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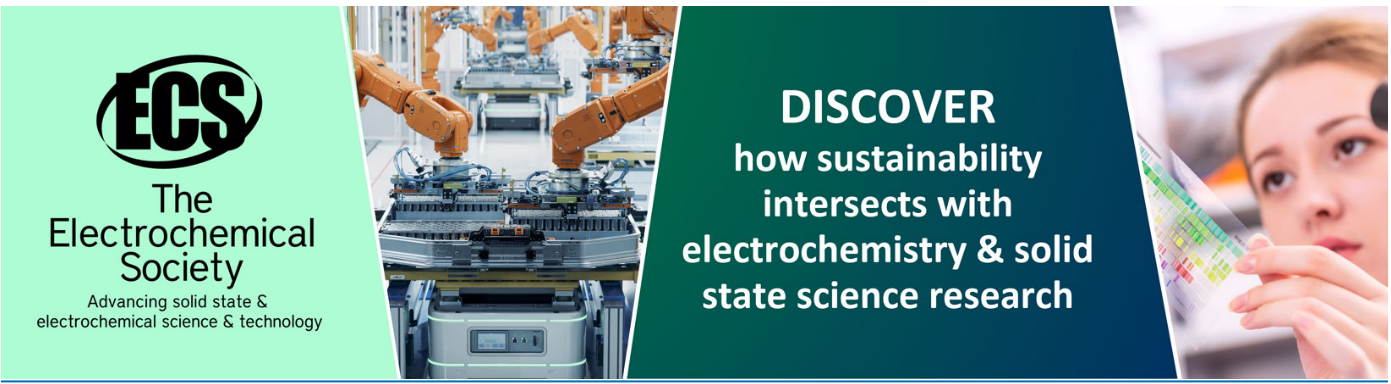
To cite this article: Syafiadi Rizki Abdila *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **864** 012013



IOP Conf. Series: Materials Science and Engineering **864** (2020) 012013 doi:10.1088/1757-899X/864/1/012013

Characterization of Fly ash and Ground Granulated Blast Slag for Soil Stabilization Application Using Geopolymerization Method

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Abstract. Clay soils provide several challenges for geotechnical and civil engineers. This type of soils has a low strength, high plasticity and can cause damage to the road pavement such as crack and soil strength reduction. Thus, require stabilization method. Continue of research and investigations have been done to find other alternative in soil stabilization that eco-friendly. Geopolymer, one of the alterative eco-friendly soil stabilization method offering small swelling potential and outstanding adhesion to soil properties, which could be an effective soil stabilizer. Geopolymer is a reaction that chemically integrates minerals that involves naturally occurring silicoaluminates sources. The geopolymer synthetized from soil, fly ash, ground granulated blast slag and an alkaline solution made from sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). The characterization testing includes physical properties, X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) to examine the physical properties, elemental chemical composition, mineralogical properties, microstructure, and bonding chemical of the raw material, respectively. Based on the characterization result, the soil, fly ash and ground granulated blast slag consists mainly of silica (SiO_2) and alumina (Al_2O_3) which make it suitable to be used as raw materials for geopolymer formation. This paper presents a characterization analysis of soil, fly ash and ground granulated blast slag as raw materials for soil stabilization application using geopolymerization method.

1. Introduction

In parts of Asia region especially in Malaysia and Indonesia, the development of road construction, building foundation, and residential properties have encroached into the areas with soft soils conditions. The soft soil was also called as soft clay, where soft clay was typically flake shaped particle and consisted of clay minerals and other minerals [1]. Clay soil is easy to swell in wet condition and will shrink if the soil is dry in the dry season. Swelling and shrinkage happens because water content in the soil change the volume of soil [2]. The characteristic of clay soft soil is high compressibility, low shear

IOP Conf. Series: Materials Science and Engineering **864** (2020) 012013 doi:10.1088/1757-899X/864/1/012013

strength, low permeability, low strength and high plasticity [3]. Furthermore, soft soil can cause damage to the road pavement such as crack, settlement to the road pavement, and reduction of soil strength [4].

Stabilization of soil is required in order to prevent the problem. Generally, soil stabilization is a process to improve and stabilize the physical and mechanical properties of soil by changing at least one of the soil characteristics [5]. The most common soil stabilization method is by replacing the soil with a stronger material such as crushed rock, but higher cost was involved [6]. Practical and sustainable alternative is always been searched in civil engineering industry. Geopolymers offered small swelling potential and outstanding adhesion to soil properties, which could be an effective soil stabilizer [1].

Geopolymer is a binder produced by combining aluminosilicate source materials with a strong alkali solution [7-13]. Source materials, which are rich in silica (Si), and alumina (Al) minerals are highly recommended and possible to be used as the main precursor such as fly ash and ground granulated blast slag (GGBS) for geopolymerization in soil stabilization application [14-15]. Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) are widely used as alkali activator solutions [16-20]. Thus, this paper aims to present the characterization of soil, fly ash and GGBS as potential raw material for soil stabilization based on geopolymer process.

