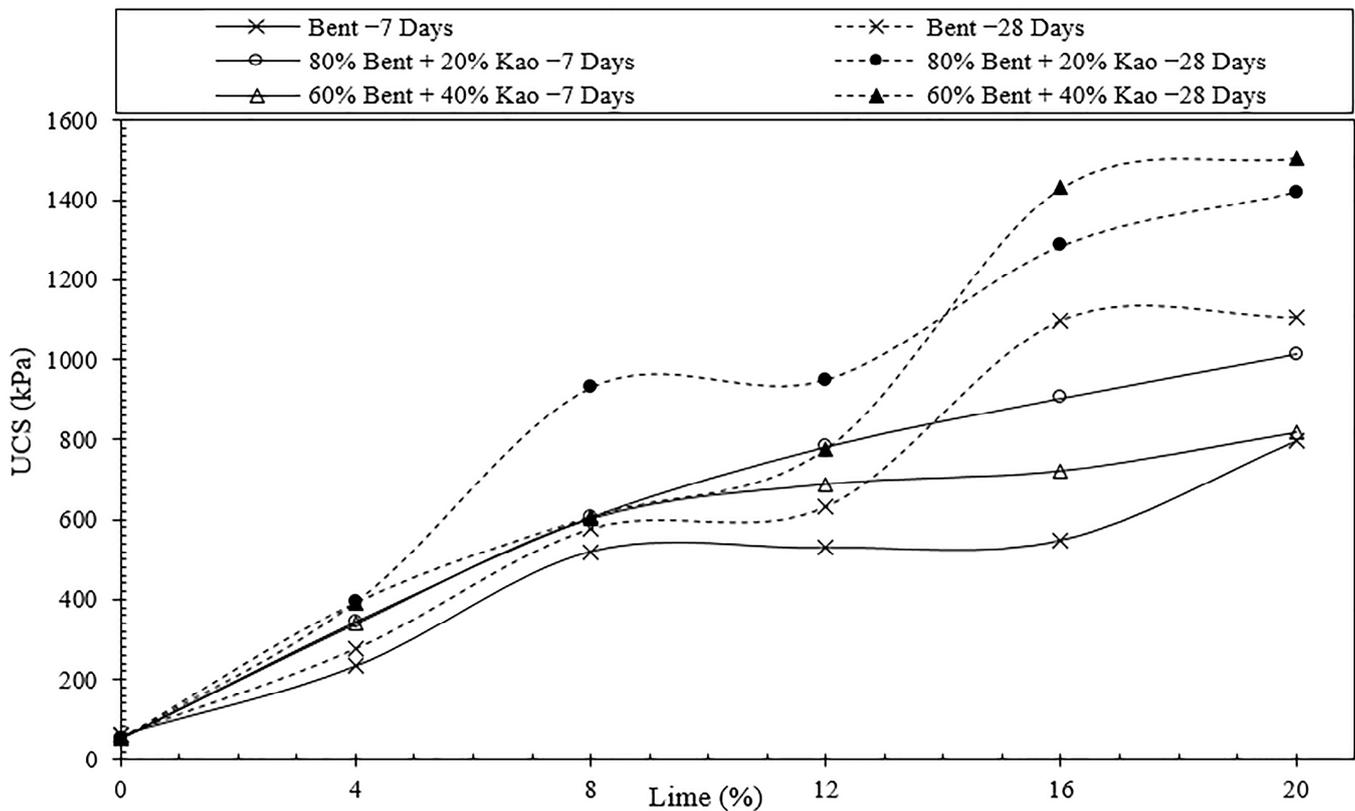


Figure 7. The UCS variation versus lime content for the samples with the Bent soil share of 60, 80, and 100% at the end of the 7-day and 28-day curing periods.

The addition of lime into clay soil, which resulted in immediate reactions, reduced the swelling potential and plasticity of stabilized samples compared with the untreated soil sample. The reduction in the tendency of lime-stabilized clay soil to absorb water occurs because of the immediate reactions of lime and clay soil. The sample with 4% lime lost its plasticity compared with the natural sample. In addition, the compressive strength of the 28-day sample increased by 18.6% compared with the 7-day sample. Adding 4% lime to Bent soil with 45 wt% montmorillonite mineral of the total amount did not cause pozzolanic reactions to improve in the long term. It soon broke up because of the brittleness caused by the plasticity properties modification with lime.

Upon adding 8% lime, the samples' UCSs for 7-day and 28-day curing periods increased by 121% and 107% compared with the samples with 4% lime, respectively. Only a tiny shell of the sample with 8% lime disintegrated at the end of wetting in the first cycle. Then, the sample was dried for 48 h. Severe shrinkage of the specimen during the first cycle of drying resulted in the creation of cracks throughout the specimen surface (see Figure 6c,d). As soon as the sample was immersed for the second cycle, water began to enter it through the cavities and cracks, and it collapsed.

