Strength Development and Microstructural Behavior of Soils Stabilized with Palm Oil Fuel Ash (POFA)-Based Geopolymer

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Abstract: Using geopolymer in soil stabilization has gained much attention recently due to its efficiency in improving soil properties and being environmentally friendly at the same time. This research aims to investigate the effect of palm oil fuel ash (POFA)-based geopolymer on soft soil stabilization. The mechanical and microstructural performance of two types of clay soil treated with geopolymer produce from POFA material was the focus of this study. In this respect, a series of unconfined compression and direct shear tests were conducted to investigate the mechanical properties of soils treated with POFA-based geopolymer. Furthermore, the microstructural changes in the treated samples were analyzed using field emission electron microscopy (FESEM) and energy dispersive X-ray (EDX). In accordance with the results, it was indicated that the shear strength of both soils soared by increasing the dosage of POFA-based geopolymer. Geopolymer with 40% POFA of the dry weight of soils yielded the highest UCS value at both curing periods, 7 and 28 days. Furthermore, the microstructural analysis revealed material modifications (N-A-S-H gel formation) related to strength enhancement. These results suggest the potentiality of using a POFA-based geopolymer binder to stabilize soft soil.

Keywords: alkaline activator; ground improvement; soft soil; stabilization; compressive strength; direct shear; alkali activation

1. Introduction

Population increase and space restrictions make soil enhancement essential to provide a strong subsoil layer to facilitate infrastructure, buildings and railway construction. High plasticity soil causes collapses in the foundations, road networks and water systems, so it is considered a massive challenge in geotechnical engineering. Moreover, the development of infrastructural and industrial sectors consumed a large number of building materials resulting in a detrimental effect on the environment. Therefore, the need of having a stable and strong subgrade soil layer to carry the applied loads is a challenging task that brought the idea of soil stabilization.

The most widespread binders, cement and lime, were used in soft soil stabilization during the past decades due to their efficiency in enhancing soil strength, reducing settlement [1,2] and controlling shrinkage and swelling [3]. Although they are effective in improving the geotechnical properties of soil, there are some disadvantages accompanying these binders such as cost and environmental effects. For 1 ton of cement production, 1.5-ton of raw materials will be consumed, accompanied by 5.6 GJ/ton energy consumption and approximately 0.95 tons of CO$_2$ release [4–6], while lime production releases around 0.86 tons of CO$_2$ per ton of lime [7]. Moreover, about 7% of all greenhouse gases in the atmosphere are CO$_2$ emissions caused by cement manufacturing [8,9]. At the same time, waste disposal—mainly fly ash from plants—has greatly increased in recent years and the majority of the waste is disposed of conventionally: landfills [10].

In recent years, researchers began to look for binders that can provide a solution for waste disposal, replace the disadvantages of calcium-based binders and be environmen-
tally friendly, simultaneously. This developed the idea of what is termed geopolymer to be considered as the next generation. Geopolymer (also described as alkali-activated materials) is an inorganic polymer formed of Al and Si ions alternately tetrahedrally connected with shared oxygen atoms. The general formula for the description of the chemical composition of geopolymers is \( Mn[-(SiO_2)_z-AlO_2]_n.wH_2O \), in which \( z \) can be 1, 2 or 3, \( M \) represents the alkali cation (i.e., K or Na) and \( n \) is polymerization degree. Consequently, the structure of geopolymer can be polysialate (–Si–O–Al–O–), polysialate-siloxo (–Si–O–Al–O–Si–O–) or polysialate-disiloxo (–Si–O–Al–O–Si–O–Si–O–), based on the silica amount in the precursor [11].

The geopolymerization process can be summarized as follows [12,13]: dissolving and breaking of the reactive aluminum and silicate chains in the aluminosilicate material (precursor) because of the pH increase generated by the activator solution, and formation of monomers. Subsequently, polycondensation reactions take place to accumulate and precipitate the resulting products to form an amorphous three-dimensional structure which will tend to crystalize.

