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Microstructure Behavior of Batu Pahat Soft Clay (BPSC) Stabilized with Sodium Silicate (TX-85) And Biomass Silica (SH-85)

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Abstract: The soft clay soil usually causes problems to civil engineers due to its characters which are low shear strength and high compressibility, and many are sensitive in that their strength is reduced by disturbances. Strengthening and improving the soft clay soil is crucial before any construction work is done by improving the soft ground improvement. This research uses non-traditional chemical stabilization method by combining a fixed percentage (3%) of Sodium Silicate (TX-85) to varying percentage (6%, 9%, and 12%) of Biomass Silica (SH-85) then the samples were cured for (7, 14 and 28 days). The microstructural and mineralogy changes of the sample was observed using X-Ray Diffraction (XRD), and Scanning Electrons Microscopic (SEM) analysis. The results of all tests conducted showed changes in the microstructure behavior of soft clay soil stabilized with Sodium Silicate (TX-85) and Biomass Silica (SH-85). Thus, the XRD results of the study indicated the presence of a new mineral composition but at a low intensity, as well as an increase in the amount of Quartz mineral due to the addition of stabilizer. This is because pozzolanic reactions occur between the calcium ions of the stabilizer and the silica and alumina of the clay minerals resulting in the formation of cementitious products such as calcium-silicate-hydrates (CSH), calcium-aluminate-hydrates (CAH), and calcium-aluminum-silicate-hydrates (C-A-S-H). Additionally, the void was filled with a stabilizing substance during SEM testing. Additionally, cementation product was detected via the SEM test as a result of the stabilizer given to treated samples. Additionally, the EDX test established that the appearance of the Calcium (Ca) element in the treated sample is a result of the stabilizer's chemical composition. As a result of this research, the relationship between the microstructure and mechanical properties of treated soil was established.

Keywords: Soft Clay Soil, Microstructure and Mineralogy, Stabilizer

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1. Introduction

Due to the rapid development in Batu Pahat areas, the sites which were formerly considered unsuitable had to be used for construction, which is depending on the increase of population tremendously and economy are boosting up in Batu Pahat areas. Strengthening and improving the soft clay soil is crucial before any construction work is done by improving the soft ground improvement [9,12]. Each performance of the building load carried must have a satisfactory soil bearing capacity and a satisfactory physical strength to ensure the soil's bearing capacity is just not impacted.

Stabilization is an excellent choice for the modification of soil properties and one of the treatments to improve strength of soil. Soil stabilization is divided into three categories which are physical, mechanical and chemical method. In this study, stabilization in chemical method involved a process of stabilizing agent which chemical was mixed with the soils to improve geotechnical properties of natural soils. The natural and stabilized soils will be conducted in accordance with BS 1377:1990 which included the classification and mechanical properties tests [7-11].

Numerous suppliers and manufacturers have developed a new agent for soil stabilization and strength enhancement which is sodium silicate (TX-85) and biomass silica (SH-85) as the stabilizing agent [13 and 14]. Sodium silicate (TX-85) exists in the form of a white powder or a colorless solution that is water soluble [13]. The function of these stabilizers is to improved construction material and enhancing many of the engineering properties of the soil. In addition, also can reduces welling and bind particles of soil at the same time fill the void between particles of soil. The literature demonstrates that these ionic stabilizers can improve the strength of soil through cationic exchange and flocculation. A laboratory examination revealed that the material is made up of sodium, aluminium, silicon, and iron, and has a pH of 12.54, making it very alkaline [1-4]. The effects of Sodium Silicate (TX-85) on the physic-chemical properties of organic soil have been the subject of several investigations and study. According to the findings of these studies, adding 3 mol/L of Sodium Silicate (TX-85) can increase the strength by 220 %. [1-4]. According to earlier research, the chemical composition of Biomass Silica (SH-85) is almost identical to that of cement and lime [14]. Calcium oxide (CaO) is the major chemical composition on these stabilizers. In the presence of water, the calcium ions released from these stabilizers reduce the thickness of double diffused layer through cation-exchange and flocculation-agglomeration reactions. This is primarily responsible for improvement in workability through reduction of adsorbed water and decrease in plasticity index. In long-term, pozzolanic reactions occur between the calcium ions of the stabilizer and the silica and alumina of the clay minerals resulting in the formation of cementitious products such as calcium-silicate-hydrates (CSH), calcium-aluminate-hydrates (CAH), and calcium-aluminum-silicate-hydrates (C-A-S-H). This stabilizer enhances the strength of the natural soil it has been mixed with as the curing period increases, while also creating compaction gel to fill the porous spaces within the soil. As a result of this scenario, the soil becomes denser [1, 2, 5-6]