The increase in the 28-day UCS compared with the 7-day for the samples with 8 and 12% lime was under 20%, but for the sample with 16% lime was 100.6%. The specimen with 16% lime showed no weight loss during the 48 h of soaking in the first cycle. After oven drying, the intensity of its cracks was much lower than the samples with 8 and 12% lime. This was due to adding 16% lime, which reduced the shrinkage and the crack propagation

on the sample surface. When this cracked sample was soaked in the water, it collapsed, but dissimilar from the sample with 12% lime, which disintegrated suddenly. According to Figure 7, the 28-day compressive strength of the sample with 20% lime remained almost constant compared with the sample with 16% lime. The durability of this sample was approximately similar to the sample with 16% lime.

3.2.2. 80% Bent + 20% Kao Soil Sample Pattern

When the untreated soil sample of this pattern was soaked in the water, similar to the untreated Bent soil sample, it began to absorb water because of the share of 80% Bent soil in its composition. Its moisture content increased from 40 to 143.6% and swelled after 48 h of wetting. Figure 7 shows the UCS variations of this soil sample pattern with lime after 7 and 28 days of curing. This soil combination's durability was almost similar to the Bent soil sample upon adding lime.

3.2.3. 60% Bent + 40% Kao Soil Sample Pattern

Based on Figure 7, the UCS of the 28-day samples of this soil combination increased slightly by 1 to 16% upon adding 4, 8, and 12% lime compared with the 7-day samples, which indicates a low degree of pozzolanic reaction. At 4, 8, and 12% lime, the samples collapsed when they were submerged in water for the second cycle. The sample with 4% lime at the end of the W/D of the first cycle is shown in Figure 8.

Upon adding 16% lime, the UCS of the 7-day sample showed a slight increase compared with the sample with 12% lime, and it collapsed when immersed in the water for the second cycle. Both the strength and durability of the 7-day sample indicated that the pozzolanic reactions did not improve in the short term despite the increase in lime by 4%. Based on Figure 7, the UCS of the 28-day sample with 16% lime increased by 84% compared with 12% lime. This sample collapsed at the end of the cycle but not immediately after immersion.

The UCS of the 7-day and 28-day samples with 20% lime had slight changes of 13.3% and 5.3% compared with the samples with 16% lime, respectively. The 7-day sample with 20% lime disintegrated at the end of soaking in the second cycle. Figure 8c shows it at the end of drying in the first cycle. The 28-day sample with 20% lime collapsed at the beginning of the immersion in the water for the fourth cycle (see Figure 8d). Therefore, the durability of the sample with 20% lime improved compared with the sample with 16% lime. When there was any content of montmorillonite mineral in the soil texture, the durability results gradually improved as the content of Kao soil or lime increased. In addition, the microstructural studies showed that the Kao soil contained clay minerals that could be considered pozzolanic materials. This issue was investigated in the following sections by progressively increasing the content of Kao soil in the soil composition.

3.2.4. 60% Kao + 40% Bent Soil Sample Pattern

Figure 9 shows the UCS versus lime inclusion of 60% Kao + 40% Bent soil pattern sample after curing for 28 days. The sample of this soil pattern with 4% lime, after 28 days of curing, collapsed when it was submerged in the water for the second cycle. Upon adding 8% lime, the strength of the 28-day sample increased by 155% compared with the sample with 4% lime. This sample collapsed at the end of 48 h of immersion in the second cycle. The sample of this soil pattern with 4% lime after drying in the first cycle and with 8% lime at the end of wetting in the second cycle is represented in Figure 10a,b, respectively.

With the addition of 12 and 16% lime, the 28-day strength remained almost constant compared with the sample with 8% lime inclusion (see Figure 9). Despite the consistency of the strength, this soil pattern sample with 12% lime lasted the second cycle and disintegrated in the third cycle, and with 16% lime lasted into the third cycle and disintegrated at the end of wetting in the fourth cycle. The addition of 20% lime caused a slight decrease in the 28-day strength compared with the sample with 8, 12, and 16% lime inclusion. The durability of this sample with 20% lime showed a more stable behavior than the sample

with 8, 12, and 16% lime. This sample lasted until the end of wetting in the fifth cycle. Therefore, as long as a certain amount of the montmorillonite mineral existed in the soil texture, a high content of lime was required to ensure the completion of more cycles in the durability process.