An enormous amount of geotechnical research has shown the effectiveness of using geopolymers produced from different aluminosilicate materials in soil stabilization such as metakaolin [14,15], olivine [16], class F fly ash [17], class C fly ash [18] and granulated blast furnace slag [19,20]. Between the different aluminosilicate precursors, palm oil fuel ash (POFA) utilization merits more recognition due to its high potentiality to produce geopolymer and its abundance as a waste, especially in Southeast Asian countries such as Malaysia [21].

In very limited attempts, POFA, which is agricultural waste, has been employed in geopolymer production for soft soil stabilization purposes. Abdeldjouad et al. [22] investigated the use of POFA along with potassium hydroxide (KOH) for an alkali activation process to be utilized in clayey soil stabilization. Laboratory tests focused on the shear strength and microstructural tests including scanning electron microscope (SEM) and x-ray diffraction tests. Sukmak et al. [23] studied stabilization of subgrade soft soil with geopolymer made from POFA as a precursor and sodium hydroxide (NaOH) with sodium silicate (\( Na_2SiO_3 \)) as alkali activators to find the optimum \( Na_2SiO_3/NaOH \) ratio and POFA/soil ratio based on the compressive strength developed and the microstructural improvement of the soil. Moreover, Khasib and Nik Daud [24] evaluated the effectiveness of using POFA-based geopolymer to stabilize high plasticity silt to conclude that physical and chemical properties of soft soil were enhanced by POFA-based geopolymer.

Despite these developments and research achievements, using geopolymer for soil stabilization applications is still at its early stages and requires more demonstration. Moreover, comprehensive studies about geopolymers produced from POFA as a precursor with NaOH and \( Na_2SiO_3 \) as activators in soft soil stabilization are limited. The role of different clayey soils and alkaline solutions type in geopolymerization need to be well addressed as well. However, considering the method of geopolymer application to soil, previous research mixed POFA with the soil and then added the alkaline activators. Therefore, for a better understanding of the effect of POFA-based geopolymer on soil’s properties, a well-designed geopolymer should be prepared and applied to the soil.

This research focuses on the effectiveness of using POFA-based geopolymer in clayey soil stabilization. Due to that matter, the changes in the mechanical performance of two types of clayey soil before and after treatment with POFA-based geopolymer were evaluated based on unconfined compression (UCS) and direct shear tests (DST). Furthermore, the changes in the microstructural composition of both treated and untreated soils were determined with the aid of field emission electron microscopic (FESEM) with energy dispersive X-ray tests (EDX) to understand the underlying mechanisms of stabilization.
2. Materials and Methods

2.1. Materials

2.1.1. Soil

Two soil samples were investigated in this research. Soil 1 was a residual soil that is widely distributed in Universiti Putra Malaysia, Selangor, Malaysia, while Soil 2 was a typical marine clay obtained from Kampung Sungai Burong, Tanjong Karang, Selangor, Malaysia, and was in a slurry state. Table 1 illustrates the physical and engineering characteristics of both soils investigated in this study according to the British Standard (BS 1377:1990). Considering pH values of both soils, Soil 1 was nearly neutral whereas Soil 2 revealed a small acidic behavior. In addition, the high specific gravity values (GS) indicate very low organic matter found in the soils which refers to non-agricultural areas where the samples have been taken. The particle's size distribution of selected soils is depicted in Figure 1. As stated by the Unified Soil Classification System (USCS), Soil 1 was categorized as low plasticity clay (CL) and Soil 2 was high plasticity clay (CH). These types of clayey soils are usually too weak and unsuitable for infrastructure facilities construction such as highways. The chemical composition of the soils tested is demonstrated in Table 2.

