



Investigation on Soil Strength and Microstructure of Palm Oil Boiler Ash with Sodium Hydroxide and Sodium Silicate as Alkaline Solution

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Abstract: Palm oil boiler ash is a type of industrial waste from palm oil production, which abundantly disposed on landfill, and contaminate the soil and environment. Recently, boiler ash as a by-product is introduced as an additive in chemical stabilization, known as geopolymer. An alkaline solution functions to activate the silica-alumina structure bonding to replace the Ordinary Portland Cement in concrete application but few in soil stabilization study. This study investigates the strength of laterite with a geopolymer. Boiler ash was chosen as source material, with sodium hydroxide and sodium silicate mixed at a ratio of 1:2. Unconfined compressive test (UCT) and SEM were conducted for laterite soil with different geopolymer percentages. The test was conducted by adding a different geopolymer percentage at 0%, 5%, 10%, 15%, and 20% mixed with laterite soil. The soil sample was cured for seven days for an unconfined compression test (UCT). The result shows that the highest compressive strength was obtained at 15% of geopolymer in laterite soil at 340kPa. SEM tests show that the increased compressive strength was attributable to the 15% of the geopolymer, which had a compact and dense structure and less unreacted raw materials. In conclusion, the results indicated that 15% of geopolymer gives optimum value in enhancing laterite properties' strength. The findings support boiler ash usage as by-products in geopolymers' production for potential use in soil strength stabilization.

Keywords: Unconfined compressive test, geopolymer, palm oil boiler ash, laterite soil

1. Introduction

Over the past few decades, Malaysia's palm oil industry has grown significantly, which has helped economy growth. However, coal power plants' producers have contributed to the abundance of boiler ash as waste materials and leads to environmental issues (Abdullah & Sulaiman, 2013). Malaysia will reach 15.4 million tonnes of palm oil projected in 2016 – 2020 (Teoh, 2000). The high production combustion from palm oil shell and palm fiber generates ash known as boiler ash. According to Abdullah & Sulaiman (2015), pollutant gases such as nitrogen oxide and sulfur oxide can be eliminated using bottom ash as an absorbent to remove the pollutant gases. Climate change is taking a toll on governments around the world as it is a challenge to determine the most effective environmental policies to promote

eco-innovation. Therefore, researches on the use of boiler ash waste as a potential material to become a construction material were initiated.

Dissanayake, Senanayake & Nasvi (2017) study class F fly ash (low calcium) and bottom ash stabilizers at 8, 16, and 24 % to treat expansive soil properties. The mixture of fly ash and bottom ash with expansive soil increases up to 16 % ash content in the UCS test, and then it starts decreasing with any further addition of the ash content. The UCS increase is higher for fly ash as compared with bottom ash in treated expansive soil. In the swell testing of admixture with expansive soil, a reduction of 70% for fly ash and 48% for bottom ash were observed, and the properties of expansive soil were enhanced. It can be concluded that both fly ash and boiler ash can reduce the soil's swell pressure and increase soil strength.

Laterite soil is available in a tropical area like Malaysia, categorized as alumina-silicate, and are widely utilized in civil engineering works as construction materials for highway roads, houses, a landfill for foundations or dams. The abundance of laterite contributes to the general economy due to the low cost and convenience (AN Zainuddin et al, 2019). Studies in some lateritic soils show that they have a porous granular structure consisting of iron impregnated clayey material in minute spherical aggregation (Hamilton, 1964). However, according to Gidigas (2012), the substitute of Al_{3+} by Fe_{2+} or Fe_{3+} in laterite can undergo chemical alteration to enhance the laterite strength. Abdullah, Shahin & Walske (2020) reported the addition of a chemical binder to the soil develops artificial cementation bonds between the soil particles. This current study investigates the process of synthesizing from by-product materials, bottom ash that is rich in silica and aluminum in the development of inorganic alumina-silicate polymer, called geopolymers.