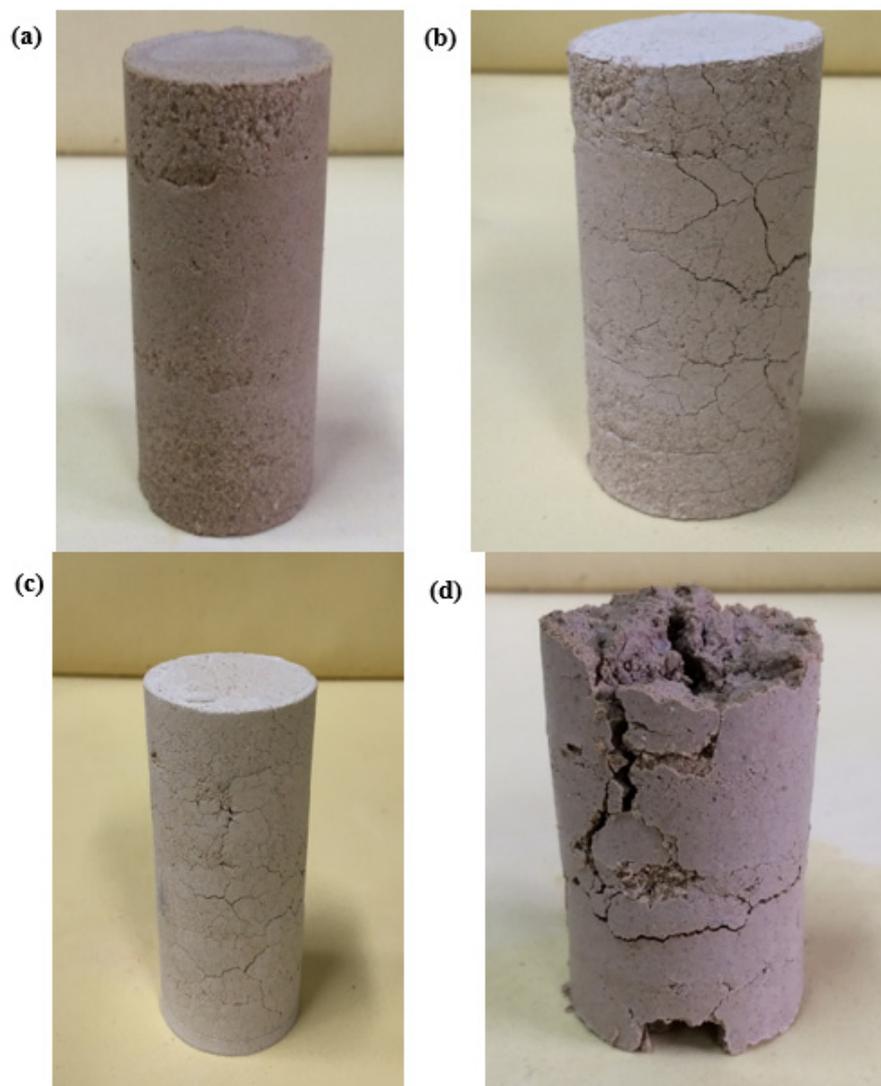


Figure 8. 60% Bent + 40% Kao + 4% L—28-day sample at the end of 48 h of (a) Soaking; (b) Drying for the first cycle; (c) 60% Bent + 40% Kao + 20% L—7-day sample at the end of 48 h of drying for the first cycle; (d) 60% Bent + 40% Kao +20% L—28-day sample which disintegrated when submerged in the water for the fourth cycle.

3.2.5. 80% Kao + 20% Bent Soil Sample Pattern

Based on Figure 9, upon adding 4% lime, its 28-day UCS became 11.5 times that of the natural soil sample. Without the addition of lime, the specimen was weak in both UCS and durability. In addition to the samples that were broken after 28 days of curing, other 28-day samples were made. If they lasted in durability, one series at the end of wetting in the third cycle and the other series at the end of wetting in the sixth cycle were subjected to the UCS test.