Table 1. Geotechnical characteristics of untreated soils.

| Soil Properties          | Unit   | Standard       | * S1   | * S2   |
|--------------------------|--------|----------------|--------|--------|
| Initial moisture content | %      | BS 1377: Part 2| 22.9   | 98.9   |
| pH                       | -      | BS 1377: Part 3| 7.35   | 6.53   |
| Specific gravity         | -      | BS 1377: Part 2| 2.77   | 2.80   |
| Plastic limit            | %      | BS 1377: Part 2| 22.2   | 35.6   |
| Liquid limit             | %      | BS 1377: Part 2| 41.6   | 80.7   |
| Plasticity Index         | %      | BS 1377: Part 2| 19.4   | 45.1   |
| Shrinkage limit          | %      | BS 1377: Part 2| 6.2    | 15.0   |
| USCS                     | -      | -              | CL     | CH     |
| Optimum moisture content | %      | BS 1377: Part 4| 15.5   | 34.4   |
| Maximum dry density      | Mg/m³  | BS 1377: Part 4| 1.76   | 1.31   |

* Note: S1—Soil 1; S2—Soil 2.

Figure 1. Particle size distribution S1 (low plasticity clay (CL)), S2 (high plasticity clay (CH)) and palm oil fuel ash (POFA).
Table 2. Chemical composition of CL, CH and POFA.

| Component          | Soil 1 (CL) | Soil 2 (CH) | Raw POFA (% by Mass) | Treated POFA (% by Mass) |
|--------------------|-------------|-------------|-----------------------|--------------------------|
| Silicon dioxide (SiO$_2$) | 36.71       | 42.46       | 39.23                 | 43.52                    |
| Aluminum oxide (Al$_2$O$_3$) | 27.18       | 22.65       | 26.2                  | 24.7                     |
| Calcium oxide (CaO)    | 0.04        | 0.13        | 8.64                  | 9.73                     |
| Magnesium oxide (MgO)  | 0.78        | 0.52        | 6.12                  | 4.31                     |
| Iron oxide (Fe$_2$O$_3$) | 9.35        | 7.69        | 5.99                  | 6.68                     |
| Potassium oxide (K$_2$O) | 5.32        | 1.66        | 2.27                  | 1.56                     |
| Sulfur Trioxide (SO$_3$) | 0.06        | 0.04        | 1.63                  | 5.28                     |
| Sodium oxide (Na$_2$O) | -           | -           | 0.89                  | 0.18                     |
| LOI (%)              | -           | -           | 8.79                  | 1.26                     |

2.1.2. Palm Oil Fuel Ash (POFA)

The sample of POFA utilized was provided from a factory in Dengkil Selangor, Malaysia. From the chemical analysis of raw POFA, as shown in Table 2, it consists of a high silica (SiO$_2$) amount which exhibits its high potentiality to produce an efficient geopolymer. It was dried inside an oven at 105 ± 5 °C for 24 h and then sieved through a 300 µm sieve [25]. In order to achieve a high level of fineness, the sieved POFA was ground in a revolving drum for 30,000 cycles for about 16 h [26,27]. The particle’s size distribution of POFA after grinding is shown in Figure 1, whereby 90% of particles are smaller than 0.048 mm and the mean particle size (d50) is 0.017 mm, in which d50 refers to the diameter where 50% of the particles are smaller than it. Loss of ignition (LOI) for ground POFA was 8.79%, which is an indication of the high unburned carbon content found in the sample. Thus, an electrical furnace was used to heat the ground POFA at 440 ± 5 °C for one hour [28,29]. After heating, LOI was reduced to 1.26%. The chemical composition of the treated POFA sample using an X-ray fluorescence (XRF) test is illustrated in Table 2.

2.1.3. Alkaline Activator

The liquid alkaline activator (L) selected for this research was a blend of sodium hydroxide (NaOH) and sodium silicate (Na$_2$SiO$_3$) solutions as advised by many researchers. They were purchased from a chemical supplier in Kuala Lumpur, Malaysia; NaOH was in pellets with 99% purity while Na$_2$SiO$_3$ was in a liquid phase (32.56% SiO$_2$, 17.82% Na$_2$O and 46.37% water by mass). A 12 molarity NaOH solution was prepared by dissolving the NaOH pellets in the distilled water. It was left for one day before being mixed with Na$_2$SiO$_3$ due to the high temperature released after mixing with water. The Na$_2$SiO$_3$/NaOH ratio was kept constant at 2.5.

