

## Review Article

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# The significance of scanning electron microscopy (SEM) analysis on the microstructure of improved clay: An overview

<https://doi.org/10.1515/geo-2020-0145>

received May 15, 2020; accepted December 01, 2020

**Abstract:** This study aims to emphasize the importance of scanning electron microscopy (SEM) in explaining the differences in the physical and mechanical behaviors of the improved clays before and after improvement. Generally, clays appear as problematic soils in the construction area. The reasons for this can be low strength, high compressibility, high level of volumetric changes, and swelling potential. The behavior of clays may not always be interpreted according to their physical and mechanical properties. In such cases, analyses are carried out according to the microstructure of the clay. Among several microstructural analyzes, SEM is very important, because the soil microstructure formed by clay particles and additives can be observed. This work discusses the studies based on the microstructural properties of the improved clay with SEM analysis. As a result of the studies carried out, it has been seen that unexplained physical or mechanical behavior can be explained by the microstructural behaviors of clay particles and additives.

**Keywords:** clay, geotechnical properties, microstructural, scanning electron microscopy

## 1 Introduction

The soils are the layer of material covering the ground surface and are formed from the weathering of rocks. Degradation and other geological processes that are effective on the surface of the earth's crust or rocks close to the surface enable soil formation. This processing consists of physical and chemical weathering. Decomposition caused

by atmospheric effects changes the structure and composition of rocks chemically and physically. Physical or mechanical degradation causes the rocks to split into smaller pieces. The factors that cause physical degradation are freezing and thawing, heat exchange, erosion, and activities of plants and people [1–7].

Chemical degradation means the changes in the composition of minerals in rock with oxidation, reduction, carbonation, and other chemical processes. Generally, chemical degradation is more important than physical degradation in soil formation [8–10]. Typically, soils are named as gravel, sand, silt, and clay based on the particle size in geotechnical engineering design. The geological history of soils determines its engineering behavior [11]. Gravels, sands, and silts are formed by the results of physical and moderate chemical weathering processes. Gravels are small pieces of rock and contain several minerals. Sands are smaller particles, and usually, each particle consists of a single mineral. If it is found difficult to see an individual particle that make up the soil, then the soil is silt or clay or consists of a mixture of these [12–16].

Natural soils are a mixture of particles of many different sizes and sometimes contain organic matter. Some soils, such as peat, can be almost entirely organic. Since the soils are a special material, they have voids inside, and these voids are often filled by water and air. What makes the soils' behavior so complex is the physical and chemical interaction of water and air in the cavities with the soil particles. Taking into account all these variabilities and heterogeneities, it can be seen that the soils are complex engineering and building materials [1]. The properties of the fine-grained soils like silt and clay are predominantly characterized by their behavior rather than the size. Therefore, to make this term more useful, it is classified using soil classification systems (Unified Soil Classification System, etc.). Thus, by understanding the behavior of soils, engineers can use such soils for required cases [17–19].

The standard classification system that is commonly used nowadays in civil engineering applications is the

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Unified Soil Classification System (USCS) [19] and the classification systems of the American State Highway and Transport Office Association (AASHTO) [20]. In some countries, in addition to this system, own country standards are also used. Hence, it can be predicted how the encountered soil will behave in the field [21]. With the identification of clayey soils at the site, one can efficiently ensure proper construction practices required for it.

One of the most important instruments for the analysis of microstructural characteristics of soils is the scanning electron microscope (SEM). The primary reason for the SEM's advantage in investigating soil microstructure is the high resolution that can be obtained when examined. Thus, SEM has been an important instrument in investigating clays. SEM analysis helps us to identify the bonding structure of the additives with the clay particles. In this discussion, the importance of SEM analysis with regard to clay and improved clay has been shown.

## 2 Definition and properties of clay

Clay minerals are very small crystalline secondary minerals formed due to the chemical decomposition of some rock-forming minerals. The common clay mineral groups include kaolin, illite, and montmorillonite [22–24]. Kaolinite consists of silica and alumina plates alternately. These plates are connected very strongly. Montmorillonite has layers made from two silica plates and one alumina plate. The bond between these layers is very weak, so large quantities of water can easily enter the structure and separate them. Illite has layers made from two silica plates and one alumina plate. Illite has layers similar to those of montmorillonite but contains potassium ions between each layer. This characteristic makes the clay's structure stronger than the montmorillonite, but weaker than the kaolinite [21–23,25–28].

Clays occur in small particle sizes ( $<0.002$  mm). Therefore, clay minerals cannot be seen with the naked eye, and an SEM image is required for a scientific study. Since the clays are unaffected by gravitational forces but rather are affected by surface forces, it should be tested accordingly. In general, the shape of the clays is similar to the plate. Due to the shape of the clay, the mass ratio of the surface area is quite large. This ratio is known as the specific surface area. The specific surface area of the clay is larger, so this state causes more area of contact between the particles and the development of interparticle forces. The electrical charge on the clay interacts with the water dipole; finally, clay absorbs more water.

Thus, clay soils especially with water cause problems such as excessive settlement and volume change. Since physical processes of clay are directly related to specific surfaces, the specific surface area is important for clay. However, to identify the clays, XRD analysis with the specific surface area should be performed. XRD analysis enables the identification of different minerals with different crystal structures [29].

Due to the unbalanced force fields on the surfaces of clay minerals, there are physical–chemical interactions between water, dissolved ions, and clay minerals. The interaction of these forces affects the engineering behavior of soils and the microstructure of clays. Clay minerals are hydrated in nature, and this is defined as adsorption [30]. Since the clay surface is negatively charged, the water molecule, which is a dipole, is attracted to the surface. This adsorbed interaction forms the physical basis of the soil structure. Due to the interaction of clays with adsorbed water, the presence of different ions, different organic materials, and different components creates differences in soil structures [21].

Generally, clayey soil is identified with some fractions of silt and/or sand at the site. Rarely, we do find a pure clay soil alone. Clay or clayey soils are identified based on their cohesion or plasticity rather than the size. Silt, sand, and gravel are the result of the physical and moderate chemical degradation processes, while clayey soils are the result of the chemical degradation process. This situation explains why the engineering properties and behavior of clays are different [31].

Geotechnical practice requires the knowledge and experience with clay; as it varies with different conditions at sites such as road construction, dams, slurry walls, airports, and waste landfills. Clay soil is problematic soil due to its low strength, high compressibility, and high level of volumetric changes. In such cases, the behavior of the soil is controlled by the properties of the clay and the percentage of the clay minerals presenting within the clayey soil. When the clay content is about 50%, sand and silt particles are like particles floating in the dough, and their effects on engineering behavior are meager. The presence of water also significantly affects clay soil behavior. Furthermore, the relative positions of clay particles in a soil mass also affect mechanical properties.

## 3 Microstructural properties of clay

Clay minerals are collected in different ways to form clay soils. Clay particles repel each other electrostatically,

depending on the ion density and intergranular distance. Simultaneously, a clay particle is under the influence of an attraction due to the hydrogen bond, van der Waals forces, and other chemical bonds. Particles can attract (lump) or push (disperse) each other. Clumping is seen as edge-to-edge, edge-to-face, or face-to-face clumps about flocculent and dispersive structures [32,33].

In geotechnical engineering, the soil structure is defined by the soil particles' geometric arrangement and the forces between these particles. Soil fabric refers only to the geometric arrangement of the intergranular forces that are very large in fine-grained cohesion soils. Therefore, these forces and fabrics are handled together in the structure of fine-grained soils. For a complete description of the structure of fine-grained cohesion soils, information on the interparticle forces and the geometrical arrangement of the particles should be available. Since the force fields between the particles are complicated to measure, only the soils' fabric is examined in the studies on the cohesive soil structure, and the forces between the particles are interpreted based on these textural features [34–40].

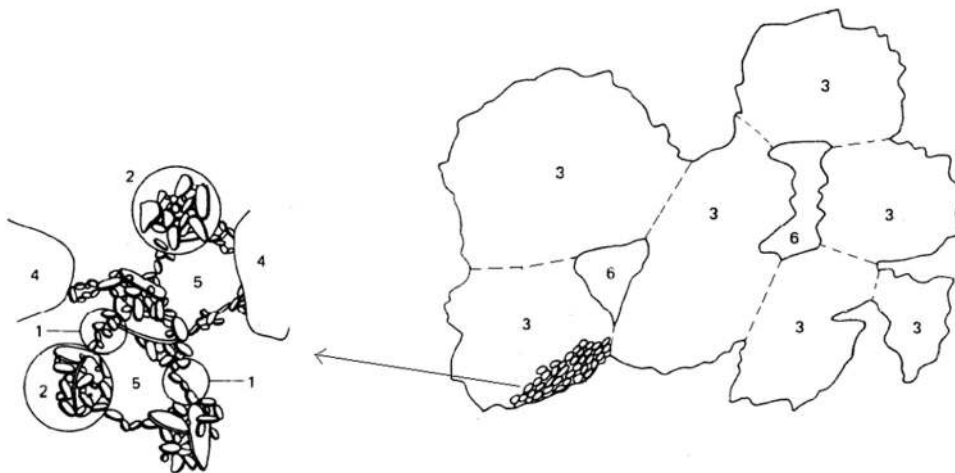
Many researchers proposed fabric models for clay particles [8,21,22,37,41,42]. Terzaghi introduced the clay arrangement concept in 1925 and indicated that clay minerals stick to each other at the points of contact, with forces sufficiently strong to construct a honeycomb structure. Honeycomb shape is a special structure in clay-containing soils, and this structure can vary depending on many characteristics of the environment [21]. Push [8] and Yong and Sheeran [37] proposed the fabric model in Figure 1. According to Figure 1, domain (1) is the structure formed by clay particles, the cluster (2) is the structure formed by the domains gathering around silt and clay, and the pad is shown as clumps (3). Among the lumps are

macropores, which can also be selected visually. Figure 1 also shows micropores (5), macropores (6), which are not visible between the silts and sand particles (4), and the bundles. Figure 2 shows the differently arranged clay particles [43]. This figure is fabric composed of (a) randomly orientated clay particles; (b) randomly orientated domains; (c) domains in parallel alignment; (d) parallelly orientated clay particles with little differentiation of domains; (e) granular particles of iron oxide, organic matter or fine silt interfere with the parallel orientation of the clay particles; (f) randomly orientated domains may occur between particles of coarse slit; and (g) large sand particles with clay particles aligned tangential to their surfaces. The structure of soils can be examined more extensively by SEM. Figure 3 displays the SEM images of kaolinite, montmorillonite, and illite [21].