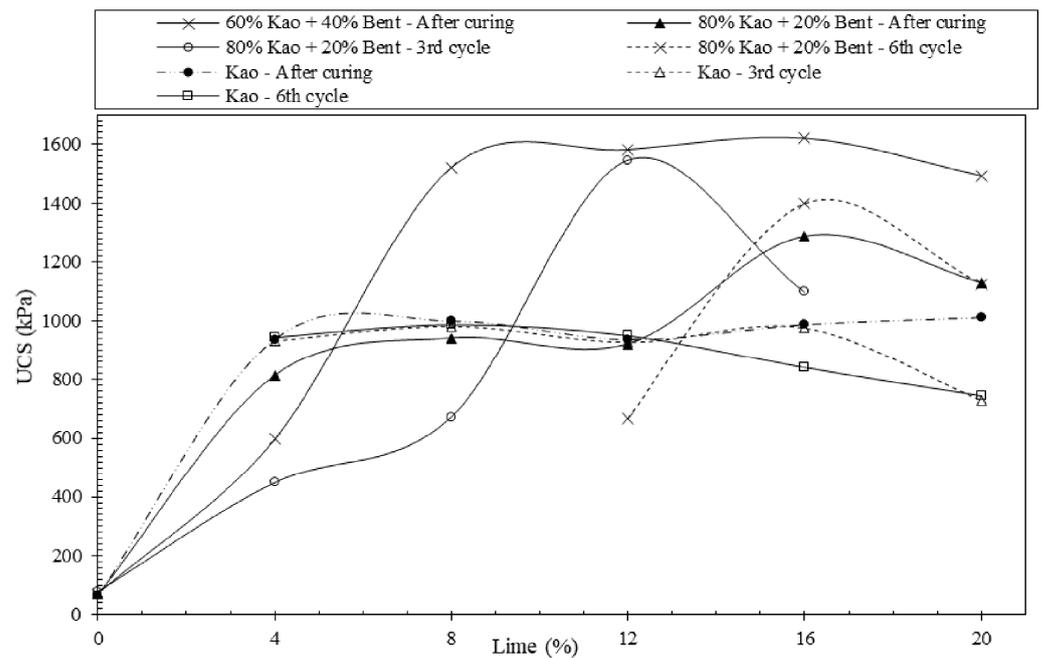


Figure 9. The UCS variations versus lime content for the samples with the share of 60, 80, and 100% of Kao soil at the end of the 28-day curing period and after wetting for the 3rd and 6th cycles.

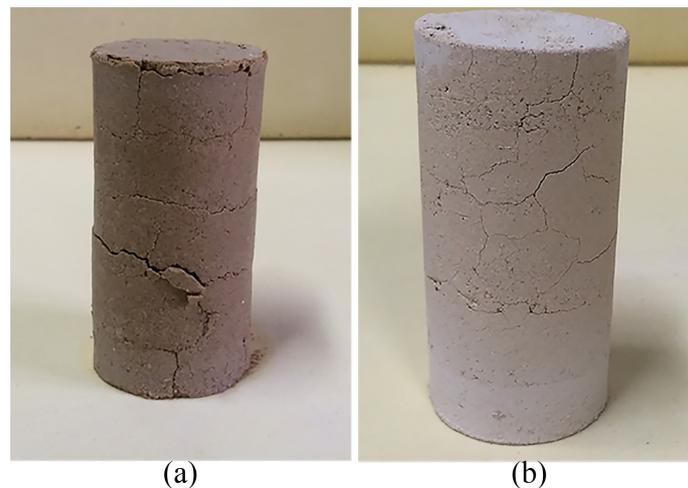


Figure 10. (a) 60% Kao + 40% Bent + 4% L—28-day sample after drying for the first cycle; (b) 60% Kao + 40% Bent + 8% L—28 day-sample disintegrated at the end of wetting for the second cycle.

A parameter called the water stability coefficient was used to evaluate the stability of the specimens based on the results of the UCS tests performed on the specimens after 28 days of curing and at the end of wetting in the third and sixth cycles. This parameter was in the form of Equation (1).

$$\text{Water Stability Coefficient}(\%) = 1 - \frac{\text{UCS}_{28 \text{ days}} - \text{UCS}_{w-n}}{\text{UCS}_{28 \text{ days}}} \quad (1)$$

where $\text{UCS}_{28 \text{ days}}$ and UCS_{w-n} are unconfined compressive strengths of the soil samples after 28 days of curing and after wetting in the n th cycle, respectively. The water stability coefficients versus lime content for the samples of Kao and 80% Kao + 20% Bent soil for the 3rd and 6th cycles are presented in Figure 11. Both specimens with 4 and 8% lime lasted through the third cycle and collapsed when immersed in water for the fourth cycle. The

samples with 4% lime at the end of wetting and drying in the second cycle are shown in Figure 12a,b, respectively. This sample at the end of wetting in the third cycle is depicted in Figure 12c. Based on Figure 9, the UCS of the sample with 8% lime increased slightly compared with the sample with 4% lime. As illustrated in Figure 11, its water stability coefficient in the third cycle was 4.5 times that of the sample with 4% lime.