2.1.4. POFA-Based Geopolymer

In this study, the solid to liquid ratio (S/L) was kept constant at 1.32. The S/L ratio represents POFA (solid) to alkaline activator solution. All these geopolymer parameters were found to be the optimum in POFA-based geopolymer production in terms of strength [30]. POFA was mixed directly with a liquid alkaline activator, which consists of NaOH and Na$_2$SiO$_3$, inside a mixer for about 10 min at slow mode to ensure the homogeneity of the geopolymer prepared. A typical POFA-based geopolymer mixture before application to soil is shown in Figure 2.

2.1.5. Samples Preparation

The method of treating soils with POFA-based geopolymer was conducted at controlled water content and dry density to simulate the conditions of soil in-situ. In this study, soils were mixed with four dosages of POFA-based geopolymer. These four dosages are based on POFA being 10%, 20%, 30% and 40% of soil’s dry weight. Soils were mixed with geopolymer and free water in the same mixer used for preparing the geopolymer for approximately ten minutes until the mixture showed total homogeneity. The free water quantity was varied to allow for the investigation of optimal compaction parameters, as
will be discussed in Section 2.2.1. UCS and direct shear test specimens were prepared at the moisture content related to the highest dry density from the compaction test. Moreover, to ensure the consistency and representativeness of the tests, three duplicate specimens were prepared for each test and the results were approved if they differed by up to 5% from the average. Table 3 depicts the mixtures design and testing for both types of soils.

![Figure 2. POFA-based geopolymer before mixing with soil.](image)

**Table 3. Mixing proportions of different tests.**

| Series | Test Number             | Curing Days | UCS  | DST |
|--------|-------------------------|-------------|------|-----|
| Soil 1 | Untreated CL            | 7 & 28      | -    | -   |
|        | CL-G10PA                | 7 & 28      | -    | -   |
|        | CL-G20PA                | 7 & 28      | -    | -   |
|        | CL-G30PA                | 7 & 28      | -    | -   |
|        | CL-G40PA                | 7 & 28      | -    | -   |
| Soil 2 | Untreated CH            | 7 & 28      | -    | -   |
|        | CH-G10PA                | 7 & 28      | -    | -   |
|        | CH-G20PA                | 7 & 28      | -    | -   |
|        | CH-G30PA                | 7 & 28      | -    | -   |
|        | CH-G40PA                | 7 & 28      | -    | -   |

Note: G10PA, G20PA, G30PA and G40PA—geopolymer produced from 10%, 20%, 30% and 40% POFA of soil’s dry weight.

In this study, it was assumed that the density/unit weight of all samples prepared has achieved the same value as observed from the compaction curves. However, the moisture content values of samples after preparation for UCS and direct shear testing were calculated to check the proximity of these values from the moisture content found from the compaction test, as indicated in Table 4. All standard deviation values are less than 5%, which indicates the small deviation of the measured values from the optimum moisture content (OMC) obtained from compaction testing.
2.2. Methods

2.2.1. Standard Proctor Compaction Test

Optimum conditions of soil (maximum dry density (MDD) and optimum moisture content (OMC)) are key considerations for assessing both the quality assurance and quality control of in-situ soil. With this regard, a standard proctor compaction test was used to determine the MDD and OMC of both soils before and after treatment with POFA-based geopolymer according to BS 1377–1990: Part 4. The test was carried out for all treated specimens immediately after mixing the soil with the geopolymer.