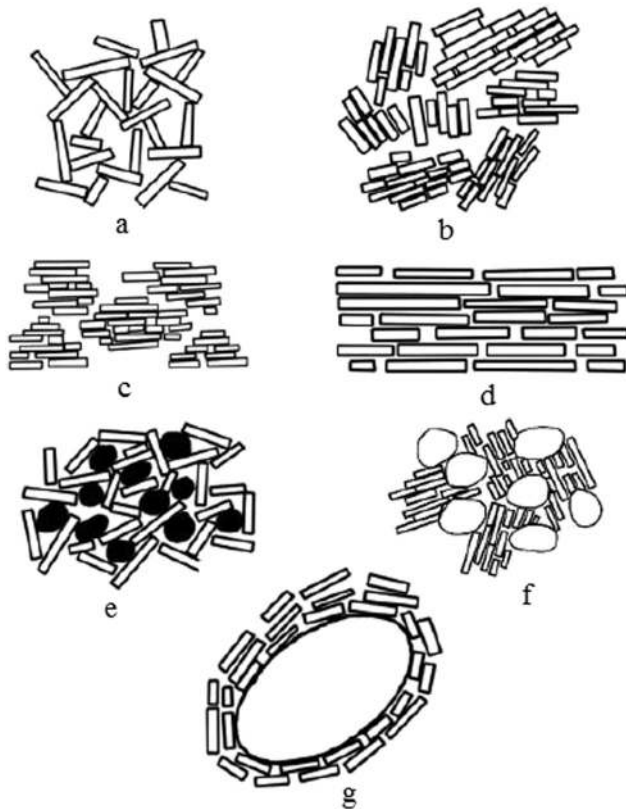
## 4 SEM analysis

The SEM gives the magnified images of the size, shape, composition, crystallography, and other physical and chemical properties of a specimen. The principle of the SEM was initially demonstrated during 1935 and 1939 by Knoll [44,45]. Later, SEM was developed by von Ardenne [46]. The modern commercial SEM studies continued with extensive development in the 1950s and 1960s by Charles Oatley and his many students at the University of Cambridge [47]. McHardy and Birnie [48] gave historical developments along with its application to clay for SEM.

SEM material sciences and surface sciences are magnified many times to view the surface structures and evaluate the differences on the surface [49–51]. SEM offers the



**Figure 1:** Soil micro and macro fabric (1) structures formed by clay particles, the cluster (2) structure formed by the domains gathering around silt and clay, and the pad is shown as clumps (3) among the lumps are macropores, which can also be selected visually. (5) Micropores (6), macropores (4), the silts and sand particles [8,37].

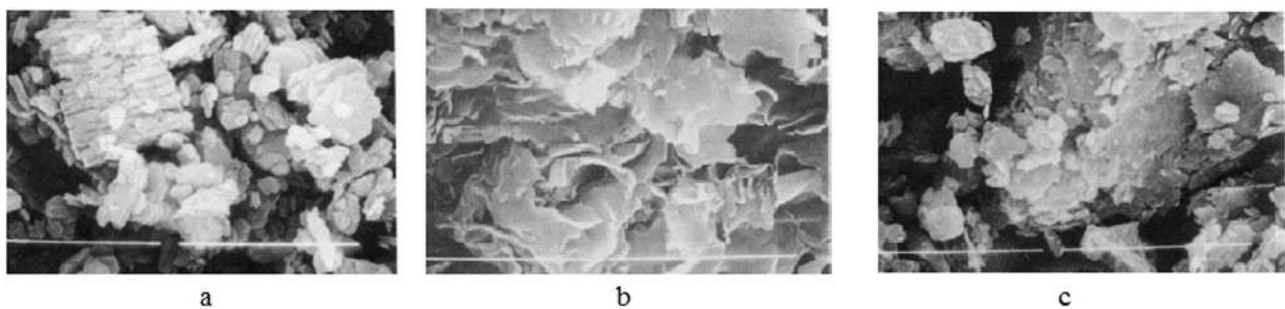


**Figure 2:** Differently arranged clay particles [43]. Fabric composed of (a) randomly orientated clay particles, (b) randomly orientated domains, (c) domains in parallel alignment, (d) parallelly orientated clay particles with little differentiation of domains, (e) granular particles of iron oxide, organic matter or fine silt interfere with the parallel orientation of the clay particles, (f) randomly orientated domains may occur between particles of coarse silt, and (g) large sand particles with clay particles aligned tangential to their surfaces.

opportunity to analyze the material to be examined with an electron beam generated in a vacuum environment and thinned with electromagnetic lenses in the same situation to create a high-resolution image. The images found in the microscope are created by counting the

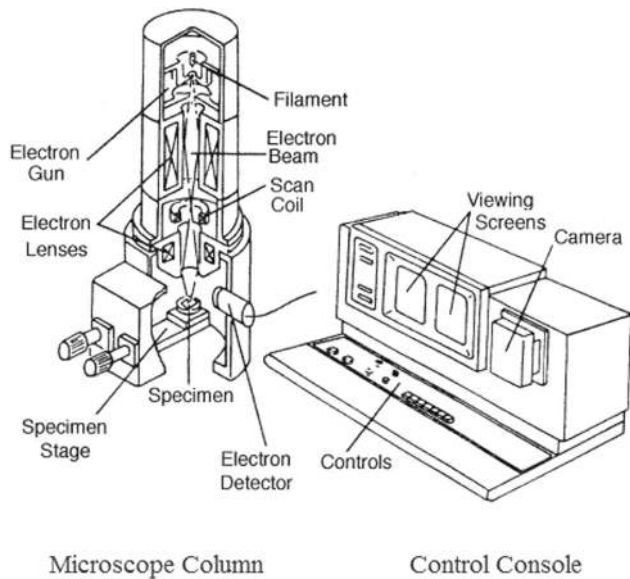
reflections or electrons reflected from the interaction of the electron beam with the material.

The SEM instrument comprises the electronic console and the electron column (Figure 4) [52]. There are control knobs and switches that allow instrument adjustments such as filament current, accelerating voltage, focus, magnification, brightness, and contrast in the electronic console. All the primary controls are accessed using a computer. Images captured are saved in digital format. The electron beam is generated under vacuum, focused to a small diameter, and scanned across the surface of a specimen by electromagnetic deflection coils in the electron column. There is a specimen chamber in the lower portion of the column. The secondary electron detector is located above the sample stage inside the specimen chamber. The specimens are mounted with a goniometer to be secured onto the scene [53]. The SEMs have facilities for detecting secondary and backscattered electrons, and knowing the location is essential. Usually, one side of the chamber is to locate the movable X-ray detector, and during microanalysis, it is moved close to the sample. Figure 5 [55] shows the secondary, backscattered, and X-ray detectors' relative position to a specimen in an SEM [54,55]. Furthermore, using an energy dispersive X-ray spectrometer (EDS) with the SEM elemental composition are determined [56]. EDS uses the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to get a localized chemical analysis. Thus, when obtaining electron images by the SEM, element mapping and point analysis can also be obtained by adding an X-ray spectrometer [54,57]. Thus, qualitative and quantitative analyzes can be made with a point, line, and area scan determined by EDS (Figure 6). For this reason, many researchers do the elemental analysis of the points or regions they choose while doing SEM analysis in their studies [58–60]. Briefly, information on topography, morphology, shape, size, composition, and crystallographic structures of materials such as ground,



**Figure 3:** SEM image of clay. (a) Kaolinite, (b) montmorillonite, (c) Illite (length of the distance between the white lines 0.5  $\mu\text{m}$ ) [21].





**Figure 4:** The two major parts of the SEM, the electron column, and the electronics console [52].

Besides, the preparation of samples is very important in a way that does not damage the soil structure. Otherwise, it may alter the soil's structure [62]. SEM is known in science as different specimens can be examined using it. Sample size may be prepared depending on the type of the material. The surface of the sample must be clean. Besides, the sample size must be of dimensions that can be accommodated within the specimen chamber of the SEM. It is essential to make the sample sizes correctly, especially in obtaining SEM photographs accurately on some sensitive soils. Thus, the surface structure of materials is analyzed correctly [55,63].

The character of the soil microstructure can be seen over a wide range of magnification by SEM. Generally, SEM analysis is employed to facilitate the microstructural comparison and explanation. In the SEM images, a microscope is used to qualitatively identify the microstructural developments in the matrix of the stabilized soil specimens. The SEM images of clays are shown in Figure 7 (different magnifications); thus, the microstructure is easily observed since the pictures can be enlarged.

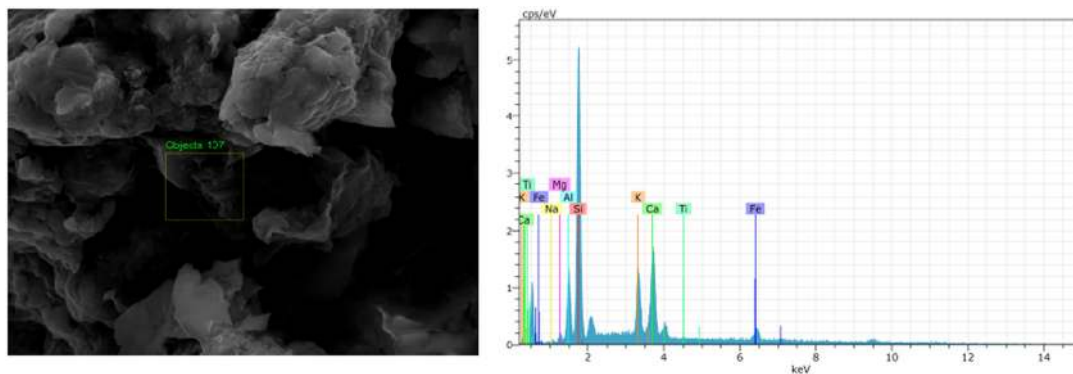


**Figure 5:** The position of secondary, back-scattered, and X-ray detectors [55].

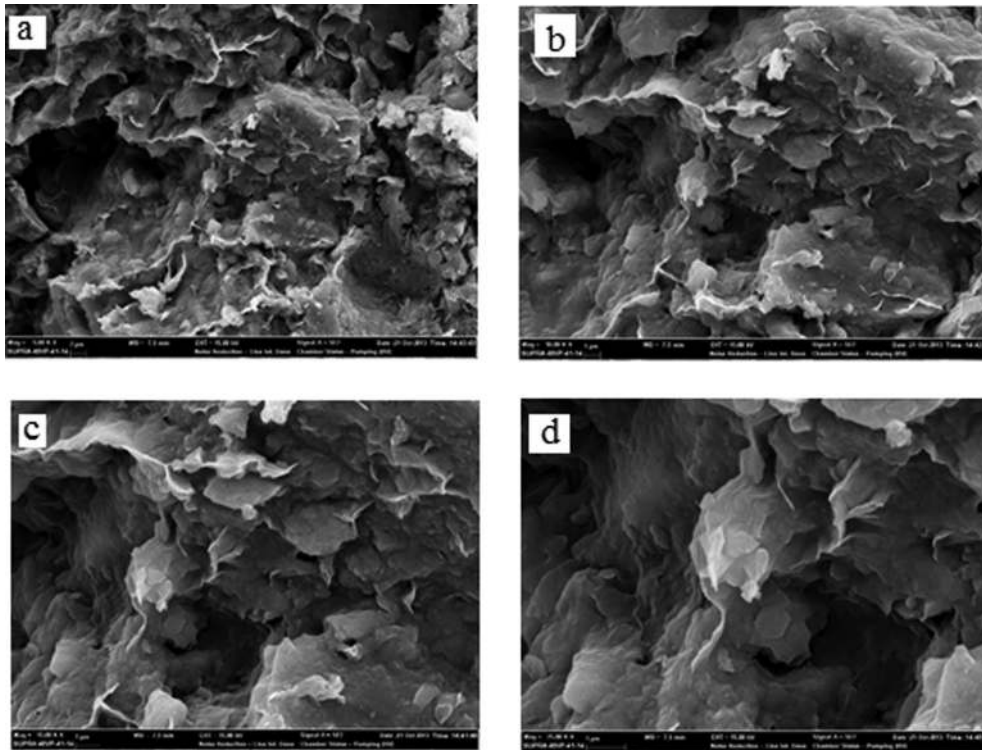
## 5 Improved clay

rock, ceramic, glass, metal, polymer, and local crystal structure is obtained with SEM [61].