The aim of this research is to investigate the microstructure behavior of Batu Pahat Soft Clay (BPSC) stabilized with Sodium Silicate (TX-85) and Biomass Silica (SH-85) [1-2]. This research also conducted to determine the microstructure and mineralogy of untreated Batu Pahat Soft Clay (BPSC) and treated Batu Pahat Soft Clay (BPSC) stabilize with Sodium Silicate (TX-85) and Biomass Silica (SH-85) with different proportion of stabilizer and different curing times.

2. Methodology

2.1 Material

The soil sample was taken at UTHM Parit Raja's Research Centre for Soft Soil (RECESS) in Batu Pahat, Johor. The 10-hectare location is around 20 kilometres from Batu Pahat and is situated on soft soil. The samples were prepared in order to determine the microstructure and mineralogical properties. The physical characteristics of soft clay soil as shown in Table 1.

Table 1: Physical Characteristics of Soft Clay Soil [9]

Physical Properties	Values
Liquid Limit, LL (%)	73
Plastic Limit, PL (%)	29.12
Plastic Index, PI (%)	43.88
Maximum Dry Density, MDD (kg/m^3)	1343
Optimum Moisture Content, OMC (%)	30

2.2 Preparation of Samples

Before laboratory tests were carried out, raw materials have to be prepared. The soil samples were in wet condition and dried it for a day by keep it under the sunlight. The soil samples were put in the oven for 24 hours at 105 ºC to remove entrapped moisture. The soil samples were then tested for classification test and microstructural test. The soft clay was follow the given mixture of sodium silicate (TX-85) and biomass silica (SH-85) which is (3%L+6%P), (3%L+9%P), and (3%L+12%P) for the microstructural and mineralogical test and had been cured within (7, 14 and 28 days) of curing times and the percentages of mixture is based on each samples in order to get different mineralogy of each sample. The percentages of the mixtures is one of the important factors that influencing the mineralogy, consistency, existing new minerals and elements as well as the strength.

The experiment material needs to be prepared prior to the experiment testing in the laboratory. For this research soft clay, TX-85 and SH-85 was used for constructed the samples. The classification test included natural moisture content. This step included the determination of the optimum moisture content (OMC) for natural soil. Besides that, the microstructure and mineralogy testing was conducted by using X-ray Diffraction (XRD), and also Scanning Electron Microscopic (SEM).

2.3 The Testing of Samples

X-Ray Diffraction (XRD) provides information on structures, powder, phases, preferred orientations of crystals and other structural parameters such as crystallinity, mean grain size and defects of crystals. Preparation of soil samples before carried out the testing is important factor to get accurate reading. The soil samples need to sieve and grind until become fine grained particles or into powder form with 63 µm particles size in order to obtain smooth surfaces, avoid spottiness and reduce preferred orientation. Powdered soil samples are used to identify the crystal like minerals composition in the origin soil and stabilize soil and to observe its mineral composition changes after the stabilization. Powdered samples are then placed in an elliptical-opening glass holder and distributed uniformly to achieve a smooth surface with the aid of a microscope slide. The glass holder was then mounted in the Bruker D8 advance diffractometer to perform angle scans (2θ) from 10° to 90° with a step size of 0.02° and a second dwelling period for each single step. Finally, the Bruker D8 advance diffractometer reported the output data of the mineral composition in the raw result. Raw data was interpret into disk to be analyze by EVA software that can shows the graph and peak of mineral composition for every samples