### Table 4. Calculated moisture content (%) after unconfined compression (UCS) and direct shear test (DST) samples preparation.

| Test Series | OMC from Compaction Test (%) | Measured Water Content after Sample Preparation (UCS) (%) | Measured Water Content after Sample Preparation (DST) (%) | * Standard Deviation (UCS) (%) | * Standard Deviation (DST) (%) |
|-------------|-------------------------------|--------------------------------------------------------|--------------------------------------------------------|-------------------|-------------------|
| Untreated CL | 15.5                          | 12.8                                                   | 14.8                                                   | 2.7               | 0.7               |
| CL-G10PA    | 14.6                          | 11.3                                                   | 13.5                                                   | 3.3               | 1.1               |
| CL-G20PA    | 13.4                          | 11.1                                                   | 13.1                                                   | 2.3               | 0.3               |
| CL-G30PA    | 13                            | 12.6                                                   | 12.7                                                   | 0.4               | 0.3               |
| CL-G40PA    | 12.8                          | 12.2                                                   | 12.2                                                   | 0.6               | 0.6               |
| Untreated CH | 34.4                          | 32.6                                                   | 33.6                                                   | 1.8               | 0.8               |
| CH-G10PA    | 29.9                          | 27.8                                                   | 29.4                                                   | 2.1               | 0.5               |
| CH-G20PA    | 24.5                          | 22.9                                                   | 22.4                                                   | 1.6               | 2.1               |
| CH-G30PA    | 23                            | 22.5                                                   | 21.4                                                   | 0.5               | 1.6               |
| CH-G40PA    | 18.6                          | 18.4                                                   | 18.3                                                   | 0.2               | 0.3               |

* standard deviation was calculated as the difference between the optimum moisture content (OMC) from the compaction test (%) and the measured water content after preparation of samples (%).

2.2.2. Unconfined Compressive Strength (UCS)

To evaluate the strength performance of POFA-based geopolymer stabilized soils, UCS testing was conducted following BS1377: Part 7:1990 with a vertical displacement (strain rate) of 1 mm per minute. Preparation of samples was done immediately after the aforesaid mixing process using static compaction of soil in three similar layers at their OMC and MDD obtained from the compaction test inside a steel mold (50 mm diameter × 100 mm height) using a 45 mm diameter rod. The cylindrical samples were extruded and wrapped with polyethylene coatings and put inside the curing chamber to prevent loss of moisture and cured at room temperature (25 ± 2 °C) at 50% relative humidity for two periods, 7 and 28 days.

2.2.3. Direct Shear Test (DST)

To determine the effect of POFA-based geopolymer on soil cohesion (c) and friction angle ($\phi$), a direct shear test (DST) was conducted in accordance with BS1377: Part 7:1990. The specimens for the DST were prepared at their OMC and MDD in the same way used for preparing UCS samples with static compaction inside the shear box (60 mm × 60 mm × 20 mm), and all tests were conducted at 100 kPa, 200 kPa and 400 kPa normal pressures with 1.5 mm/min strain rate until failure of the soil was noticed.

2.2.4. Microstructural Analysis

To examine the micromorphological changes and chemical composition of POFA-based geopolymer stabilized samples and highlight the mechanism of stabilization, field emission scanning electron microscopic (FESEM) along with energy dispersive X-ray (EDX) was carried out on both natural soil samples and some stabilized specimens.

The specimens were tested using a Hitachi SU8010 machine for the analysis of both FESEM and EDX. For sample preparation, small representative test specimens were collected from the specimens crushed after the UCS test and then installed on aluminum stubs with two sides of carbon tabs. A thin film of platinum was used as a coating material in a sputter coater to achieve surface conductivity.
3. Results and Discussion