As a result of an increase in the population worldwide and decrease in areas with high-bearing capacity, some buildings are built on lands with weak bearing capacity. Geotechnical engineers want it to have the carrying power to keep the load on the soil bearable and at desired seating values. Other physical and/or chemical stabilizations are required. Stabilization processes cover the operations performed to bring the unit weight, strength, and hydraulic conductivity to the desired level. The geotechnical engineer, with the soils requiring stabilization, often encounters the design and construction of land



**Figure 6:** SEM-EDS analysis with area scan of clay.



**Figure 7:** SEM images of a clay. (a)  $\times 5,000$  magnification, (b)  $\times 10,000$  magnification, (c)  $\times 15,000$  magnification, (d)  $\times 25,000$  magnification.

and railway embankments, airports, soil and rock-filled dams, graves, and water channels [21]. Improving the soil is modifying the soil, expediting construction, and improving the soil's strength and durability [25,64,65]. Also, nowadays there are problems due to the increasing demand for construction in coastal and offshore areas involving marine clay. Marine clay usually consists of different soil minerals (montmorillonite, illite) and other stone minerals (quartz, feldspar) that are all bonded together by the presence of organic matters [66,67]. There are also many studies related to change in the properties of this clay by different additive materials [68–73].

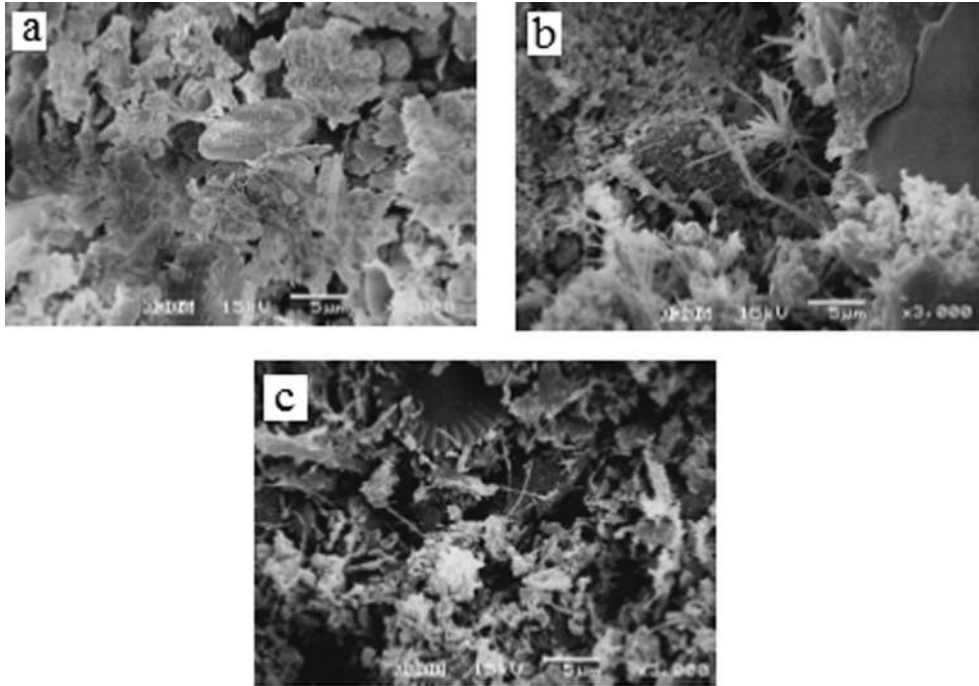
### 5.1 Stabilization application method

It can be classified as temporary improvement methods applied during the construction phase, permanent improvement methods applied without mixing any materials to the soil, and improvement methods applied by mixing various materials. Microstructural investigations are frequently used in geotechnical engineering, especially in the stabilization of clay soils [70,71]. As a result of reactions between clay soil and additives, changes in its microstructure are examined. Additives such as lime and cement are added. However, wastes such as fly ash,

metal slag, and marble waste, produced as industrial waste, are also used. The soils are mixed with one or more additives to improve engineering properties [69,74,75].

On stabilization, certain amount of lime is added to clay soils, and hence cation exchange and flocculation – agglomeration occurs in a short duration. Any cation can replace the ions. The fabric of clay soils was changed due to flocculation – agglomeration produces. The clay particles tend to clump together to form larger particles. Hence, expected to decrease the liquid limit, increasing the plastic limit, reducing the plasticity index, increasing the shrinkage limit, increasing the workability, and improving the strength and deformation properties of soil. The pozzolanic reaction may continue for a long time, so the pozzolanic reaction is expected to be more effective in the long term. As a result of the reaction between clay minerals and lime, calcium silicate gel is formed. The gel fills the pores and acts as a binder between the soil particles, thereby increasing the soil strength.

Stabilization with cement is similar to that with lime. The cement provides calcium-based minerals that increase the strength of soils, which in turn increases with curing time. Especially, clayey soil with low plasticity is most suitable for cement stabilization [25,64,65,76]. After the cement comes in contact with water, the hydration



**Figure 8:** (a) Clay 1, (b) clay 1 mixed with 20% cement at curing time 7 days, and (c) clay 1 mixed with 20% lime at curing time 28 days [89].

reaction starts quickly. The hydration of Portland cement is a complex pozzolanic reaction that creates various compounds and gels and causes degradation in the chemical structure of the soil. The hydration reaction consists of two stages, in the first stage, bonds are formed between the particles, and in the second stage, pozzolanic reactions occur, which cause the soil to harden. The strength of the soil and cement mixture continues to increase until the pozzolanic reaction ends [77]. As a result of improvement, the liquid limit of the soil decreases, the plastic limit and workability increase. Stabilization with cement causes increased strength. In general, soil improvement provides improved gradation, reduced plasticity and swelling potential, increased strength and workability, and improved stability of clay.

## 6 Studies about SEM images of improved clay

Physico-chemical analyses, in addition to physical and mechanical experiments, are important to observe the reactions occurring between soil and additive as a result of soil improvement. According to the physical tests we saw that for example, a decrease in plasticity in improved clay soil by additive material and increase particle size.

The interpretation of the SEM analysis is based on the grain size before and after the improvement. According to a mechanical experiment, the increase in the free pressure test of a clay soil after improvement means that the clay and additive react and the pores between the particles decrease. Results obtained from mineralogical, microstructural, and porosimetric tests allow a better understanding of soil behavior, which traditional mechanical tests do not explain [78]. For this reason, there are many studies on both clay microstructure [40,79] and improved clay microstructure in the literature [80–88].

Onitsuka et al. [89] investigated on microstructure and strength of lime and cement stabilized by two different Ariake clays. They performed Atterberg limit, SEM, permeability, unconfined compression, and oedometer tests on clays mixed with lime and cement. Figure 8 shows the SEM images of cement stabilized by two different clays at a curing time of 7 days. The microstructure of stabilized clays and the remolded clays are different. The formation of cementing products induced a decrease in pore spaces. According to the figure, the microstructure of lime-stabilized clays at the curing time of 7 and 28 days is different from the remolded clays. Researchers said that strength development relates to the microstructure due to fabric and cementation. The strength of both the stabilized clays was increased with the increased curing time and admixture content.

Nontananandh et al. [90] investigated SEM on stabilized soil with cement. The soil was mixed with Ordinary Portland Cement type I and so tested to obtain strengths, coefficients of permeability, and analyzed SEM images at curing times of 7, 28, and 90 days. According to the mechanical test, the strengths of cement-stabilized soil increased and hydraulic conductivity coefficient decreased in a short term. Calcium silicate hydrate (C-S-H) and ettringite formed on clay fabrics are determined using SEM (Figure 9). As a result, reduced pore spaces occurred hardened soil structures, increased strength and decreased hydraulic conductivity coefficient.

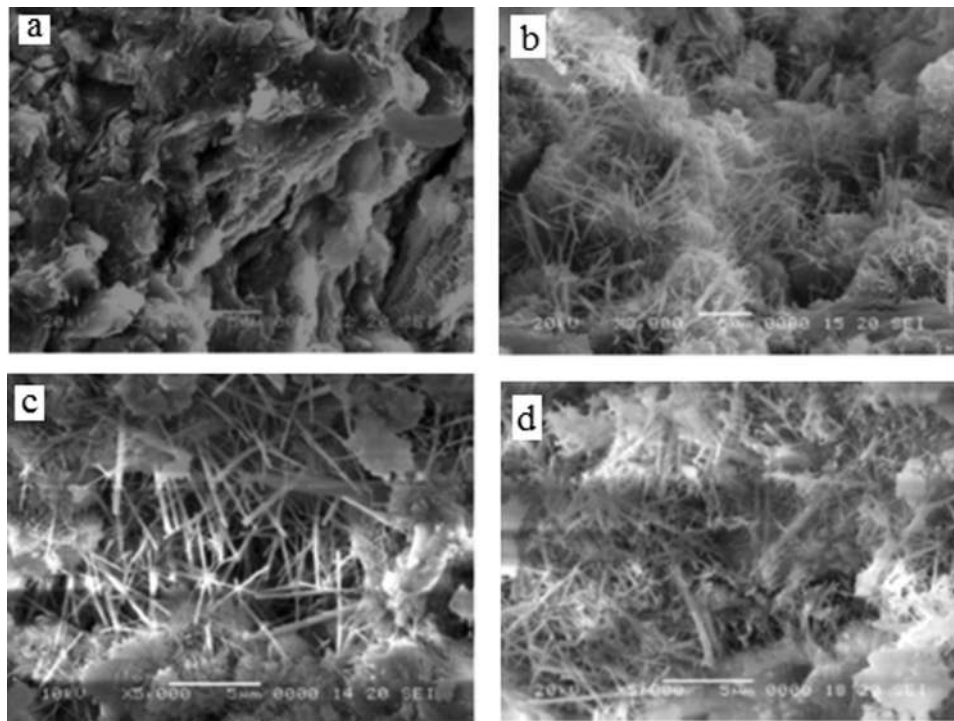
Liu et al. [91] investigated the effects of magnification on expansive soils on SEM images. Researchers conducted magnification and enlargement of frame sizes to define size proportion values. Within the matrix, values were combined with fractal analysis to relate magnification/frame size to planar porosity and fractal dimensions of soils. As a result, they observed excessive scatter in estimated porosities about an expansive soil.