Scanning Electron Microscopic (SEM) is used for analysis image of microstructure and chemical composition of samples. This technique offers some details about the shape, size and location of soil particle aggregation and orientation. This approach has been used to observe the alteration of different soil minerals and to identify new cementation products that are not detected in the XRD process. Moreover, the elemental composition or chemical composition of this materials may be determined by means of an Energy Dispersive X-ray (EDX) attached to the SEM instrument. Samples preparation almost same as XRD testing. The particles size are not specified to be into powder form. Soil samples were put onto stub plate that already have double salotape on it to ensure the samples is firmly attached to the plate. It is forbidden to press the double salotape to the sample as it can affect the result. Then, air duster were used to blew away the retained particles and disperse sample homogenously. Before placed in the SEM machine, Platinum or gold is used as a conductor to coat the surface of samples under vacuum condition in the Sputter Coater device to enhance the conductivity of samples and prevent the samples from fly away due to vacuum. The magnification factor used to scanned the samples was 1000x until 10 000x. After that, the samples are mounted on a special holder and inserted through an exchange chamber into the high vacuum part of the microscope with anchoring on a movable stage. Next process is analysis the scanned image with provided software to get the particles structure, shape and void. After done with the process, the analysis was continued to Energy Dispersive X-ray (EDX) software to get the chemical element composition contains in each samples and as a proved for XRD result if there is new mineral appeared.

3. Results and Discussion

3.1 The Characteristics of Microstructure and Mineralogy

This chapter discusses the microstructure behaviour and data analysis from laboratory tests for untreated and treated soft clay soils. The study was carried out to investigate the microstructure and mineralogy of untreated soft clay soils, as well as the changes in microstructural behaviour of treated soft clay soils, using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray (EDX), as well as pH testing to determine whether each sample was alkaline or acidic.