Geopolymer introduced by Davidovits (1988) is an inorganic polymer synthesized by triggering an aluminosilicate source with hydroxide alkaline solution. To produce geopolymer, binder source material with high silica-alumina (slag, fly ash, silica fume) are dissolved by alkaline solution (sodium hydroxide (NaOH) or potassium hydroxide (KOH) with sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3) formed during the polymerization process (Zhang, Guo, El-Korchi, Zhang & Tao, 2013; Davidovits, 2015; Kuo, Liu, Juang, 2019; Xie, Zhao, Wang, Wang, & Huang, 2019). Alkali solution is used to activate the precursor by dissolving them into $Si(OH)_4$ and $Al(OH)_4$ monomer (Weng, Luqian & Sagoe, 2007). Sodium hydroxide (NaOH) is used to induce the reaction of aluminum and silicate by adding Na^+ ion. Na^+ ion is only used during the polymerization (Saloma, Elysandi & Meykan, 2017).

Geopolymers have been reported to exhibit excellent adhesion to solid particles and low shrinkage potential (Wallah, 2019), and high resistance to acids where they can be used effectively in soil enhancement (Zhang, Provis, Reid & Wang, 2014). In the concrete application, geopolymer is preferable due to their characteristics of being lightweight, have low energy consumption, and replacing ordinary portland cement (OPC) material because of the high CO_2 emissions emitted into the atmosphere (Davidovits, 1994).

Zarina, Kamarudin, & Razak (2015) investigate geopolymer bottom ash-based prepared with different S/L ratios (0.5, 1.0, 1.25, 1.5, and 1.75) and with alkaline solution of sodium hydroxide and sodium silicate ratios (0.5, 1.0, 1.5, 2.0, 2.5, and 3.0). According to the strength test, maximum compressive strength of 11.9 MPa was obtained at a S/L ratio and $Na_2SiO_3/NaOH$ ratio of 1.5 and 2.5 at seven days of testing. Meanwhile, palm oil boiler ash with curing temperature at 80 °C led to maximum compressive strength of 11.5 MPa at a 24 h curing period. The study proves that the geopolymerization of bottom ash as source material positively improves the strength. The morphology using SEM of the geopolymer samples showed changes in the matrix when the curing temperature was increased.

In this investigation, an attempt has been made to use industrial waste admixtures of a boiler ash-based geopolymer for soil stabilization, with particular attention to laterite soils. This study specifically discusses the effectiveness of compressive strength and morphology structure of different percentages of boiler-ash geopolymer in laterite soil for possible future trends of this technology for soil improvement.

2. Material & Methods

2.1 Materials

The laterite soil as raw material in this study was collected from Pasir Gudang, Johor, Malaysia (1° 27' 59.99" N, 104° 00' 60.00" E). The laterite soil was collected at a depth of 1 m -1.5 m. Sieve analysis test for laterite soil in this study consisted of about 25.98% of gravel, 35.55% of sand, and 38.47% of fine size grain and classified under very silty SAND. The laterite soil has a specific gravity of 2.62.

Rigaku NEX CG (XRF) spectrometer analyses were used to determine the chemical compositions of the material study. The XRF is working on the principle of absorbing Florence by detectors its proportion conductance is a change in the energy of the Florence which is processed by the electronics (Faten, Amara & Najeba, 2017). All measurements were carried out under vacuum using a Rigaku NEX CG with RX9, Mo, and Cu targets. These samples irradiated with X-Rays; the intensity identified as a concentration of the element in a sample can be calculated over energy range 1-14 keV. The XRF measurements indicated that all the materials contained oxygen (O), sulfur (S), aluminum (Al), potassium (K), copper (Cu), iron (Fe), zinc (Zn), calcium (Ca), and silicon (Si). The chemical composition of laterite soil tabulated in Table 1.

Table 1 - Oxide compositions of laterite soil in XRF

| Oxides | Composition (%) |
|--------------------------------|-----------------|
| SiO ₂ | 56.0 |
| Al ₂ O ₃ | 30.3 |
| Fe ₂ O ₃ | 9.54 |
| K ₂ O | 0.290 |
| CaO | 0.381 |

The boiler ash was applied as the geopolymer source material

1 in this study. It was obtained from Palm Mill Teluk Sengat, Kuala Tinggi, Johor, Malaysia. The chemical composition of boiler ash is mainly composed of a large percentage of silica as tabulated in Table 2. The size of the boiler ash was ground to obtain its powder of sieve size at 150 μ m. Its specific gravity was 2.35.