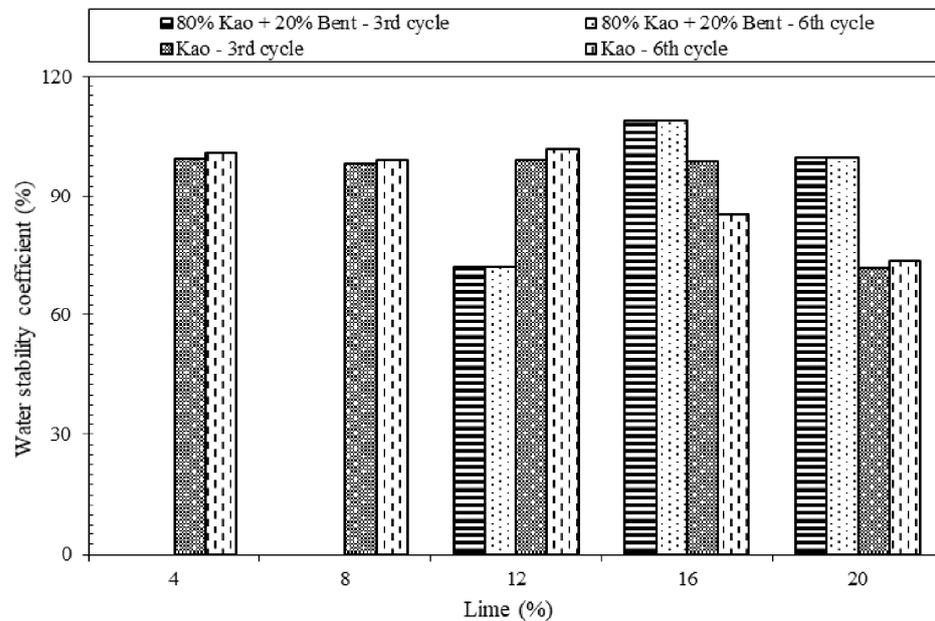


Figure 11. Water stability coefficient (%) variations versus lime content (%) for Kao and 80% Kao + 20% Bent soil sample at the 3rd and 6th cycle.

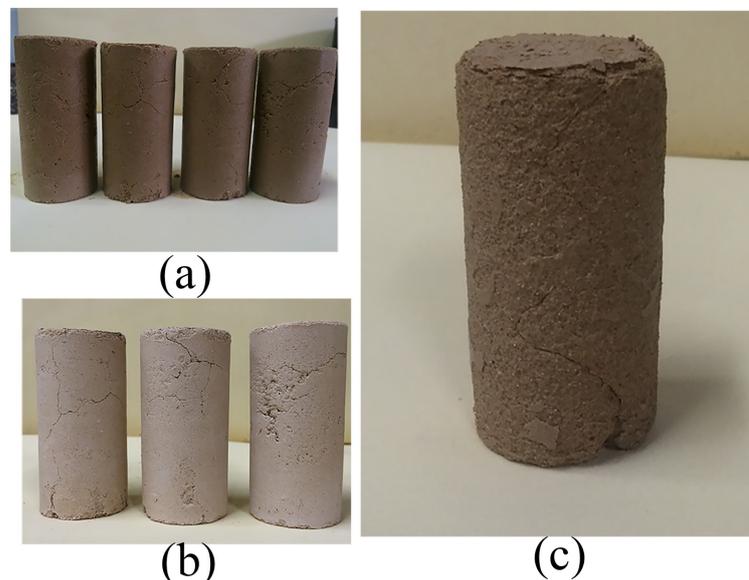


Figure 12. 80% Kao + 20% Bent + 4% L sample after (a) Soaking; (b) Drying for the second cycle; (c) 80% Kao + 20% Bent + 4% L—28-day sample at the end of wetting for the 3rd cycle.

Although the UCS of the sample with 12% lime remained almost constant compared with the sample with 8% lime, the sample durability improved significantly and lasted until the sixth cycle. The water stability coefficient of the sample with 12% lime for both the third and sixth cycles was 73%. Based on Figure 9, the uniaxial strength of the sample with 16% lime increased by 25% compared with the sample with 8% lime, which was not

significant. Therefore, the optimum lime content for the after-curing compressive strength of this soil mixture was 8%.

As can be detected in Figure 11, the water stability coefficients of the samples with 16% lime for the third and sixth cycles were 120% and 109%, respectively. This means that the compressive strength for the third cycle was higher than the after-curing compressive strength. The water stability coefficient values of the samples with 20% lime for the third and sixth cycles were 97.3% and 100%, respectively, which decreased compared with the sample with 16% lime. Therefore, the optimum lime content in the durability tests for this combination was 16%.

3.2.6. Kao Soil Sample Pattern

The Kao soil contained 15 wt% kaolinite mineral and 25 wt% mixed layer of illite–montmorillonite mineral of the total amount by approximately 95% illite and 5% montmorillonite (see Table 3). According to Figure 13a, the Kao soil sample collapsed immediately after being submerged in water. The value of its PI was 11.3%. Therefore, the clay soil did not have significant adhesion, and without the presence of lime as a binder of soil particles, it collapsed immediately. Upon adding 4% lime, the UCS of the sample cured for 28 days became 14 times that of a natural soil sample. Unlike all other soil combinations, the Kao soil sample with 4% lime progressed significantly and lasted until the sixth cycle. Figure 13b shows the sample with 4% lime at the end of wetting in the sixth cycle.