Horpibulsuk et al. [92] analyzed the strength development in cement-stabilized silty clay based on microstructural. Researchers performed test water content, curing time, and cement content on the silty clay. Figure 10 shows SEM images. According to SEM images, after 4 h

of curing, the soil clusters and the pores are covered and filled by the cement gel. As time progresses, the hydration products in the pores are observed, and the soil-cement clusters tend to be larger. This state is seen as the reason for increased strength as time progresses.

Behera and Mishra [87] investigated the effect of lime on CBR behavior of fly ash-mine overburden mixes by the laboratory California bearing ratio (CBR) tests. They carried out experiments with different lime percentages, and lime treatment results showed increase in bearing ratio. Researchers also performed SEM analyses on cured specimens. The SEM images at 28 days are shown in Figure 11. Images show the coating of fly ash and overburden particles with lime addition. Moreover, images showed that calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H) were formed around fly ash and overburden particles due to the pozzolanic reaction. Thus, the SEM images show the relationship between bearing ratio development and microstructural development.

Millogo et al. [93] investigated microstructure, geotechnical, and mechanical characteristics of quicklime-lateritic gravels mixtures. The microstructure properties of quicklime-lateritic gravels mixture were determined using X-ray diffraction (XRD), infrared spectrometry, differential thermal analysis, SEM, and energy dispersive spectrometry. Also,



**Figure 9:** SEM images cement stabilized soils at various curing times (a) untreated soil, (b) soil mixed with cement 7 days of curing, (c) soil mixed with cement 14 days of curing, (d) soil mixed with cement 28 days of curing [90].



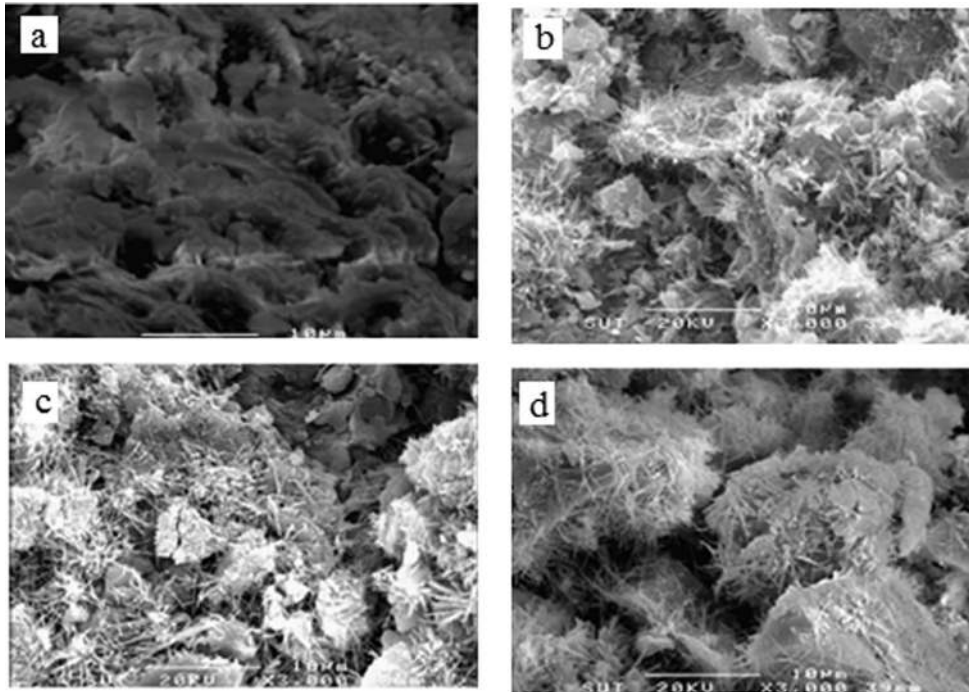


Figure 10: SEM images of the compacted soil at (a) 4 hours of curing, (b) 7 days of curing, (c) 28 days of curing, (d) 60 days of curing [92].

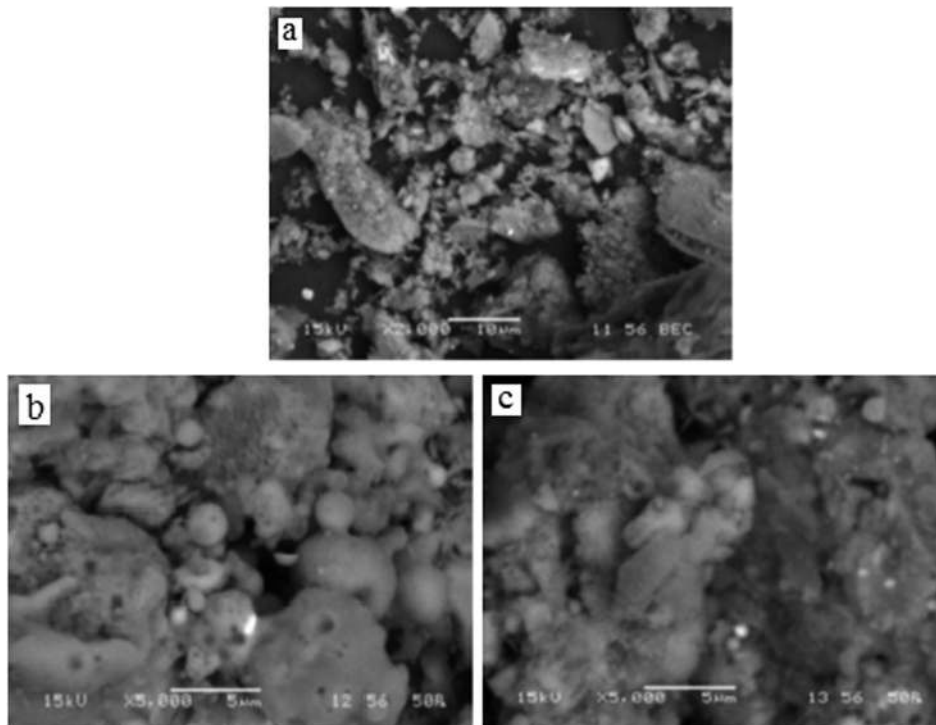


Figure 11: SEM images of (a) mine overburden ( $\times 2,000$  magnification), (b) mine overburden mixed with 3% lime ( $\times 5,000$  magnification), (c) mine overburden mixed with 9% lime ( $\times 5,000$  magnification) [87].

the geotechnical and mechanical properties of the mixtures were determined using these tests. On adding quicklime,

clayey fraction decreased, plasticity index, methylene blue value, and maximum dry density and optimum moisture

content increased. According to SEM images, formation of calcium silicate hydrate type I (C-S-H(i)), portlandite and calcite with quicklime admixtures occurred due to the pozzolanic reaction, which contributed to the improvement of mechanical properties (Figure 12).

Saeed et al. [94] investigated the effect of different curing times and cement contents on strength and compressibility of cement-treated kaolin clay samples. They conducted compaction, unconfined compressive strength, one-dimensional consolidation oedometer tests, XRD analysis, SEM analysis, and pH test on clay samples mixed with 5 and 10% cement. According to SEM, the compressibility test, and the unconfined compressive strength test, strength increased and decreased in the compression index in the long term (Figure 13).

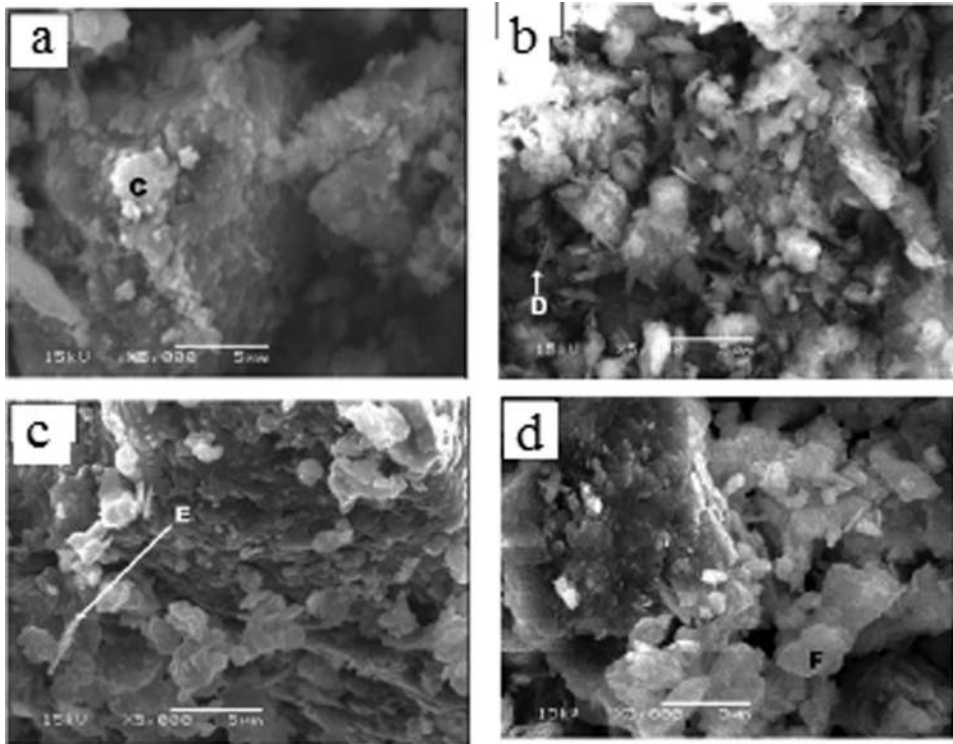
Du et al. [95] investigated the engineering properties and microstructural characteristics of cement-stabilized zinc-contaminated kaolin. Tests determined Atterberg limits, water content, pH, stress-strain properties, unconfined compressive strength, and secant modulus. Also, XRD, SEM, and mercury intrusion porosimetry studies were carried out on stabilized kaolin clay. Figure 14 shows the microstructures of the kaolin stabilized 28 days cured with cement 12%. First, from SEM images, it covers the surface of the soil aggregates together with a tiny amount of C-S-H gels. In 0.02% Zn concentration, a large quantity of cubic crystallized hydration product (C3AH6) was observed. In 0.2% Zn concentration, the fine network of reticulation is almost invisible. Small particles covered the surface of kaolin clay clusters ( $\text{CaZn}_2(\text{OH})_6 \cdot 2\text{H}_2\text{O}$ ). In 2% Zn concentration, surfaces of the clay clusters were almost wholly covered by  $[\text{CaZn}_2(\text{OH})_6 \cdot 2\text{H}_2\text{O}]$  crystals, and C-S-H was not identified. Consequently, the retardant effect of Zn on the hydration and pozzolanic reactions has changed phases of hydration products and cementation structure – bonding of the soils. Researchers said that Zn concentration had affected the engineering properties, phases of hydration products formed, and the microstructural characteristics of the stabilized clay.