Table 2 - Oxide compositions of boiler ash in XRF

| Oxides | Composition (%) |
|--------------------------------|-----------------|
| SiO ₂ | 43.9 |
| Al ₂ O ₃ | 4.55 |
| Fe ₂ O ₃ | 4.11 |
| K ₂ O | 12.5 |
| CaO | 13.1 |
| MgO | 3.27 |
| P ₂ O ₅ | 2.45 |

Sodium Hydroxide (NaOH) and sodium silicate (Na₂SiO₃) act as activator solution in geopolymerization. The NaOH is pallet form of 97% to 98% purity dissolved with distilled water at five molar (Thiha, 2010) and cooled to room temperature. Sodium silicate (Na₂SiO₃) taken from chemical suppliers in Johor was a liquid solution that consists of 9.4% Na₂O, 30% SiO₂, and 60.5% H₂O. The sodium silicate solution added to NaOH at a ratio of 0.5 was allowed to cool and equilibrate for 3 hours before use. In geotechnical testing related to chemical solutions treat, deionized water used in sample preparation due to reaction of atom in water to chemical (Saeed, Khitam, Eisazadeh & Khairul, 2012).

2.2 Specimen Preparation and Mix Proportion

Laterite soil was oven dried at 150°C for 24 hours and later sieved at 200 μ m using standard sieving method (BS 1377: Part 2:1990). NaOH pellet and deionized water first prepared in a volumetric flask to obtain a concentration of 5 M and allowed to be cool down to room temperature. To obtain a homogeneous solution, the ration of the alkaline solution is essential. To the prepared alkaline solution, NaOH solution mixed with Na₂SiO₃ with a ratio of 0.5, and placed in room temperature for 24hours before use. Bottom ash was sieved to 150 μ m to get small particle to activate the silica content when mixed with alkaline activator. In producing geopolymer binder, 80% content of bottom ash was mixed with 20% ready alkaline solution and stirred well for 10 minutes by using a mechanical mixer. The fresh geopolymer was stored in zipper bag, as shown in Fig. 1 for at least three days to activate the geopolymerization before testing. Zipper bag was used to prevent the water evaporation during the geopolymerization process.

**Fig. 1 - Sample at different percentage of geopolymer**

Five different percentages of geopolymer were used to determine the optimum content of geopolymer in enhancing the laterite soil. Based on previous research data in soil stabilization, the optimum percentages of additives (lime, fly ash, kaolin, rice husk) added to laterite soil are between 5% to 30% (Behak, 2017; Zhang & Cao, 2002; Azhar, Fazlina & Nizam, 2017). Dry laterite soil was homogeneously mixed with geopolymer paste at 0%, 5%, 10%, 15% and 20% of geopolymers in the determination of laterite strength. The soil description of the sample in this study is as tabulated in Table 3.

Table 3 - Sample description

| No | Sample Number | Soil Description |
|----|---------------|----------------------------------|
| 1 | LG0 | Laterite Soil + 0% geopolymer |
| 2 | LG5.0 | Laterite Soil + 5.0% geopolymer |
| 3 | LG10.0 | Laterite Soil + 10.0% geopolymer |
| 4 | LG15.0 | Laterite Soil + 15.0% geopolymer |
| 5 | LG20.0 | Laterite Soil + 20.0% geopolymer |

2.3 Test Procedure

Unconfined compression strength (UCT) engineering tests and Scanning Elektron Machine (SEM) were performed to investigate the reaction of soil strength to geopolymer. Fig. 2 shows the study flow chart in this study.

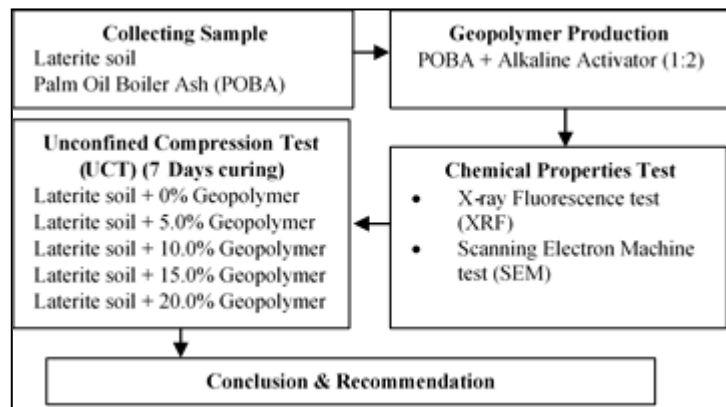


Fig. 2 - Flow chart of study

The aims of unconfined compressive strength (UCT) are to measure the strength of laterite soil at different geopolymer percentages. All specimens were compressed and taken out after curing process for seven days and placed in room temperature 2 hours before UCT was conducted. All samples were tested using ASTM D2166 method as indicated in Fig. 3 and was conducted at Advance Soil Laboratory, Faculty of Civil Engineering, UiTM Shah Alam.