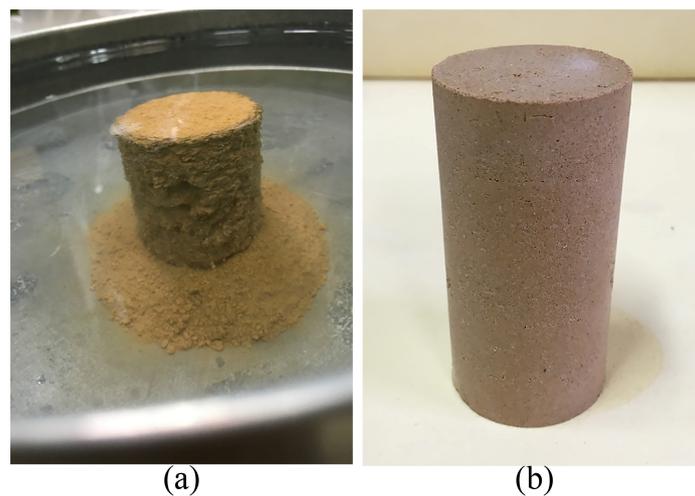


Figure 13. (a) The disintegration of the natural Kao sample immediately after immersion in water; (b) Kao + 4% L—28-day sample at the end of wetting for the 6th cycle.

The kaolinite mineral tends not to absorb water and does not swell when water reaches it [38,39]. Therefore, the kaolinite minerals are non-swelling. The ingress of H₂O into the formation of illite is avoided by the K, Ca, or Mg interlayer cations [40]. Weak hydration of these cations fills the illite interlayer space, which causes a lack of swelling characteristics [41]. Therefore, illite is ranked as a non-swelling or low-swelling mineral [42]. The total content of clay minerals in the Kao soil was 40%, which approximates non-swelling minerals. The non-swelling clay minerals in Kao soil reacted with lime in the best possible way. The strength of the sample after 28 days of curing was so high that the sample lasted until the sixth cycle.

Upon adding 8, 12, 16, and 20% lime, their 28-day UCSs increased slightly compared with the sample with 4% lime (see Figure 9). With the addition of lime content higher than 4%, the compressive strength remained almost constant. On the other hand, based on Figure 11, upon adding 8 and 12% lime, the durability coefficients of the third and sixth cycles remained almost constant compared with the sample with 4% lime. As shown in Figure 11, the water stability coefficients fell in the sixth cycle for the samples with 12, 16,

and 20% lime because of the negative effect of excess lime on the reaction with Kao soil. Therefore, the optimum content of lime for both the UCS and durability tests was 4%.

The strength of Kao samples upon adding lime initially had an upward trend and then decreased. Based on the results of SEM-EDX analysis of this study as well as previous studies [43,44], the soil strength in the presence of a large amount of lime decreased because of the complete dissolution of clay particles and a lack of sufficient silica and alumina in the system for the continuity of pozzolanic reactions. Under these conditions, some of the lime particles remained free in the chemical reactions and reduced the strength of the samples because of their low friction and adhesion. Therefore, it was practically possible to increase the strength of lime-stabilized samples up to a certain limit, and the increase in the amount of lime would not affect the further rise in the strength.

3.3. Volume Changes Assessment of Lime-Stabilized Kao Soil in the Durability Process

When the untreated Kao soil sample was submerged in the water, it collapsed without absorbing water and swelling. The untreated Kao soil did not have the adhesion of swollen clay minerals to maintain its cohesiveness in the water. The volumetric strain values of swelling during wetting and shrinkage during drying in each cycle for the lime-stabilized Kao soil samples are given in Figure 14. The sum of the total amounts of swelling strain during wetting and shrinkage strain during drying in each cycle for a sample was considered the total volumetric strain (TVS).