3.1. Compaction Behavior

The moisture content-dry density relationships of untreated and treated samples with POFA-based geopolymer of both soils are shown in Figure 3a,b. For untreated CL and CH, the maximum dry density (MDD) was 1.76 Mg/m$^3$ and 1.31 Mg/m$^3$, whereas the optimum moisture content (OMC) was 15.5% and 34.4%, respectively. The results indicate a strong relationship between moisture content, dry density and POFA-based geopolymer dosage. At all dosages of geopolymer, MDD revealed an increment while OMC showed a decreasing pattern. Obviously, POFA-based geopolymer-coated soil particles performed as a lubricant which reduced the frictional resistance and enhanced sliding among soil particles resulting in an increase in maximum dry density. This lubrication effect will decrease the needed amount of free water to achieve optimal conditions since the geopolymer effect will replace the function of water, thus decreasing the optimum moisture content [31]. Moreover, the lubrication induced by the geopolymer improved compaction by decreasing the volume of air pockets significantly, which resulted in an MDD increase and OMC decrease [32]. CL- and CH-treated specimens with POFA-based geopolymer with 40% POFA of soil’s dry weight (CL-G40PA, CH-G40PA) exhibited the highest MDD (1.87 Mg/m$^3$ and 1.51 Mg/m$^3$, respectively) and lowest OMC (12.8% and 18.6%, respectively) compared to all treated samples. This outcome was anticipated due to the highest availability of alkali source ions accompanying this geopolymer mix that could dissolve the majority of Al and Si found in POFA compared to other geopolymer dosages [33]. However, there is no noticeable enhancement in MDD and OMC for treated CL soil between G20PA and G40PA geopolymer dosages. The increase in MDD of soil is usually an expectation of initial strength gain and the modifications in the compaction properties can be seen as a benefit and shall be included in any approach of stabilization using geopolymer.

3.2. Unconfined Compressive Strength

Figure 4 illustrates the stress-strain relationships of CL and CH soils before and after treatment with POFA-based geopolymer at curing periods 7 and 28 days. The highest strain/deformation before failure was presented by untreated samples at a strain value of nearly 3.5% and 3% for CL and CH, respectively. However, there is an axial stress increase via the addition of POFA-based geopolymer to the soils accompanied by a strain failure reduction.

![Figure 3](image-url)
Figure 3. Moisture content-dry density relationships for soils treated with POFA-based geopolymer: (a) CL, (b) CH.

Figure 4. Stress-strain curves of POFA-based geopolymer-treated soils: (a) 7 days CL, (b) 28 days CL, (c) 7 days CH, (d) 28 days CH.
The POFA-based geopolymer impact on the compressive strength (UCS) of both soils is depicted in Figure 5a,b. Accordingly, the results show that the UCS of both soils is highly dependent upon the geopolymer dosage and curing time. The higher the dose of POFA-based geopolymer applied to the soils, the higher is the maximum compressive strength attained. Treated soil samples with G40PA show the maximum strength developed among geopolymer mixtures with some deviations considering the curing time, which resulted in very strong and dense mixtures. The results show that a moderate strength change was developed by the geopolymer treated specimens in the initial stages (7 days) and a greater strength was achieved after 28 days of curing. The improvement in UCS values with time can be addressed by two mechanisms. The first one is the development of highly cross-linked sodium aluminate silicate hydrate gel (N-A-S-H), which becomes stronger with time and was reported as the main mechanism by which the strength is enhanced [34]. The second minor mechanism is the pozzolanic reaction producing calcium silicate hydrate (C-S-H) due to the reaction of soluble calcium and silica found in the precursor (POFA) [22]. For the CL sample, the UCS increased from 0.26 MPa for untreated soil to 1.71 MPa and 4.18 MPa for soil treated with G40PA (CL-G40PA) at 7 and 28 days, respectively. Regarding CH soil, the UCS of untreated soil was 0.13 MPa and reached 1.06 MPa and 2.86 MPa when treated with G40PA (CH-G40PA) at 7 and 28 days, respectively. The Department of Public Works in Malaysia stated that a minimum strength of 0.8 MPa for stabilized subgrade soil should be achieved [35]. Based on this, most samples stabilized with the geopolymer in this study meet the requirement after 28 days of curing. This improvement indicates the high impact of POFA as a precursor, which is rich with reactive Al and Si ions in the existence of alkali cations, forming a strong and dense soil mixture by developing Al-O-Si and Si-O-Si chains associated with geopolymerization [36]. The fly ash reactivity is also represented by the small size of particles since it has a high influence on the strength of the geopolymer produced [37] and the amount of residual carbon that may hinder the process of alkali activation [38,39]. Moreover, the noticeable improvement in strength between 7 and 28 curing days confirms that the geopolymer reaction is time-dependent and soil particles bonding with POFA-based geopolymer progresses with time. However, the dependence of soils’ UCS on the dosage of geopolymer refers to the increase in the formation of the cementitious products accompanied by the geopolymer content increase in the soil [40].