Fig. 3 - Unconfined compressive strength machine

The morphology analysis of specimens was conducted by using scanning electron microscopy (SEM) at the Faculty of Chemical Engineering, UiTM Johor Kampus Pasir Gudang. Characteristic morphological test was conducted to investigate the soil structure and formation at various geopolymer percentages performed. Fig 4 (a) and (b) show the preparation of SEM sample and SEM machine in conducting the SEM analysis. The specimens were placed with gold-coated and analyzed under 10000x magnifications.



Fig. 4 - (a) SEM Machine; (b) SEM Sample Preparation

3. Results & Discussion

3.1 Compressive Strength

The use of the unconfined compressive strength test for soil stabilizers is a quick and simple test and provides a convenient basis for comparison between stabilizer types. The UCT test performed according to ASTM D2166 standard with samples size of 50 mm × 100 mm moulded at its maximum dry density and OMC of each soil composition. To obtain the average maximum strength value of samples, the experiment was repeated thrice for each test sample. The prepared UCT samples were cured for seven days before conducting the test to activate the geopolymerization and sealed in a plastic bag where the temperature is maintained at $20 \pm 2^\circ\text{C}$ (Jingping, Yingliang, Jun & Xiaogang (2019).



Fig. 5 - (a) Sample before UCT; (b) Sample after UCT

According to Wu & Zhou (2012), pressure increases by increasing strain and reaches a peak at a particular strain. The top or maximum pressure is higher for the sample with higher cement content in the mixture. The strain at maximum pressure decreases by increasing cement content. In this study, the unconfined compression tests were conducted on a strain-controlled triaxial testing frame at a strain rate of 1%/min without application of the cell pressure ($r^3 = \text{zero}$). The maximum load was converted to the unconfined compression strength of the sample.