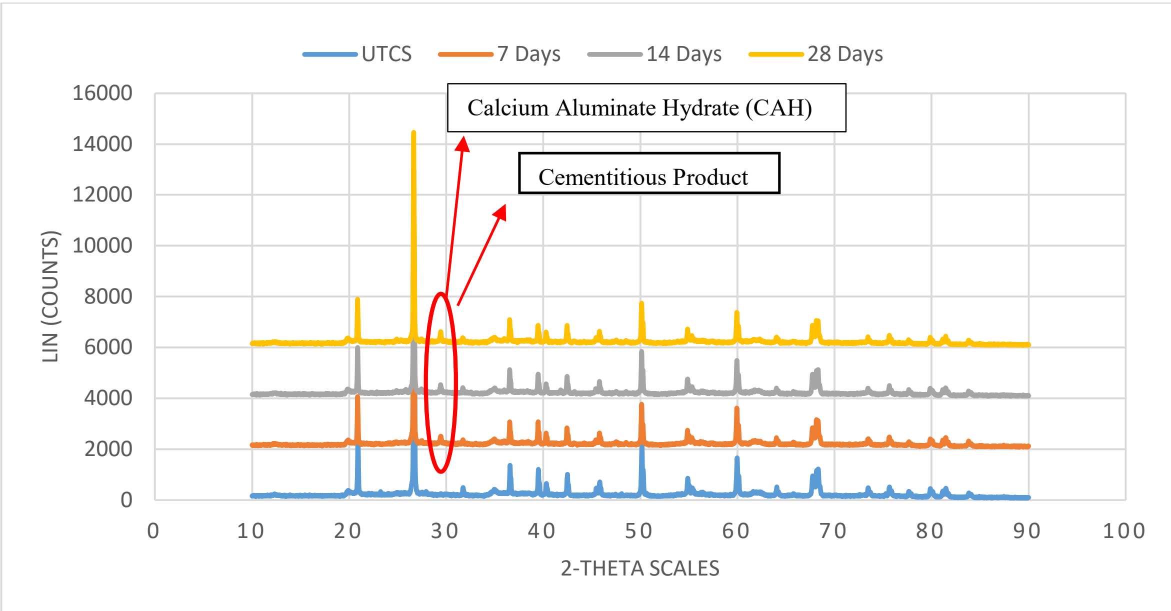

Figure 1: The XRD Results for (3%L+9%P) Stabilizer Proportion

Figure 1 indicate the results from the XRD testing for soft clay soil treated stabilized with Sodium Silicate (TX-85) and Biomass Silica (SH-85) with the proportion of stabilizer (3%L+9%P) based on different curing times which (7, 14 and 28 days). Based on the Figure above the stabilizer proportion (3%L+9%P) is the optimum proportion compared to (3%L+6%P) and (3%L+12%P). The major mineral observed in untreated clay soil were Quartz (SiO_2), Halite (NaCl) and Nontronite ($Na_{0.3}Fe^{3+}_2$ Si_3 $AlO_{10}(OH)_2$ $4(H_2O)$). There is no pozzolanic reaction and no cementation product happen for untreated clay soil. Also, mineral composition observed in (3%L+6%P), (3%L+9%P) and (3%L+12%P) of stabilizer were Quartz (SiO_2), Halite (NaCl), Nontronite ($Na_{0.3}Fe^{3+}{}_2$ Si_3 $AlO_{10}(OH)_2$ $4(H_2O)$), Montmorillonite (Ca, $Na)_{0.3}Al_2Si_3AlO_{10}(OH)_2$ $4(H_2O)$) and Calcium Magnesium Aluminum Silicate (Ca-Mg-Al-Si-O) with different intensity compare to untreated clay soil. New peak has appeared as a cemented product due to pozzolanic reaction with Sodium Silicate (TX-85) and Biomass Silica (SH-85). This presence mineral is called Calcium Aluminate Hydrate (CAH) that occurs between alumina alter with calcium hydroxide ($Ca(OH)_2$). The reaction happened from the cementitious process with water to produce Calcium Aluminate Hydrate (CAH), which developed bonding strength in treated soils.

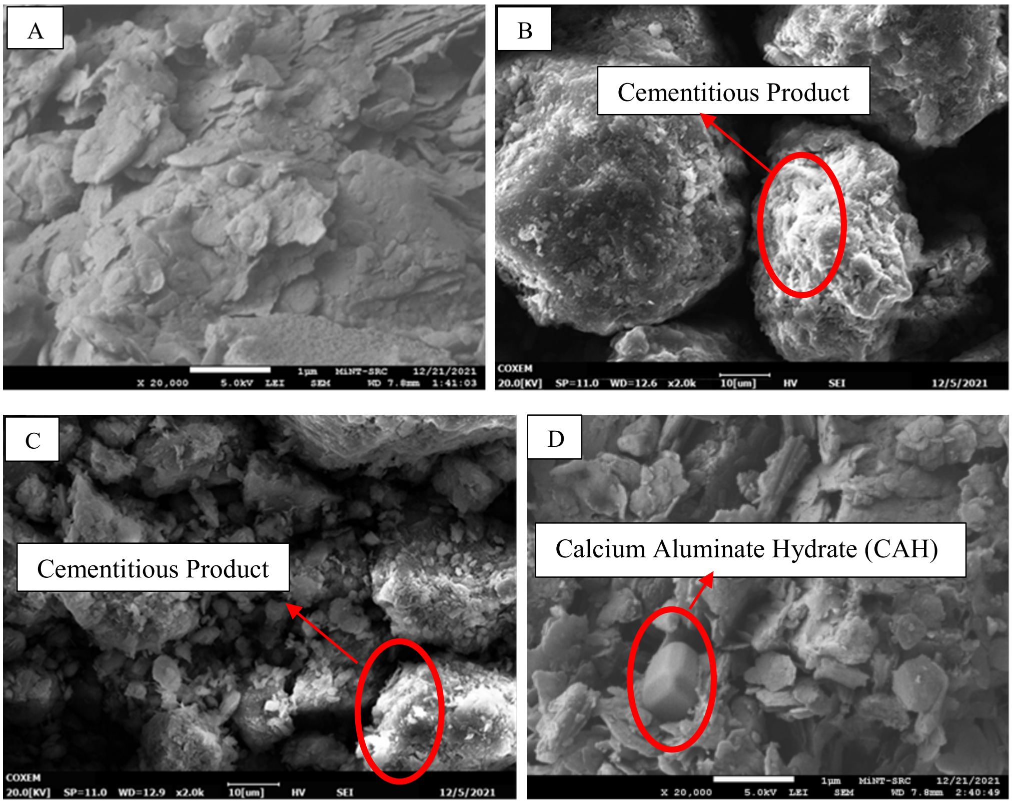

Figure 2: The SEM Image for Untreated Sample (A) and (3%L+9%P) at different curing times, 7 days (B), 14 days (C) and 28 days (D)