Ural [96] investigated the relationship between geotechnical index properties and the pore-size distribution of compacted clayey silts. The mixtures were obtained by replacing two different clays (three different clay percentages) with silt. Atterberg's limit tests, standard compaction tests, mercury intrusion porosimetry, XRD, SEM analysis (Figure 15), and specific surface analyses were carried out on mixtures. From SEM images, a dispersed structure for compacted silt samples was observed. With the addition of clay, soil structure has increased flocculation.

Fauzi et al. [97] investigated waste plastic high-density polyethylene (HDPE) engineering properties and

waste crushed glass as additives on subgrade improvement. Soil engineering properties, standard compaction, 4 days soaked California Bearing Ratio (CBR), and Triaxial test were carried out on some clayey soil samples. The optimum design of mixed soil samples was conducted for the energy dispersive X-ray characterization by Integrated Electron Microscope and Energy-Dispersive X-ray Spectroscopy (SEM-EDS) (Figure 16). Figure shows that the samples were contented of C, O, Al, Si, K, Fe, Ti, and O. The most prevalent compound present in this soil mixture was  $\text{SiO}_2$  (54–60%). Researchers said that the bonding of silica and alumina had given significant distribution effects among the elements. The values of samples  $\rho_{\text{dmax}}$  stabilized soil with HDPE were showed different trends when HDPE content increased. The  $\rho_{\text{dmax}}$  of the stabilized soil was decreased when the glass content was increased. The values of  $\omega_{\text{opt}}$  stabilized samples were decreased when waste HDPE and glass content were increased. Consequently, the increase of  $\rho_{\text{dmax}}$  in stabilized samples increased the mean compaction properties.

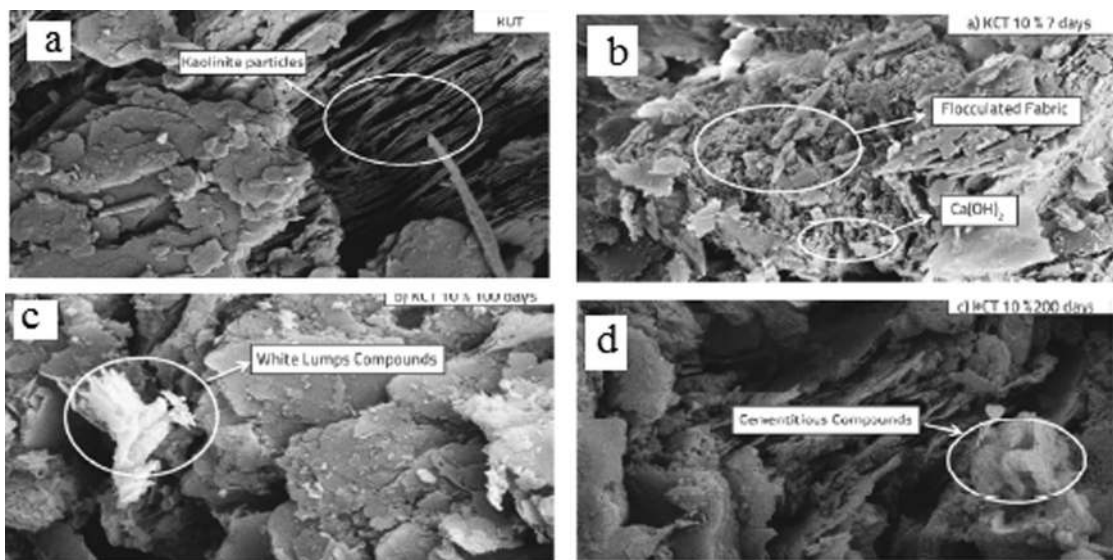
Ural [98] investigated the effects of additives on the microstructure of the clay uncured and cured lime/cement-stabilized clay samples. For this purpose, mercury intrusion porosimetry, XRD, SEM analyses, and specific surface area analysis were carried out on compacted soil samples for 0, 14, and 28 days curing. Thereby, the microstructures of the soils were assessed for short- and long-term reactions. From the SEM images of lime mixtures obtained with different percentages, the microstructures of the clay sample contain fewer flaky layers with an increase in the lime percentage due to flocculation. From the SEM images of cement mixtures obtained with different percentages, the microstructures of the clay sample contain a reduction in flaky layers because the percentage of cement is sufficiently high to be active. When an increase in the percentage of lime and curing time yielded a more flocculated structure, an increase in cement and curing time yielded a more aggregated structure. With increased curing time, the increase in the percentage of lime produced a more pronounced flocculation structure. Similarly, the increase in cement percentage caused the pores between the pellets to decrease due to flocculation. Also, white lumps were observed after 14 and 28 days of curing. Similarly white lumps were observed by Kassim [99] and Muhmed and Wanatowski [88]. The researcher said that greater additions of lime and cement increased the pozzolanic reactions. This finding is also consistent with those of Al-Mukhtar et al. [100]. The study showed that the addition of lime and cement was effective in the treatment of compaction properties.



**Figure 12:** SEM images of (a) raw material, (b) raw material mixed 2% lime, (c) raw material mixed 3% lime, (d) raw material mixed 8% lime (C: isolated and irregular particle of kaolinite, D: joined the isolated particles, E: the isolated particles were linked with the cementitious compounds (C–S–H), F: bright areas from portlandite and calcite that were extensively formed) [93].

Wang et al. [101] investigated the aggregate size effect on changes in mineralogical 26 compositions and microstructure of lime-treated compacted soils. For this purpose, three soil powders with different maximum aggregate sizes were prepared before treatment with 2%

lime. Later, XRD, environmental SEM coupled with chemical analysis using energy dispersive X-ray spectrometry (EDX) and mercury intrusion porosimetry (MIP) were conducted to analyze untreated and treated samples at various curing times. Crystallized C–S–H was identified in the



**Figure 13:** FESEM images at different curing times (magnification of  $\times 10,000$ ). (a) Untreated clay, (b) clay mixed 10% cement, 7 days of curing, (c) clay mixed 10% cement, 100 days of curing, (d) clay mixed 10% cement, 200 days of curing [94].



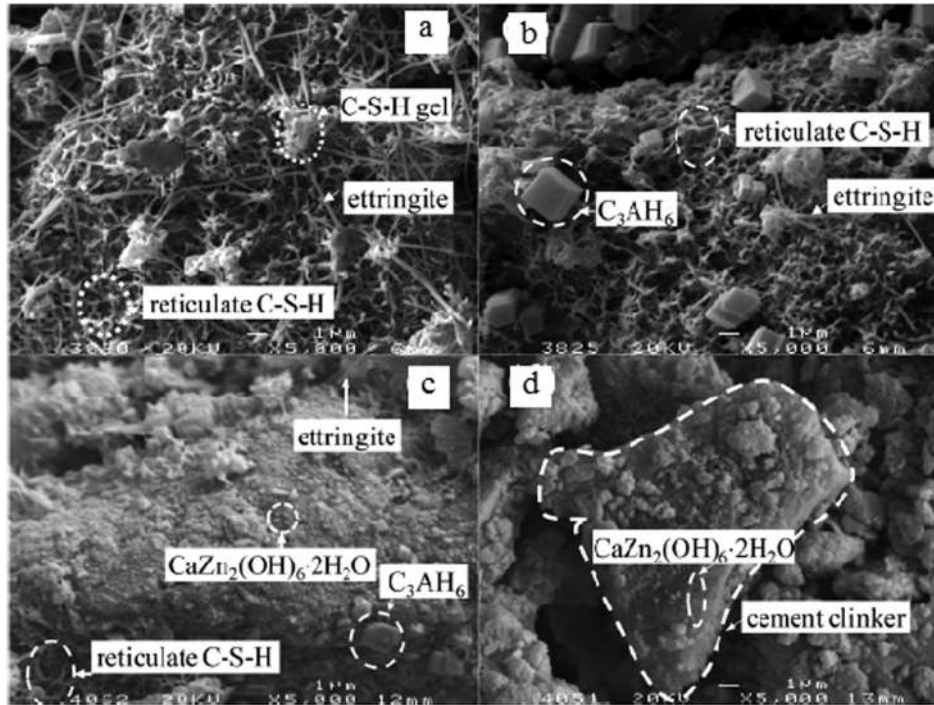


Figure 14: SEM images of the kaolin clays stabilized with 12% cement and 28 days of curing (a) 0% Zn; (b) 0.02% Zn; (c) 0.2% Zn; (d) 2% Zn [95].

lime-treated soil prepared with large aggregates after 1 year of curing (Figure 17). An evident increase in

nanopores less than  $0.1\mu\text{m}$  C-S-H was also observed due to C-S-H 34 creation. Researchers said that C-S-H