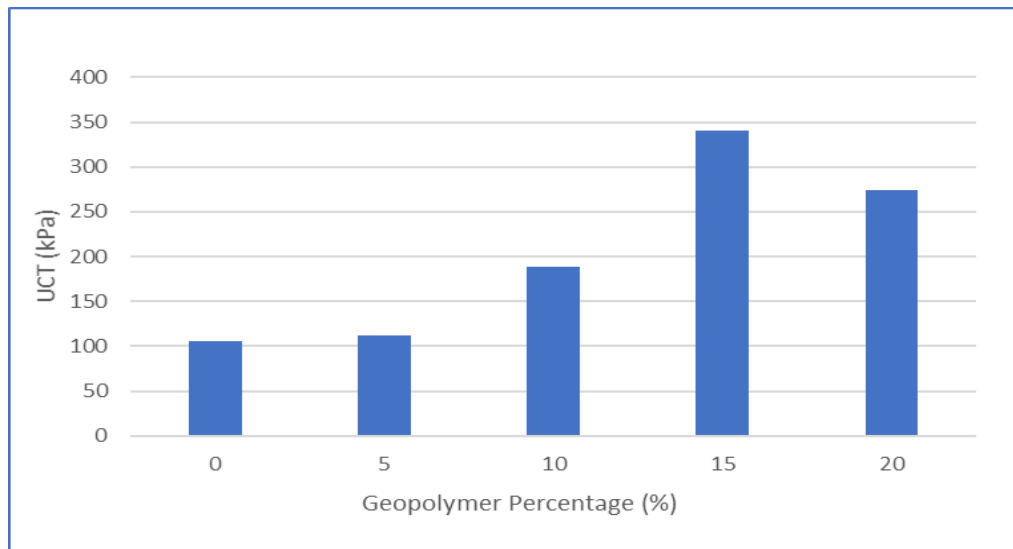


Fig. 6 - Shear strength of laterite soil with different percentage

The results of the test shown in Fig. 6 indicates that unconfined compressive stress values of 0 per cent geopolymer found to be lower in the laterite soil at 106 kPa whereas in the laterite soil at 5 per cent, geopolymer stress marginally increased to 112 kPa. According to Vafaei & Allahverdi (2017) relatively low alumina content (1%–1.3%) may be a source for the low performance at an earlier stage. Soil compressive strength which used 10% and 15% of geopolymer added to laterite soil increased value of 189kPa and 340kPa drastically. The reaction proves geopolymer binder in laterite soil increased the soil strength. The reaction of silica-alumina in laterite soil form new strong bonding and enhance the soil strength. This is supported by Xu & Gong (2014) who stated that the enhancement of fly ash by addition of geopolymer binder can be explained by enhancing silica-to-alumina and silica-to-calcium ratios in the treated combination. The latter was probably due to the increase in the positive surcharge and the subsequent repulsion of soil particles inside the mixture (Eisazadeh 2010; Tingle and Santoni 2003; Katz et al. 2001; Rauch et al. 2002; Tingle et al. 2007).

Meanwhile, 20% of geopolymer added to laterite soil has decreased the value of UCT to 274kPa. It could be noted that the rate of strength increment was somewhat limited at 20%. Excessed water in the 20% of geopolymer binder decreased the bonding of silica and alumina. As a result, the laterite soil decreased its compressive strength. Based on all the percentage, the strength of laterite soil increased significantly, and 15% of geopolymer provides 340kPa and was selected as the best value for laterite soil.

3.2 Microstructure of Specimen

Fig. 7 to Fig. 11 indicates microscope analysis SEM for laterite soil at 0%, 5%, 10%, 15% and 20% of geopolymer accordingly.