The SEM image (A) demonstrates that untreated BPSC at 1µm contains platy and feathery particles with a uniform texture organised in a scattered configuration. As seen in the image, the pores spacing in the untreated soft clay soil is quite large, indicating a high permeability of the clay soil. The treated sample Figure (B and C) at 5µm and Figure (D) at 1 µm was also scanned through a scanning electron microscope (SEM) to determine how the microstructure behaved and the appearance of the cementation products, as shown in the figure above. The cemented product's appearances as a result of the pozzolanic reaction with Sodium Silicate (TX-85) and Biomass Silica (SH-85) are depicted in Figure (D). Calcium Aluminate Hydrate (CAH) is a mineral that occurs when alumina alters with calcium hydroxide (Ca (OH) 2). In comparison to sample 7D and 14D, the reaction occurred during the cementitious process with water to produce Calcium Aluminate Hydrate (CAH) and cementitious that were more clearly defined and solid. Additionally, this resulted in a more porous and dense soil fabric. Additionally, the filling of voids in stabilised soil increases the bonding and interlocking forces between soil particles, which accounts for the majority of the strength gained. This demonstrates how the constituents of treated soils, soft clay, and TX-85, and SH-85, can change or mix in a variety of ways, as illustrated in this figure.

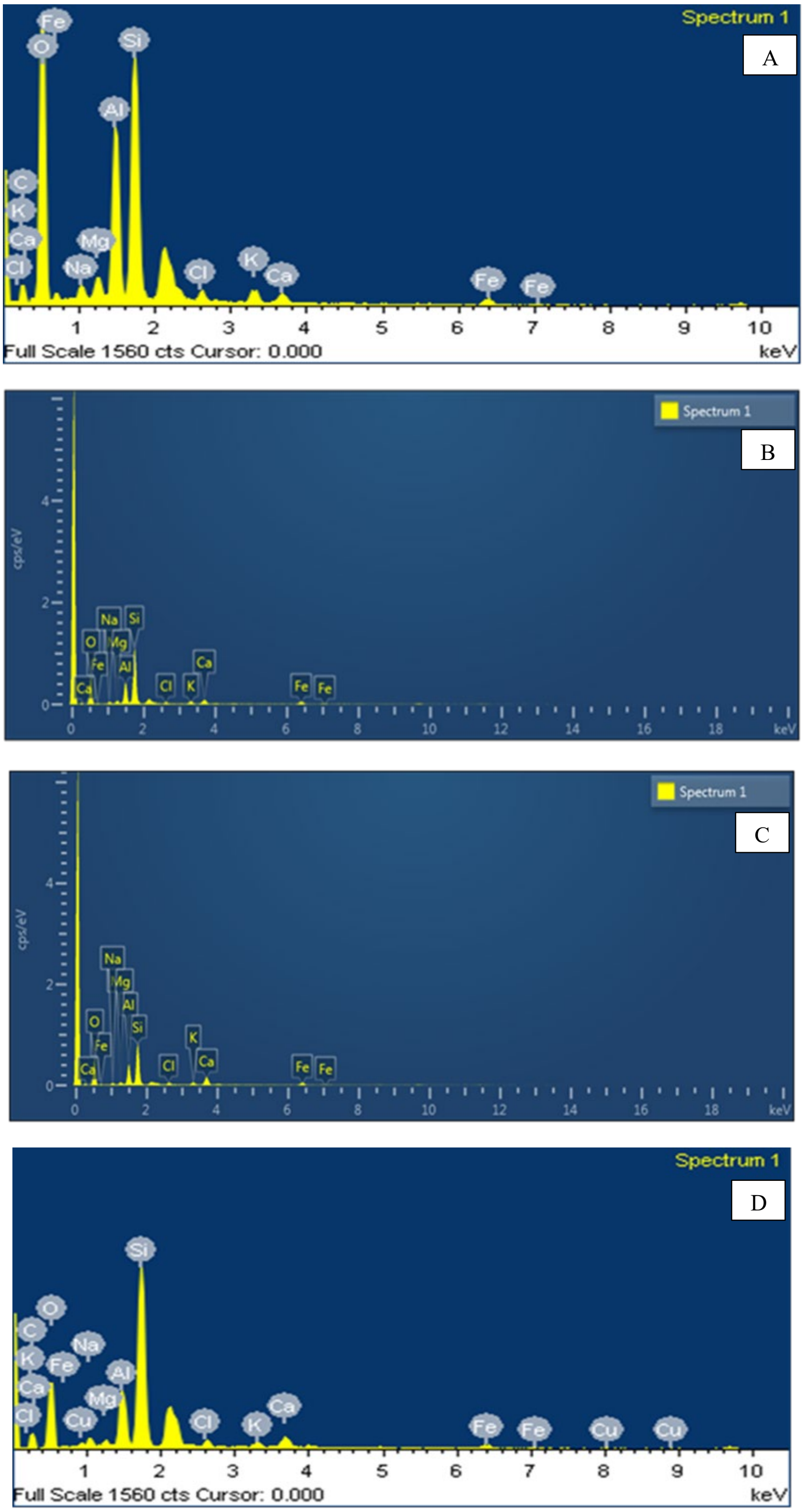

Figure 3: EDX Results for Untreated (A) and (3%L+9%P) at different curing times, 7 days (B), 14 days (C) and 28 days (D)