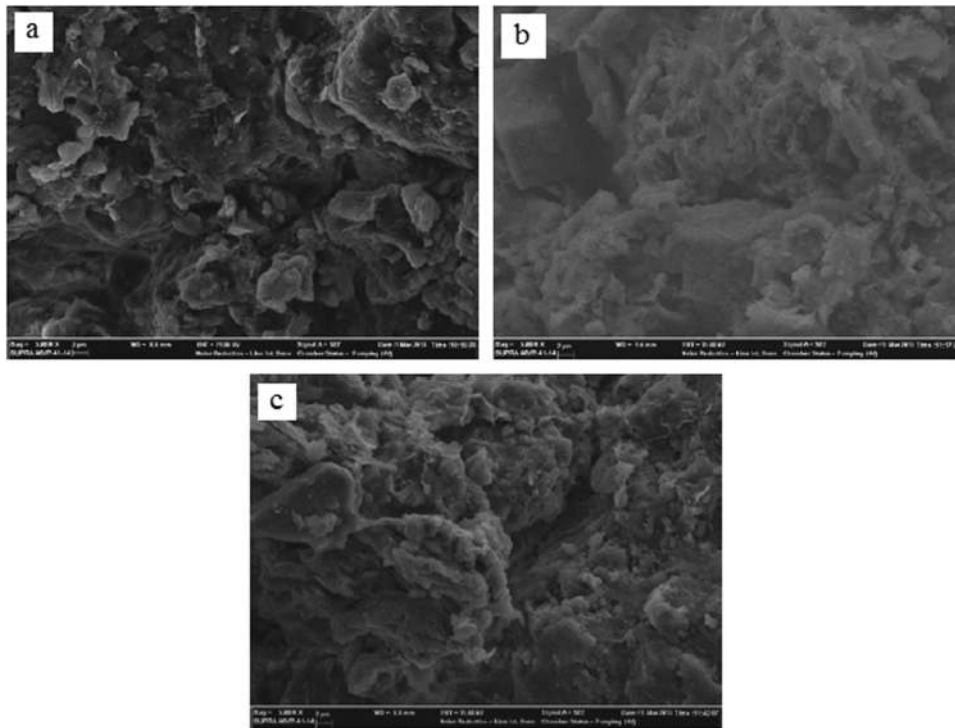
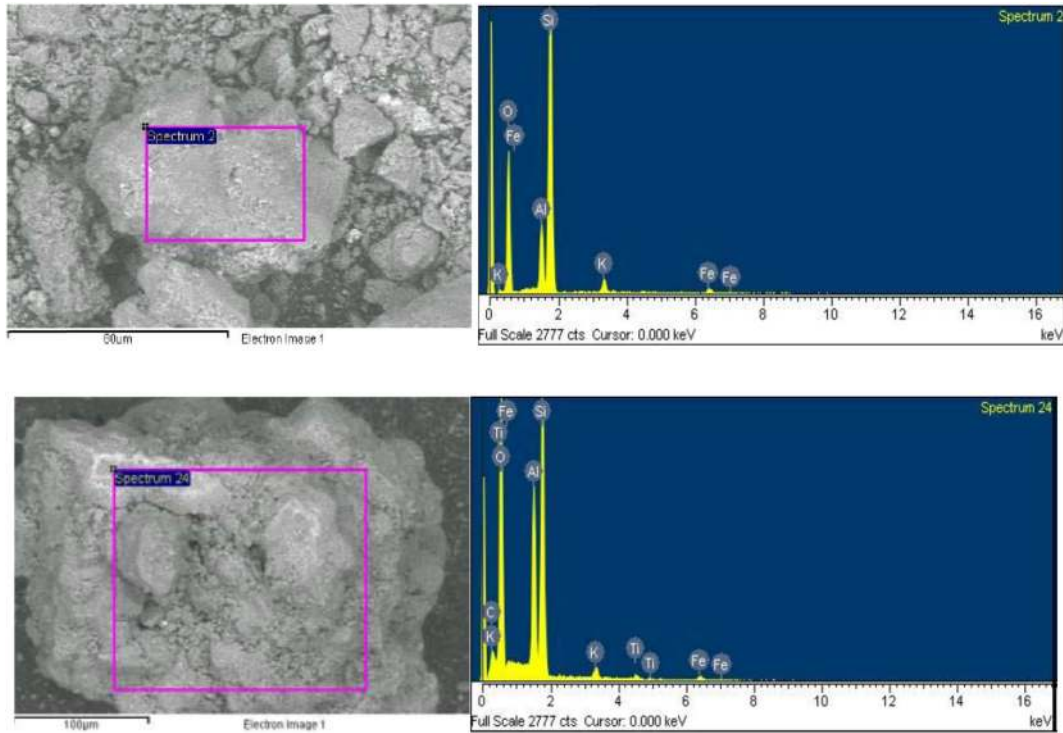


Figure 15: SEM images of mixtures (magnification  $\times 5,000$ ) (a) 10% clay II, (b) 20% clay II, and (c) 40% clay II [96].



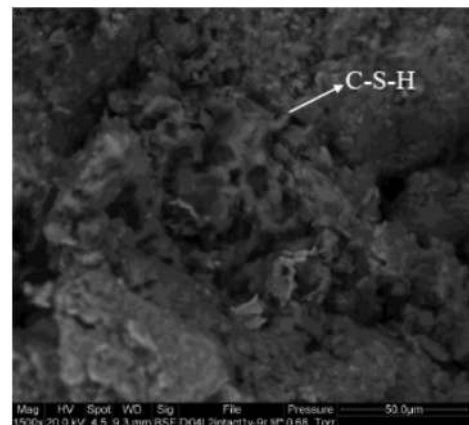


**Figure 16:** The SEM of two different soil samples were tested by SEM-EDS [97].

phases occurred when lime was coated with a thin layer on the large surface associated with treated soil prepared with small aggregates.

Nowadays, the widespread use of recycled material is shown for improving problematic soil [71,102–104]. Al-Bared et al. [102] investigated the sustainable improvement of marine clay using recycled blended tiles. Researchers used waste from ceramic tiles containing high sodium and magnesium for marine clay improvement in this study. In this work scope, tests were carried out to determine their size distribution, Atterberg limits, compaction, and unconfined compressive strength (UCS) of marine clay. Besides, the SEM, energy dispersive X-ray spectroscopy, and XRD were conducted. Thus, the microstructure was examined to understand the mechanism of strength development to understand the resistance. The UCS of marine clay increased with waste ceramic tiles. Microstructural analysis for untreated and treated marine clay samples was performed using SEM and EDS analyses, respectively. The SEM images of unimproved and improved clay at optimum different RBT contents at 14 and 28 days of curing are shown in Figure 18. The untreated marine clay sample entails a discontinuous and porous surface structure marked by the absence of hydration compounds. After improving clay, crystalline white lumps were observed on the surface of samples. These lumps were

responsible for the denser and less-porous surface structure and led to a heightened interlocking within the soil due to the coated marine clay particles. Finally, this state caused improvement in particles and their strength. With curing, improved samples performed with better strength due to the higher amount of cementitious compounds formed as reported in other studies [105,106]. In another study, the bearing capacity of the high-plasticity clay soil with bacteria stabilization (*Bacillus subtilis*) was

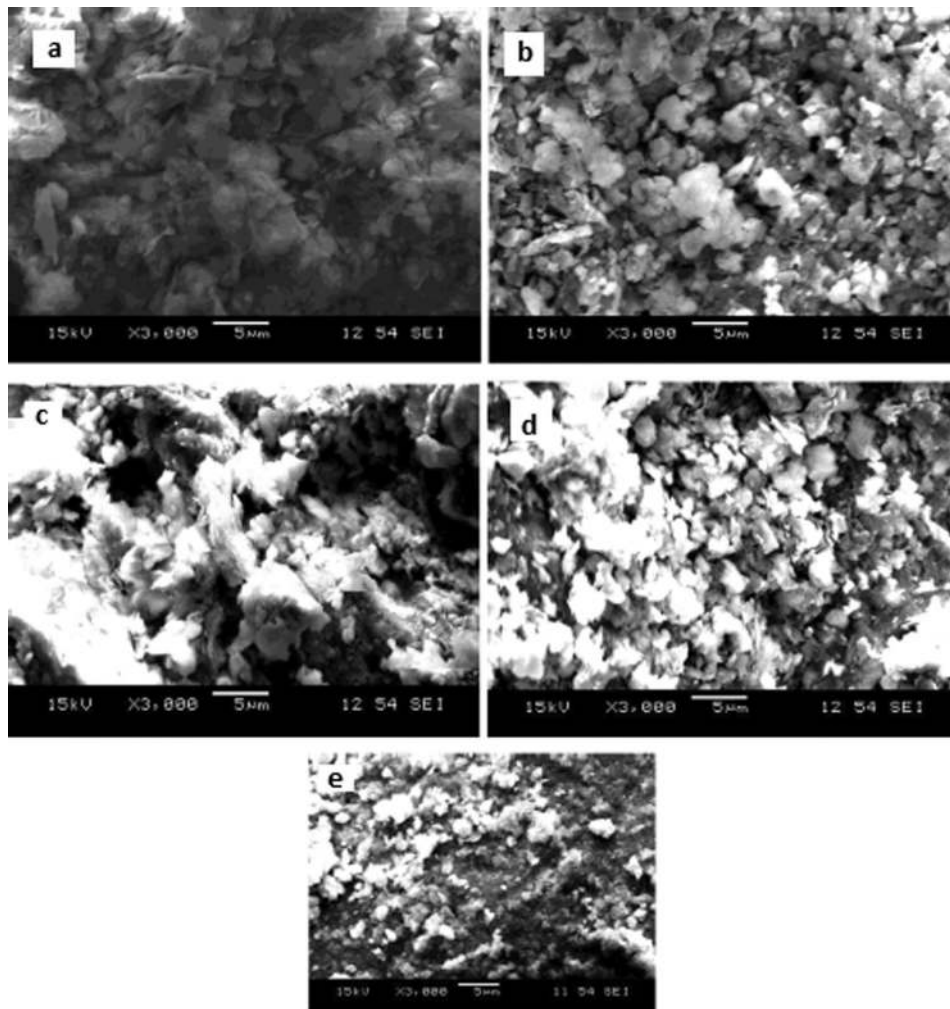


**Figure 17:** SEM picture of the lime-treated soil, at a curing time of 1 year [101].

investigated. In this study, CaO increased with the use of *Bacillus subtilis* bacteria in SEM-EDS analysis. Researchers indicated that bearing capacity assessment was based on unconfined compressive strength value, CBR, and soil reaction modulus as a subbase layer. This state corresponds to the increase in CaO value due to Microbial-Induced Calcite Precipitation (MICP) microstructurally. Consequently, researchers said that the significance of technical implications of *Bacillus subtilis* bacteria as a biotechnology stabilization material is to increase the bearing capacity and modulus of soil reaction with high plasticity clay [107].

Keramatikerman et al. [108] investigated the effect of fly ash on the liquefaction behavior of sand–bentonite mixture by conducting a series of undrained monotonic triaxial compression tests. Samples were prepared to add

5% bentonite and three fly ash contents at three relative densities on clean sandy soil liquefaction behavior. Also, XRD and SEM (Figure 19) analyses were conducted on mixtures. The main constituents of the used fly ash were found as quartz (Q), Mullite (Mu), and hematite (H) from the XRD analysis. The main constituents of the bentonite were found as quartz (Q), montmorillonite (M), and potassium (K). SEM images of soil treated with fly ash samples showed the generation of calcium silicate hydrate (C–S–H) and calcium aluminate hydrate (C–A–H). This produced strong bonds among clay particles. Increasing the fly ash contents intensified the generation of these hydration bonds. Researchers said that this situation caused a higher value of deviatoric stress to be recorded at triaxial testing. Consequently, it was indicated that the untreated soil and soil samples have a



**Figure 18:** SEM images (a) marine clay, (b) improved marine clay with 20% 0.063 mm RBT (14 days of curing), (c) improved marine clay with 20% 0.063 mm RBT (28 days of curing), (d) improved marine clay with 30% 0.15 mm RBT (14 days of curing), (e) treated marine clay with 30% 0.15 mm RBT (28 days of curing) [102].