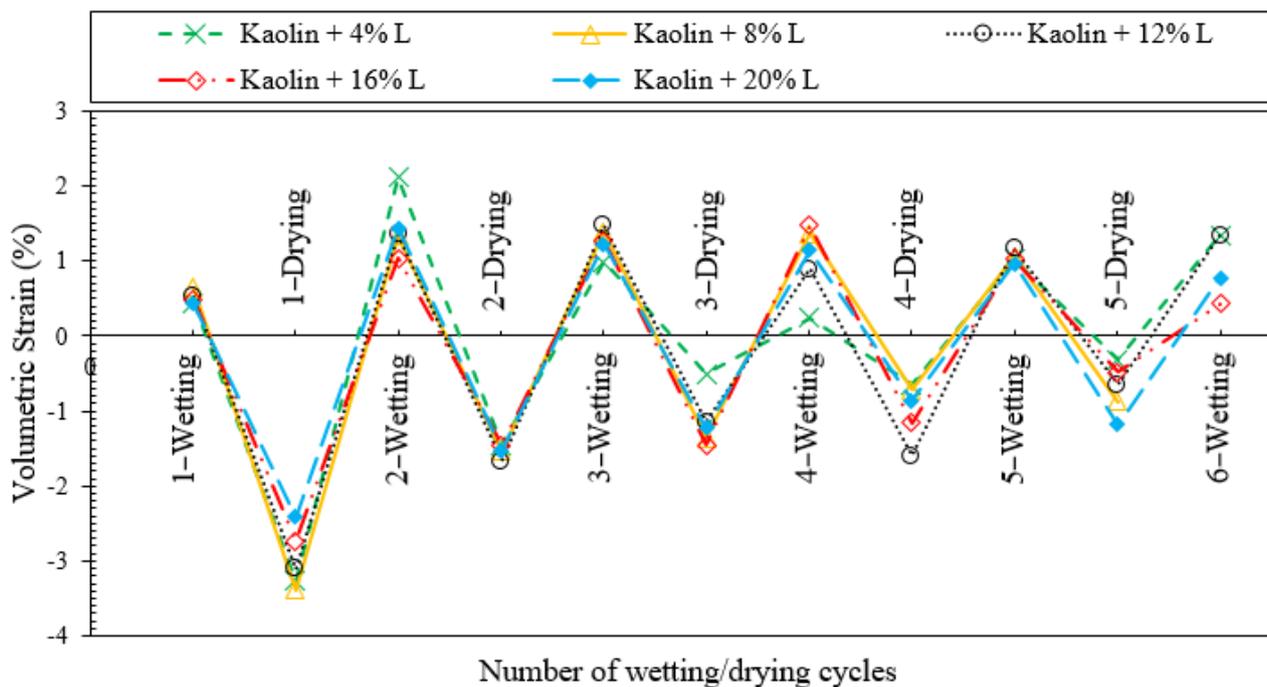


Figure 14. The volumetric strains amount of lime-treated Kaolin soil samples after each cycle of wetting and drying.

For all of the lime-stabilized Kao samples, a maximum TVS was exhibited in the first cycle. For the subsequent cycles, the TVS of the samples decreased. The lowest TVS occurred in the sixth cycle. Therefore, the first cycle of wetting and drying had the most significant effect on the samples' TVS.

The maximum TVS of the sample with 4% lime was approximately 4%. This small amount of TVS showed that the Kao soil did not contain swollen clay minerals and participated in the best possible way in the pozzolanic reactions with 4% lime. The maximum TVS of the samples with 8 and 12% lime compared with the sample with 4% lime varied by almost less than 9%. For the samples with 16 and 20% lime, the maximum TVSs were

approximately 14 and 24% less than the sample with 4% lime, respectively. Therefore, the TVVs of the samples with lime content from 8 to 20% had slight changes compared with the sample with 4% lime.

The TVV of lime-stabilized Kao samples was low during W/D cycles. Therefore, adding lime to more than 4%, even up to 20%, had little effect on the amount of the TVV. In addition, according to Figure 14, for all samples, on average, 85, 50, and 40% of the TVV was related to the shrinking during drying in the first cycle, second to fifth cycles, and the sixth cycle, respectively.

3.4. Weight Loss Assessment of Lime-Stabilized Kao Soil in Durability Process

When the sample with 4% lime was subjected to a weight loss process with a wire scratch brush in the drying phase in each cycle, it lasted all six cycles. In each cycle, some wire brushstrokes were applied to the sample. The strength and rigidity of the sample decreased. Finally, the sample had less stiffness and more weight loss in the sixth cycle. For all lime-treated samples, the weight loss rate in the sixth cycle was higher than the other cycles. Figure 15 shows the sample with 4% lime in the soil–lime weight-loss process. The total weight loss content after six W/D cycles for the sample with 4% lime was approximately 20% by dry weight. In addition, for the samples with 8 to 20% lime, it was approximately 20% by dry weight. The pozzolanic reactions of Kao soil with 4% lime caused cementation compounds whose solidity and durability in the weight loss process were similar to that of the samples with 8 to 20% lime.