Energy Dispersive X-ray (EDX) spectroscopy was used to determine the chemical composition of untreated clay soil and treated samples. The EDX test can be used to determine whether samples have undergone changes, reactions, or the addition of new chemical elements. As illustrated in the figures above, the EDX result will display the concentration of chemical elements in samples as a spectrum graph. The EDX analysis of untreated BPSC (A) reveals that Oxygen (O) has the highest weight percentage of 51.53 %, followed by Silicon (Si) (17.52 %), Carbon (C) (11.02 %), and Alumina (Al) (10.27 %). These chemical elements clearly outnumber all other elements. These chemicals were chosen to demonstrate the existence of novel minerals in accordance with the XRD results. Oxygen (O) has fluctuated slightly to 51.98 % in figure (B), while Silicon (Si) has increased to 31.44 %, Carbon (C) has disappeared, and Alumina (Al) has decreased dramatically to 7.19 %. Meanwhile, figure (C) demonstrated that Oxygen (O) content increased to 53.07 %, Silicon (Si) content decreased to 21.06 %, Carbon (C) content remained constant, and Alumina (Al) content increased to 10.17 %. The 14-day curing time sample demonstrates definitively that the weight percentage does not differ significantly from the untreated and 7-day curing time samples. Oxygen (O) has decreased to 26.94 % in Figure (D), followed by Silicon (Si) at 19.19 %, Carbon (C) at 17.51 %, and Alumina (Al) at 5.30 %. The weight percentage changes significantly between the untreated clay soil and 14-day curing time samples after 28 days.

3.2 The Characteristics of pH

The litmus paper was used to determine the pH value of the soil specimen. A 10g sample was weighted and diluted with 50ml of deionized water. The litmus paper was put inside the solutions at least 2 seconds. Then, shake off the excess liquid from litmus paper and wait 3 to 5 seconds. After that, compare the color of litmus paper to get the actual result of pH value of the solutions. Figure 4 shows the pH experiment carried out by accordance to the specification for the untreated clay soil scale of pH is 7 which is natural. The pH of the treated clay soil mix with Sodium Silicate (TX-85), and Biomass Silica (SH-85) for mixture (3%L+9%P) the results was 10 which is indicating that is alkaline as shown in Figure below.

4. Conclusion

The microstructure behavior of stabilized soft clay soils was altered by adding Sodium Silicate (TX-85) and Biomass Silica (SH-85). The microstructure behavior of stabilized soft clay soils was investigated and measured using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Microscopy (EDX). The following summarizes the findings of this study:

i. In unsaturated clay soil, no changes occur, no chemical reaction (pozzolanic reaction) occurs, and no cementation product is produced.
ii. As seen in the image, the pores spacing in the untreated soft clay soil is quite large, indicating a high permeability of the clay soil.
iii. Significant improvements and reactions between soft clay soils and stabilizers such as Sodium Silicate (TX-85) and Biomass Silica (SH-85) are less visible in treated samples (3%L+9%P) after 7, 14, and 28 days curing time.
iv. However, XRD analysis shows Calcium (Ca), has appeared in treated samples, which will produce the pozzolanic reaction, Calcium Aluminate Hydroxide (CAH), and new cementations products into the soil structure.
v. Additionally, the concentration of Silica (Si) in treated soils is increased because of the addition of Sodium Silicate (TX-85) and Biomass Silica (SH-85).
vi. The SEM analysis revealed a higher concentration of crystalline cementation products, as evidenced by the white structure that coated the treated soils.
vii. Furthermore, SEM experiments show that the pores space has been reduced and minerals from Sodium Silicate (TX-85) and Biomass Silica (SH-85) have been used to fill the voids in the structure.

viii. In terms of EDX results, there is an increase in concentration and the presence of new chemical elements in the treated samples when compared to the untreated samples.

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