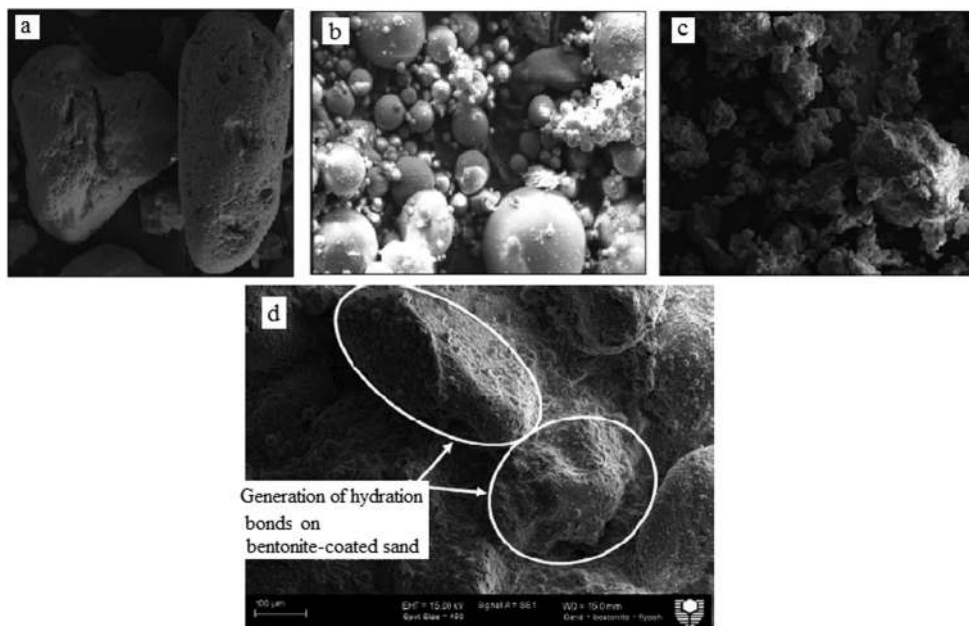
lower liquefaction strength than the fly ash-treated specimens.

Liu et al. [109] studied the geotechnical properties and microstructure of lime-stabilized saline soils under freeze-thaw cycling. Researchers conducted the proctor compaction test, particle size composition test, boundary moisture content test, cation exchange capacity test (CEC), mercury intrusion porosimetry (MIP), SEM, and unconfined compressive strength (UCS). They said that the results reveal that adding lime to saline soil transforms clay particles into sand and silt particles, and hence causes the pore size distribution (PSD) to become bimodal, with small pore and macropore. With an increased lime content, an increase in the unconfined compressive strength (UCS) and a gradual decrease in the cation exchange capacity (CEC) and the plasticity index were observed. They observed the geotechnical properties of the soil change with the addition of lime, and this state indicates that the cation exchange reaction takes place very quickly. Figure 20 shows SEM micrographs of saline soil treated with 15% lime after freeze-thaw cycles after 28 days of curing. The figure shows SEM images of saline soil treated with 15% lime after freeze-thaw cycles after 28 days of curing. Figure 20a and b shows observed aggregated soil particles of varying shape and size with the addition of lime. Besides, Figure 20c shows needle-like cementing materials. In this case, a fine network occurred in large pores and

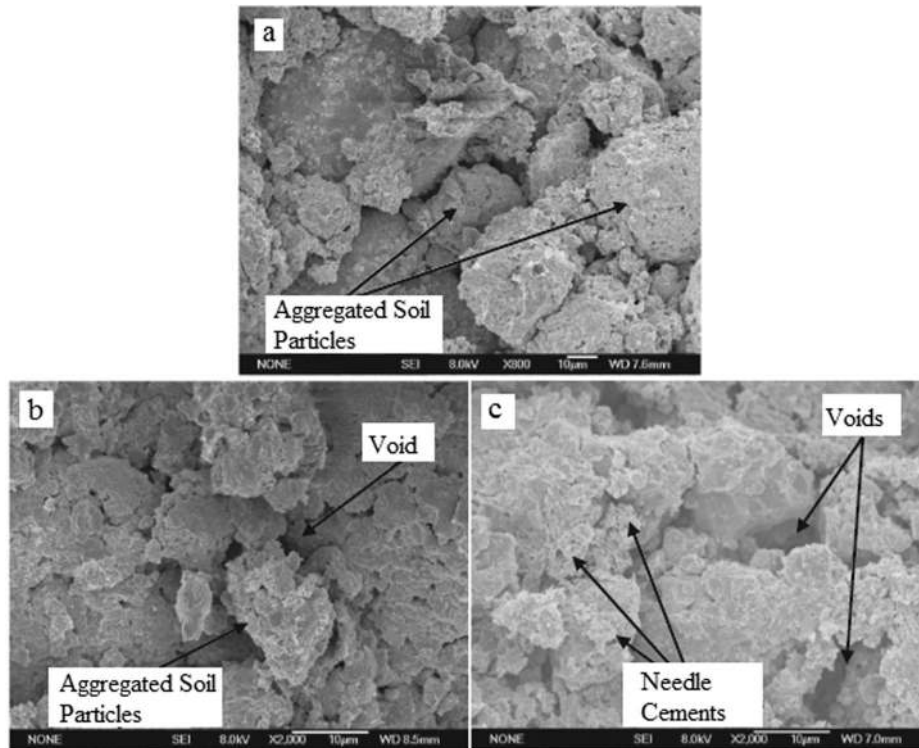
seem bonded in the soil aggregates. It is explained by an increase in sand-sized particles and a decrease in porosity with these changes in SEM photos.

Zhu et al. [110] investigated the microstructure of lime-stabilized silt clay. They evaluated the effects of lime content and curing time on the overall soil properties, including compaction characteristics, Atterberg limits, particle size distribution, pH, stress-strain behavior, peak strength, shear strength parameters, and CBR. SEM images showed that a white cementitious gel was formed after the addition of lime and that peaks related to smectite, illite, kaolinite, and quartz appeared sharper after stabilization with lime and 90 days of curing (Figure 21). In the case of kaolinite, illite, or montmorillonite, differences in the microstructure of the clay are observed according to the clay mineralogy [111].

On SEM and XRD investigations, Sekhar and Nayak [112] reported that lithomargic clay is stabilized using granulated blast furnace slag (GBFS) and cement. Physical test, strength test, SEM-EDS, and XRD analyses were conducted on mixtures. The SEM images of natural soil were observed with a smooth texture, wavy with visibly larger void spaces. The SEM images of replacement soil with GBFS were shown to produce agglomerations, and thus the particles were found to be flocculated and the pore or air spaces have reduced. This situation gained strength to mixtures. High strength and stiffness were achieved by eliminating large pores by bonding particles



**Figure 19:** SEM images of (a) untreated soil, (b) fly ash, (c) bentonite, (d) generation of hydration bonds in mixture with 5% bentonite and 7% fly ash [108].



**Figure 20:** SEM micrographs of saline soil treated with 15% of lime after freeze-thaw cycles after 28 days of curing (a) 60th freeze-thaw cycles; (b) 3rd freeze-thaw cycles; (c) no freeze-thaw cycles [109].

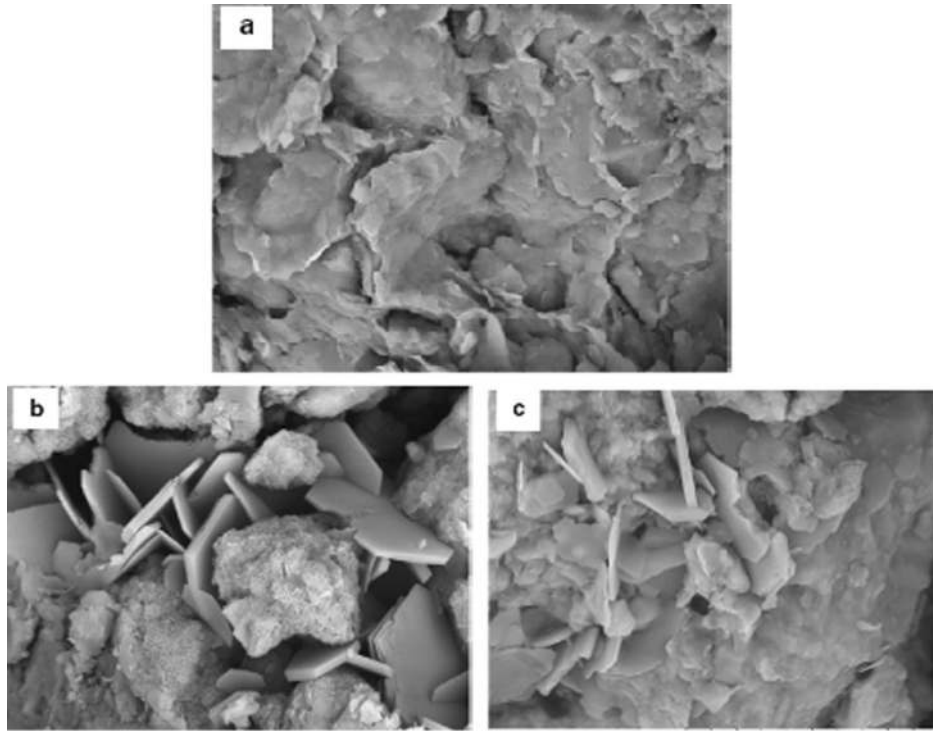
and flocculent particles arrangements. Researchers said that cement generates hydration products at higher curing periods, which helps those cementitious products fill the voids and pores. As a result, the strength of the soil improved.

Farhadi-Machekposhti et al. [113] investigated the potential improvement of sandy soil hydraulic properties with the addition of marble powder. The saturated hydraulic conductivity and soil water retention curves were obtained for six marble powder and soil levels. Thus, pore space properties were investigated using soil water retention curves by mercury intrusion porosimetry and SEM (Figure 22). The figure shows that as the percentages of marble powder in the mixture increase, there is a gradual process of filling the small pores. Researchers said that the original soil is coated by other much smaller particles increasing the specific surface. As a result, it was noted that marble powder appears to be located in the original soil's intergrain pores, thus reducing the effective space for water flow, which explains the reduction in saturated hydraulic conductivity. They indicated that marble powder connects the original soil particles, fills larger pores, and creates a finer structure for pores. In conclusion, the addition of marble powder can improve the ability of soil to store water, providing an

advantage for irrigation water management in water-scarce environments.