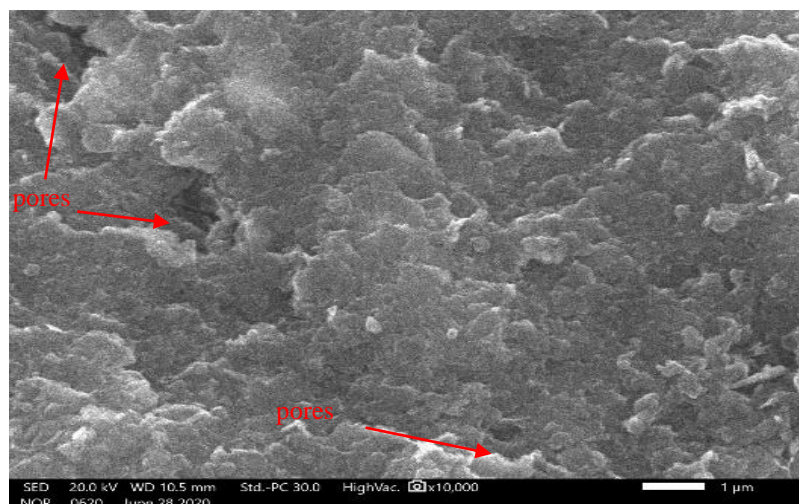


Fig. 7 - SEM image of LS + 0% geopolymer

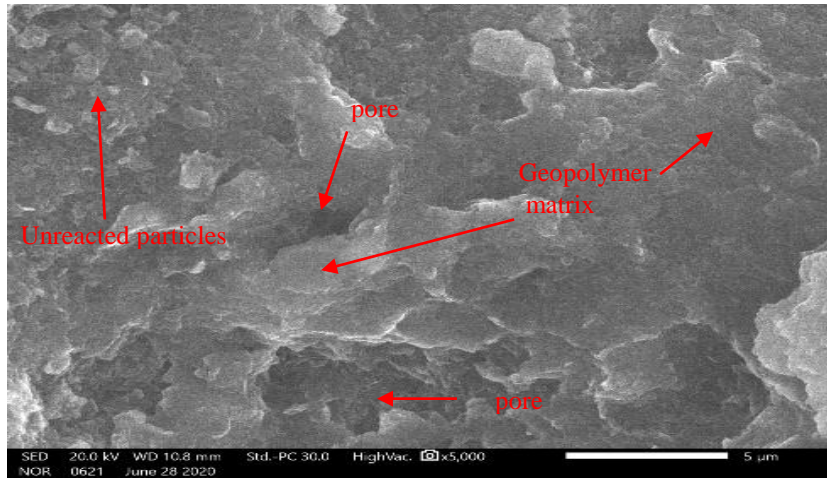


Fig. 8 - SEM image of LS + 5% geopolymer

The SEM analysis in Fig. 7 and Fig.8 show laterite soil with 0 per cent and 5.0 per cent of geopolymer accordingly. The SEM structure characteristic of original laterite soil in Fig 7 shows some angular and sharp-edged grain with some spherical (fine particles) attached to the surface of larger grains. The particles have larger voids and pores due to the presence of silt and clay particles. As a contrast, Fig 8 at 5.0 per cent geopolymer added to the soil noted to have partially polymerized boiler ash in the matrix and have dense surface. An unfinished chemical reaction in the soil produces pores less than five µm and several unreacted bottom ash particles noted on the surface. According to Jaarsveld & Van Deventer (2003), the dissolution of the source material not reacting on surface reactions are in certain instances responsible for forming the geopolymerstructure of the unreleased particles bonding. This is supported by Duan, Yan & Zhou (2017) who mentioned that unreacted particles during geopolymerization, could lead to a poorer microstructure with increased porosity.