![Figure 5. Compressive strength (UCS) of untreated and treated samples: (a) CL, (b) CH.](image-url)
3.3. Direct Shear Test

The parameters of shear strength including cohesion (c) and friction angle (φ) of untreated and treated samples are demonstrated in Figure 6a,b. Untreated CL showed better shear parameters compared to untreated CH. Comparatively, there was a considerable increase in c of both soils by increasing the POFA-based geopolymer dosage, and the highest values were found for specimens treated with G40PA.

![Figure 6. Parameters of shear strength for soils treated with G10PA, G20PA, G30PA and G40PA: (a) CL, (b) CH.](image)

The c value of CL-G40PA was 65 kPa, which is nearly 1.5 times that of untreated CL soil, while c for CH-G40PA was 53 kPa, which is more than twice the c of untreated CH. This enhancement and increase in the shear strength parameters without curing can be attributed to the cations exchange between the high concentration ions in the geopolymer binder and those of a similar charge found on the soil particles, which leads to their flocculation and subsequent improvement [41].

Similar behavior for soils was observed by Latifi et al. [42] when chemically treated soils with geopolymer were studied to confirm that geopolymer can improve soil cohesion. However, a marginal increase in φ was obtained for all treated samples, which is typical in chemically treated clay soils [43,44]. From the findings, it can be observed that the pattern of strength parameters was almost similar for all the dosages of geopolymer content in both soils.

3.4. Microstructural Analysis

For a deeper explanation of the stabilization mechanism, Figure 7 shows FESEM micromorphology pictures of the fractured surface of 28 day-cured untreated samples (CL and CH) and POFA-based geopolymer stabilized samples (CL-G40PA and CH-G40PA) since they achieved the best UCS performance. It is claimed that the higher strength geopolymer is associated with a firmer internal microstructure [45]. As can be seen in Figure 7, untreated CL has shown a considerable open structure with weak or no bonds between soil particles, in which pores can be clearly seen in Figure 7a, whereas the cluster shape particles in Figure 7b refer actually to the isolated soil particles of untreated CH, which have a flaky and weak structure with some arrangement among them (particles became close to each other) due to the compaction effect. However, the FESEM pictures in Figure 7c,d indicate that the activator solution stimulated the POFA precursor because of chemical changes that occurred internally within the particles. The microcracks shown...
in the treated samples may be due to increased stress during the UCS test or shrinkage throughout the curing process. Treated samples (CL-G40PA and CH-G40PA) have experienced a significant enhancement in the soil structure. POFA-based geopolymer has filled interparticle voids leading to a dense and compact structure. The enhancement mechanism is explained by the production of the main geopolymer gel, which is sodium aluminate silicate hydrate (N-A-S-H) that forms well-developed 3-D networks of Si-O-Al and Si-O-Si (cementation products) and binds the soil particles together throughout the curing time [46–48]. However, the mixture of CH-G40PA indicated some roughness and particles are not well coated with the gel compared to CL-G40PA, which has shown a smooth structure. This indicates that geopolymer’s efficiency in improving soil properties is highly dependent on the soil’s type.