Islam et al. [114] investigated Microbial-Induced Calcite Precipitation (MICP) applicability to improve clayey soils with low to high plasticity. For this purpose, soils with low to high plasticity and three different artificial mixes with varying contents of clay were studied. Tests were conducted for strength gain, swelling reduction, and calcium carbonate precipitation on untreated and biostimulated soils. The SEM and EDX analyses were carried out on soils (Figure 23). From SEM-EDX analysis of untreated soils, considerable amounts of oxygen, carbon, silicon, and aluminum were observed, but no significant calcium peak was seen. From SEM-EDX analysis of treated soils, considerable calcite precipitated after the treatment, and bridged the soil particles. Researchers were noted that MICP via biostimulation would be a promising method to treat problematic clayey soils. Another study investigated microbial-induced carbonate precipitation for strengthening soft clay. Soft clay samples were prepared using a solution containing *Sporosarcina pasteurii* bacteria, solutions with different nutrient salts, and soft clay. These samples were subjected to unconfined compressive strength tests after curing for 28 days. XRD and SEM-EDS analyses were also performed

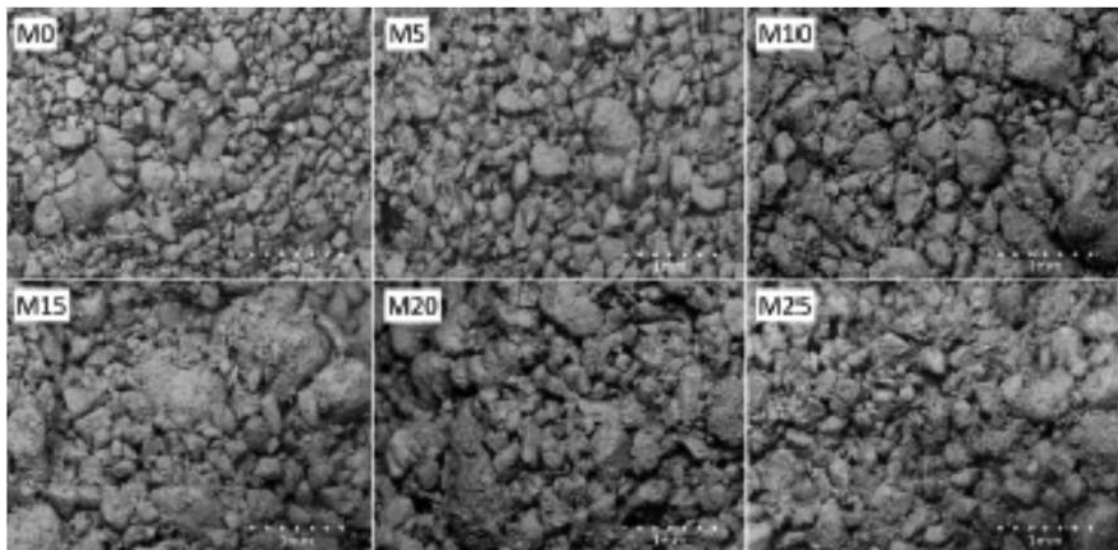




**Figure 21:** Scanning electron microscopy images (a) silt clay, (b) silt clay mixed 7% lime, 7 days of curing, (c) silt clay mixed 7% lime, 90 days of curing [110].

(Figure 24). The SEM images of soft clay shows pores and fissures. In SEM images of calcium carbonate precipitation in soft clay, the calcium carbonate crystals induced by microorganisms are distributed among the soft clay particles, with varying patterns at different positions.

Generally, these crystals are in the form of blocks or particles, and the particle size of the microbial-induced calcium carbonate is close to that of the soft clay particles. EDS analysis confirmed that the mineral observed is calcium carbonate. These laboratory tests were used to



**Figure 22:** SEM images of mixtures ( $\times 50$  magnification, the numbers indicate the percentage of marble powder added to the original sandy soil) [113].

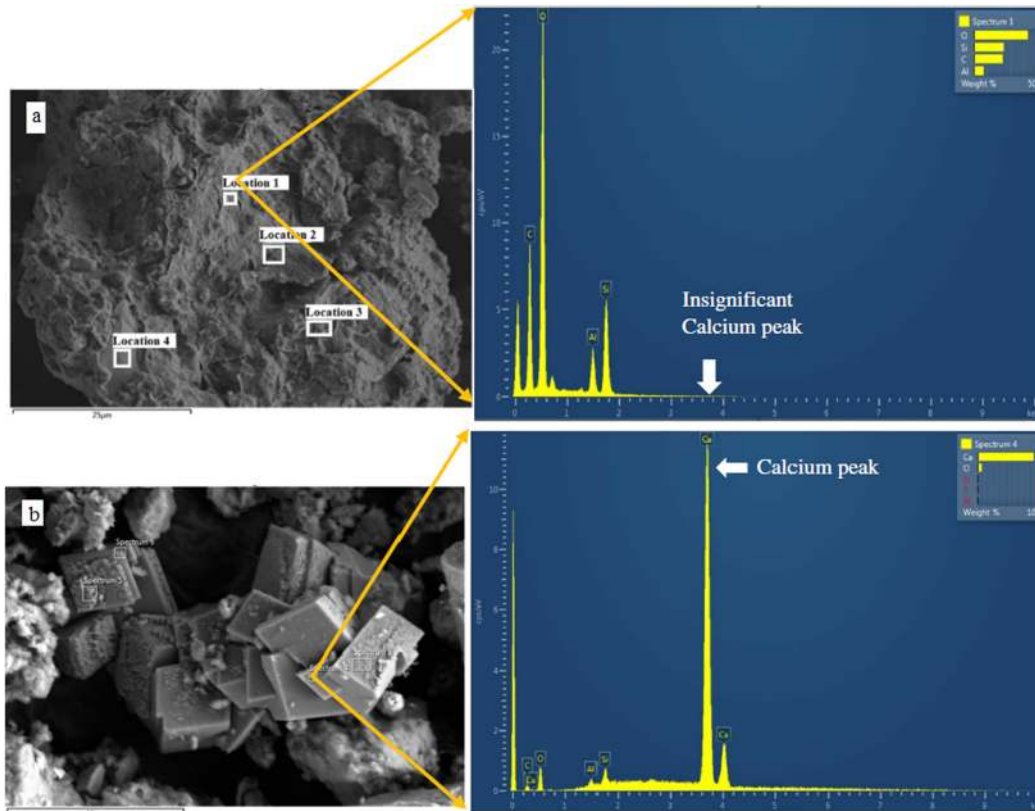


Figure 23: SEM and EDX analysis of (a) untreated and (b) treated clayey soils [114].

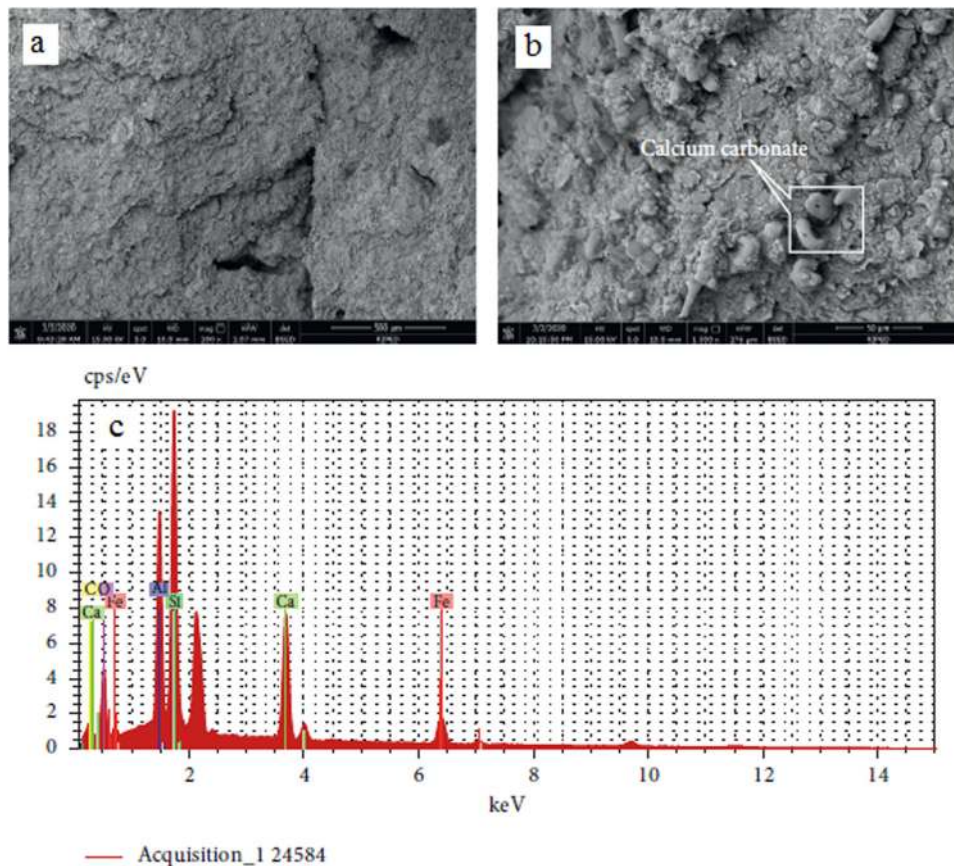


Figure 24: (a) SEM image of soft clay, (b) SEM image of calcium carbonate precipitation in soft clay, (c) The EDS test result [115].

study the chemical reactions, the strength of clay, and other influencing factors. Consequently, the formation of calcium carbonate in microbially solidified soft clay caused an increase in strength. According to this study, it is feasible to use MICP to increase the strength of soft clay [115].

## 7 Conclusion

Problems encountered in the construction of structures on problematic soils are seen in many regions of the world. The geotechnical engineer, especially with these soils, encounter the design and construction of land, railway embankments, airports, soil/rock-filled dams, and water channels. Additives such as lime, cement, fly ash, industrial waste, and tire are used to improve the soils. According to the literature, soil improvement works were performed on clayey soils due to its special structure. The clay soils reacted with additives to increase their strength and durability, and to decrease their compressibility and plasticity. Also, physico-chemical analyses are carried out to understand the reason for these changes in geotechnical properties.

SEM is used as a research tool to understand the root cause of the differing performance of various soil types under various conditions, a development tool in improving soils, and a diagnosis tool on different soil problems. Hence, the SEM has been instrumental in developing our understanding of the inside of soil bulk. In this sense, the electron microscopy offers vast opportunities in helping researchers and practicing engineers gain insights in understanding soils. The reason for the SEM's this usefulness in geotechnical engineering is the high resolution obtained when soil bulk objects are examined. In this paper, selected case studies have also been discussed. SEM tests are generally in material engineering laboratories in terms of geotechnical engineering. The disadvantage of an SEM is its cost as the SEM instruments are expensive. Despite this, the advantages of SEM are numerous applications, the detailed and topographical imaging. Besides, the SEM instrument is user friendly due to its advanced technology usage, it works fast, and allows the generation of data in digital form.

Since SEM tests were carried out generally in material engineering laboratories for all engineering research, in this paper, selected case studies have been discussed from the perspective of how materials science tests are translated into applications. Consequently, SEM images shows reaction products, variations on microstructures

due to these reactions, and micro-macro pores. Thus, in soil improvement works, additive, percentage of additive, curing time, freeze-thaw cycle, etc., effects can be observed in detail. Therefore, SEM images can be more understood and interpreted well using the strength, hydraulic conductivity coefficient, and swelling tests.

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