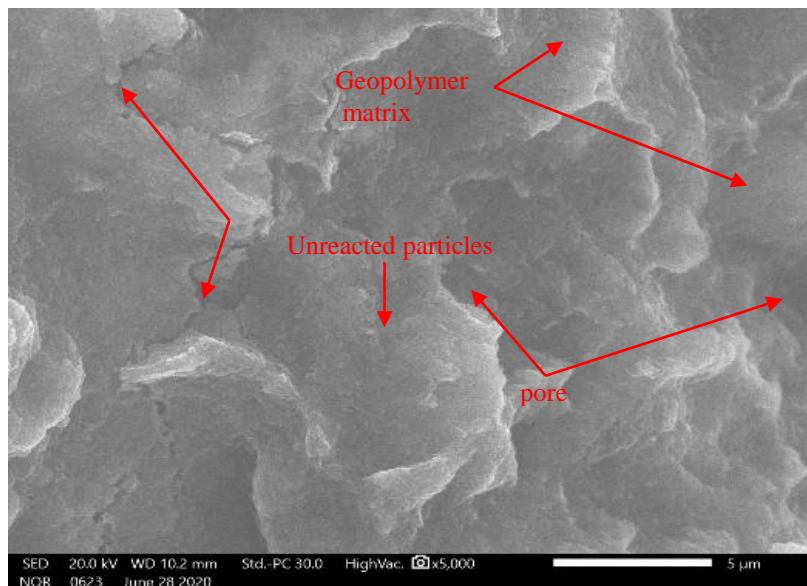


Fig. 9 - SEM image of LS + 10% geopolymer

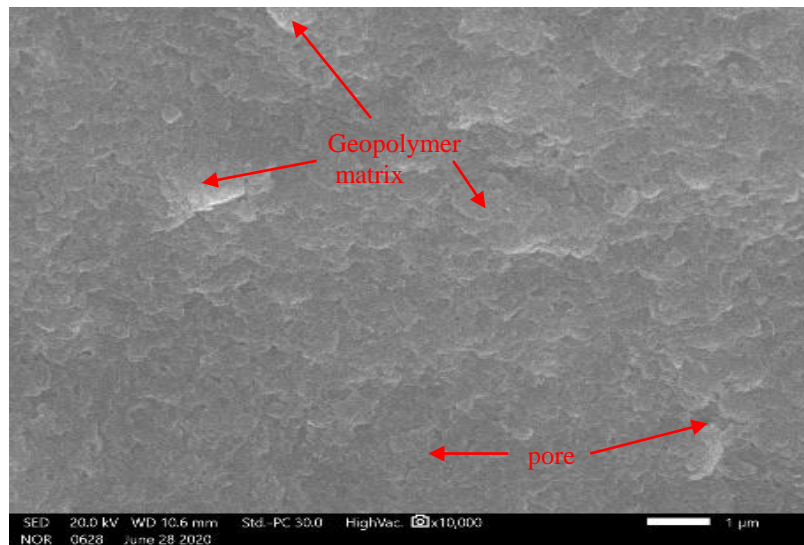


Fig. 10 - SEM image of LS + 15% geopolymer

Fig. 9 demonstrates a more homogeneous structure compared to Fig. 8 and more dense surface. The SEM image shows that the crystalline phases are smooth and well-formed, but there are still unreacted ash particles in the structure and microcrack caused by a chemical reaction on a matrix mixture. A chemical reaction is causing hydration that causes the matrix paste to break. Laterite soil with 15% of geopolymer as shown in Fig.10, indicates that there is pore formation with the size of 1 µm and below. The microstructure shows dense matrix than previous figures and tends to be well compacted and cemented, leading to smaller morphology voids and pores. Pores between particles are filled with gel formed. The increase in geopolymer content shows a less porous and more homogeneous structure by the rearrangement of particles due to the geopolymerization process that occurs between soil particles. The surface comprised of particles with irregular shapes in different sizes and rough textures which gives high interlocking particles. Study indicates that the boiler ash reacted homogeneously with the alkaline activator, and they closed the existing pore. Jaarsveld & Van Deventer (2003) stated that denser microstructures could be gained by adding more soluble silicate into original solutions.

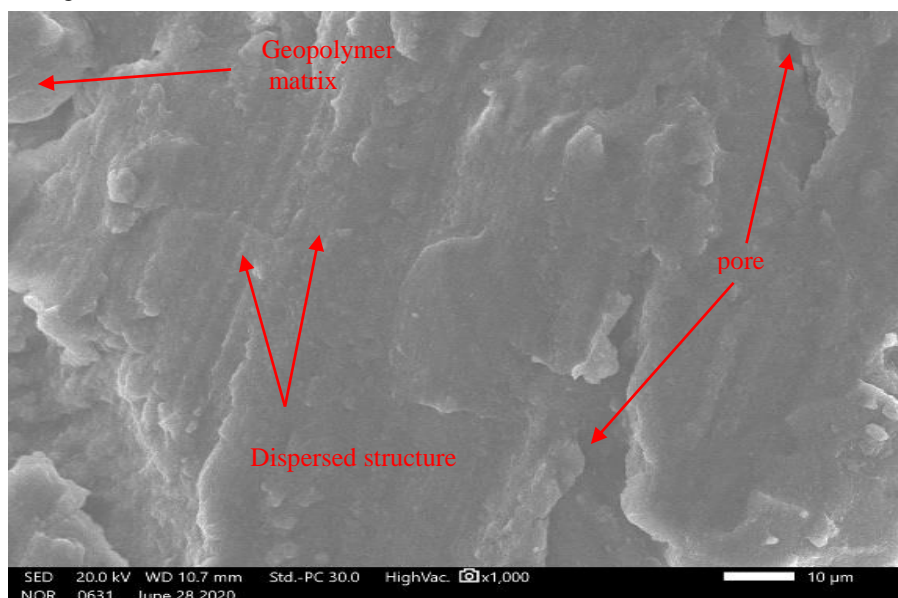


Fig. 11 - SEM image of LS + 20% geopolymer

The SEM image in Fig 11 indicates that the alkali activator solution stimulated the stabilizers due to internal chemical changes occurring with the particles. The microstructure shows dispersed fabric structure, and the size of pores are no more than ten µm, which happens because of the weakness of bond on geopolymer mortar structure. Based on the compressive strength of this sample, the sample indicates low compressive strength due to excessive moisture content which breaks the silica-alumina bond. Moreover, according to Indiramma & Sudharani (2018), soil with flaky shape is likely to have low strength due to low interlocking soil particles.

4. Conclusion

The purpose of this paper is to analyze the effects of various percentages in laterite soil of palm oil boiler ash-based geopolymer. Based on laboratory data and analysis of the result, it can be concluded that:

- i. A higher reaction of Si–Al in the geopolymerization process produced aluminosilicate. It is concluded from the UCS test that the optimum proportion of ash is 15% which gives 340kPa. The UCS value is less at 0% and 5% at 106kPa and 112kPa. When mixed with soil, fly-ash-based geopolymer creates an artificial bonding, along with the interface or contact between soil particles, which increases the integrity and stability of the soil.
- ii. Laterite soil with a geopolymer of 20 per cent shows a drop in the compressive test value because of water proximity. The image of morphology shows that a flaky shape is likely to have low strength due to low interlocking soil particles.
- iii. The SEM disclosed particles in varying sizes with irregular shapes and rough textures in laterite soil of 15% of the geopolymer, which yields strongly interlocking particles.
- iv. The increased compressive strength was attributable to the structure of the geopolymer samples which had a dense, compact matrix and contained less unreacted raw materials which are due to the formation of new crystalline phases C-A-S-H gel, which in turn enhances the strengths.

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