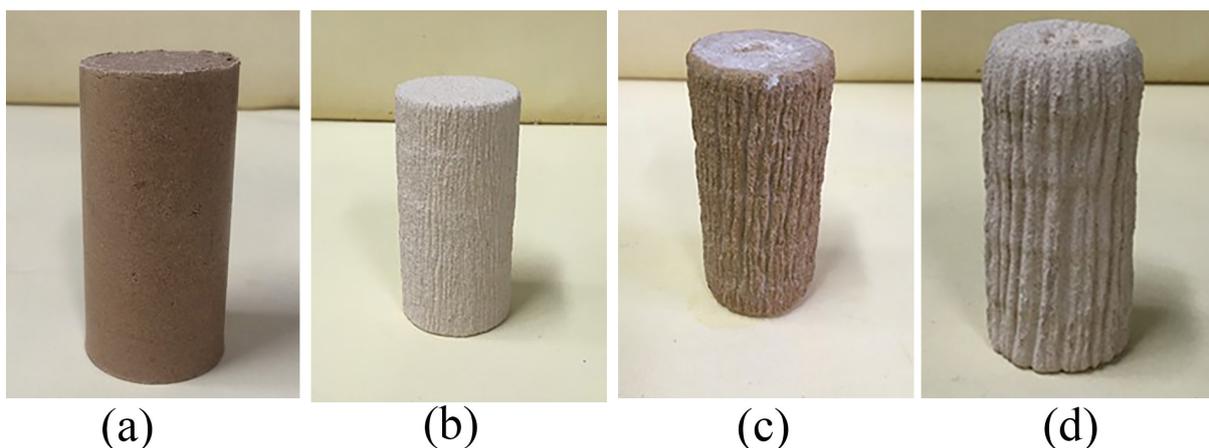


Figure 15. Kao +4% L sample (a) after 28 days of curing (b) wire scratch brushed at the end of the drying phase of the first cycle (c) wire scratch brushed at the end of the wetting phase of the sixth cycle (d) wire scratch brushed at the end of the drying phase of the sixth cycle.

3.5. Comparative Assessment of the Durability Results of Lime-Stabilized Samples

In the process of durability, soil sample patterns in which Bent soil with 45 wt% montmorillonite mineral had a share of 40 to 100% by dry weight of the soil, no specimen lasted until the sixth cycle and collapsed before the sixth cycle even with the highest content of lime. Adding lime increased the durability, but if the Kao soil content with approximately 40 wt% non-swelling clay mineral in the soil mixture pattern were less than 80% by dry weight of the soil, even upon adding 20% lime, it would not last until the sixth cycle.

This finding indicated the influence of the types and amounts of clay minerals on soil stabilization with lime to withstand W/D cycles. Provided that the Kao soil content with approximately 40 wt% non-swelling clay minerals in the soil sample pattern was 100% by dry weight of the soil, the inherent nature of non-swelling clay minerals by lime stabilization caused this sample to last in durability test in the best possible way even with 4% lime. When the share of Kao soil was 60% or more (but not 100%) by dry weight of the soil, the addition of lime more than the optimum content of the UCS test improved the

durability of the sample to last more cycles. Provided that the share of the Kao soil in the pattern was 100% by dry weight of the soil, the addition of lime in amounts higher than the optimum level did not improve the sample's behavior in the durability test. It reduced the sample's water stability coefficient after wetting for 48 h in subsequent cycles.

4. Conclusions

This study investigated the microstructural characteristics of lime-stabilized Bentonite and Kaolin soils. These soils were mixed at varied ratios, stabilized, and then subjected to the durability process. The quality of stabilization depended on the types and amounts of clay minerals in the soil. Kaolinite and illite minerals of Kaolin soil could be considered as pozzolanic materials. According to the microstructure results, it was expected that Bentonite soil had no durability with any content of lime. The main results can be summarized as follows:

1. The optimum lime content for the clay soils depended on the content and type of clay minerals.
2. The macro-structural behavior of clay soils stabilized using lime depended on their micro-structural properties and the interactions at the micrometer scale.
3. The consistency between the results of durability tests against W/D cycles and micro-structural results, including mineralogical analysis of untreated Kao and Bent soils with XRD test, SEM images, and quantitative EDX analysis of clay samples before and after stabilization, was very suitable.
4. The positive efficiency of stabilization of Kao soil with lime in the process of durability against W/D cycles could be inferred from the SEM micrographs by changing its structure from laminate to flocculated, reducing the pores and large cavities, and agglomerating the soil texture after stabilization.
5. Based on the EDX results, the amounts of the predominant clay minerals of Kao soil, including kaolinite and illite, were such that they reacted with lime in the best possible way. They drastically reduced the content of lime for chemical stabilization by having sufficient elements needed for the pozzolanic reactions. The results of the durability tests confirmed this, as the optimum lime content for Kao soil became 4% in this process. The clay soils containing a certain amount of illite or kaolinite without any content of swelling minerals are susceptible to stabilizing with the minimum amount of chemical additives and have the best performance.
6. The proper reactions of kaolinite and illite minerals with lime and the formation of cementing compounds resulted from the occurrence of the pozzolanic reactions at a high level. These minerals could be called pozzolanic materials that significantly reduce the optimum lime content.
7. Based on the results of EDX analysis and SEM images, the intrinsic nature and the amount of montmorillonite mineral of Bent soil were such that it did not have sufficient elements to occur the pozzolanic reactions with lime. Therefore, in the process of durability against wetting and drying cycles, the stabilized bentonite soil did not last with any percentage of lime. In addition, if the weight content of Bent soil in the soil composition was more than 40%, its stabilization with lime for durability against W/D failed. It is necessary to use other additives rather than lime.

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