![Field emission electron microscopic (FESEM) micrographs](image)

**Figure 7.** Field emission electron microscopic (FESEM) micrographs: (a) CL, (b) CH, (c) CL-G40PA, (d) CH-G40PA.

This can also be confirmed by the results of EDX where the chemical composition indicates detection of Si and Al elements in the samples treated, as explicitly shown in Figure 7, which implies that the POFA-based geopolymer has taken place in treated clayey
soils. Table 5 demonstrates the weight percentages and the ratios of the main elements found in the samples tested using EDX at the selected points (1, 2 and 3) in Figure 7. Point 1 is relatively smooth in both treated soils, and the EDX detected major Si, Al and O elements and minor Ca, Na, Mg and Fe elements. These elements are the basic main components of the natural soil. At points 2 and 3, the highest percentage of elements was for silicon, which likely represents the geopolymer and pozzolanic reactions. The sudden increase in the percentage of Na indicates the possibility of geopolymer gel formation (NASH) [49]. It was proved that low calcium fly ash-based geopolymer results in N-A-S-H formation as a dominant gel, and very low and slower development of calcium silicate hydrate (C-S-H) gel [50]. The decline in the percentages of Si and Al (from point 1 to 3) was mainly a result of the geopolymer reaction with the time elapsed via dissolving alumina and silica, confirming the significant growth in strength [49]. However, the calculated ratios of Si:Al explain that there were changes in the composition of soils due to the coating effect or the formation of new minerals as a result of alkaline activation reactions [51].

Table 5. Chemical composition and ratios of elements in the selected specimens.

| Sample   | Point Number | Si   | Al   | Na   | O    | Ca   | Si/Al |
|----------|--------------|------|------|------|------|------|-------|
| CL-G40PA | 1.00         | 21.53| 8.75 | 0.31 | 36.20| 0.06 | 2.46  |
|          | 2.00         | 17.44| 6.08 | 0.22 | 40.79| 0.09 | 2.87  |
|          | 3.00         | 9.60 | 2.90 | 4.27 | 33.56| 1.38 | 3.31  |
| CH-G40PA | 1.00         | 23.14| 14.46| 0.49 | 44.37| 0.24 | 1.60  |
|          | 2.00         | 37.08| 18.17| 0.43 | 41.00| 0.49 | 2.04  |
|          | 3.00         | 21.95| 8.34 | 5.10 | 39.82| 2.32 | 2.63  |

4. Conclusions

This research aimed to investigate the effect of POFA-based geopolymer on clayey soil stabilization in order to solve the issues corresponding to weak soil, protecting the environment by reducing energy consumption and using locally available agricultural waste in an efficient way.

- Based on the findings, the prepared geopolymer consisting of POFA as source material and NaOH along with Na$_2$SiO$_3$ as alkali activators was successful in enhancing the properties of the soil. However, the geotechnical properties of soil treated using POFA-based geopolymer depend mainly on the dosage of the binder, the host soil type and the curing conditions.
- POFA-based geopolymer-treated clayey soil specimens have been shown to have higher MDD values than untreated samples, with a drop in the accompanying OMC as the geopolymer dosage rises, which is an indication of improvement.
- The UCS results demonstrated that the maximum strength was accomplished by the geopolymer mixture consisting of the highest POFA percentage (G40PA) at both curing times. At 28 days, the effect of POFA-based geopolymer was more obvious due to the development of greater strength compared with 7 days curing. In addition, the shear strength parameters—especially cohesion of soil—were improved due to the addition of POFA-based geopolymer even without curing.
- Apart from the positive outcomes shown by the current research, the application of POFA-based geopolymer to low and high plasticity clayey soils played a significant role in changing the soils’ structure where the detached particles of soils in treated mixes have shown that they become more closely tied together, with the voids apparently filled, leading to a significant improvement in strength, as depicted in FESEM-EDX photographs. In addition, EDX confirmed that NASH gel is the most likely formed gel in the treated soils.
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