2. Experimental Method

2.1. Material

2.1.1 Soil
Soil used in the study from Kampung Kok Klang, Kangar, Perlis located at the coordinates 6°28'54.8"N 100°17'50.9"E. the soil collected from a depth of 30cm the natural ground level. the soil was dried and pulverized to perform the various experimental testing for the study. According Unified soil classification system, the soil was classified as clay soil with high plasticity (CH).

2.1.2 Fly ash
Fly ash is an industrial by product from coal combustion. These micron-sized earth elements consist primarily of silica, alumina, and iron. According ASTM C 618, Fly ash can be classified to two classes based on the present of calcium content. Class C fly ash usually has calcium percentage above 20% while class F fly ash, usually has calcium percentage, which is not higher than 10%. The fly ash used in the study was from type class C and collected from coal combustion plant in Manjung Power Station, Telok Rubiah, Lumut, Perak, Malaysia.

2.1.3 Ground Granulated Blast Slag
Ground granulated blast slag (GGBS) is an industrial by product from iron combustion. It mainly consists of lime, alumina, and silicate. The Ground granulated blast slag used in the study was supplied from YTL Cement Marketing Sdn Bhd, Jalan Bukit Bintang, Kuala Lumpur, Malaysia.

2.2. Characterization Method

2.2.1. Particle Size Distribution
This method covers the quantitative determination of the distribution of particle size in soil, fly ash and GGBS. The sieving test was done following the ASTM D 422 (Test sieves, technical requirements and testing). The size of sieves used were 4.75 mm, 2.36 mm, 1.18 mm, 600 µm, 300 µm, 150 µm, and 75 µm.

2.2.2. Atterberg Limits
Atterberg Limits testing included liquid limit and plastic limits. The liquid limits of soil, fly ash and GGBS was measured complying to the BS 1377-2: 1990 and the apparatus used is NL 5003 X / 002

Digital Cone Penetrometer. The soil, fly ash, and GGBS sample weighs at least 300 g that passes the 425-µm sieve test. Meanwhile, for plastic limit of soil, fly ash and GGBS was measured complying to the BS 1377-2: 1990. The soil, fly ash, and GGBS sample weighs at least 300 g that passes the 425-µm sieve test.

2.2.3. X-ray fluorescence (XRF)
The chemical composition of the soil, fly ash and GGBS were determined by using X- ray fluorescence specnometer (XRF) with a brand-named PAN analytic PW4030. In this analysis, the soil, fly ash and GGBS samples (in powder form) which passed the 75-µm sieve was used.

2.2.4. X-ray diffraction (XRD)
The phase and components of soil, fly ash and GGBS were conducted by using XR Diffractometer Shimadzu XRD-6000. Sample were prepared in powder form. XRD analysis was performed using with Cu-Kα radiation with X-ray tube operating at 40kV and 35mA. The XRD data were collected at 2θ values in the range of 10°to 80° at scan rate 2° per minutes and scan steps of 0.02° (2θ). The auto search match software, High Score Plus was used to analyze the diffraction data.

2.2.5. Fourier transform infrared spectroscopy (FTIR)
Perkin Elmer FTIR Spectrum spectrometer was used to identify the functional group of soil, fly ash, and GGBS. The samples were prepared in powder form. Sample were analyzed using the attenuated total reflectance (ATR) technique with scanning range was 600 cm^{-1} to 4000 cm^{-1}.

2.2.6. Scanning electron microscope (SEM)
The morphological characterization of soil, fly ash, and GGBS were carried out using JSM-6460LA model scanning Electron Microscope (JEOL) utilizing the secondary electron detectors. The soil, fly ash and GGBS sample was prepared in powder form.

3. Results and Discussion

3.1. Physical properties analysis
Table 1 shows the particle size distribution and of soil, fly ash, and ground granulated blast slag. Ground granulated blast slag indicate the highest content of fine-grained with 96.00% followed by fly ash and soil with 94.00% and 52.00%. Meanwhile, the highest percentage of course-grained content was recorded by soil with 48.00%, fly ash with 6.00% and the lowest was GGBS with 4.00%. The soil indicated the percentage of fine-grained particles exceeded 50% were classified as fine-grained soils and according unified soil classification system, soil was classified as clay soil with high plasticity (CH). The similar result of soil has been reported by Rama Indera K et al., (2018) also for particle size distribution of soil. For fly ash and GGBS particles, the percentage of fine-grained particles also exceeded 50 %, in which based ASTM D2487 also classified as fine-grained particle. The fine-grained particle of the fly ash and GGBS contributes to fill the large void surface area between clay soil particle and control water content of the clay soils. This can cause clay soil become stable, compact and increase the compressive strength of the clay soil [21].

The results of liquid limit of soil, fly ash and ground granulated blast slag recorder at 51,20 %, 23.40 % and 40.73 %, respectively. For plasticity index, the percentage of soil was record with 28.48%. Whereas, for fly ash and ground granulated blast slag do not have a plastic limit value. In this condition the term 'non plastic' meaning this material have low cohesive value. Material that has a low cohesive value lead to reducing plasticity index and control swelling behavior for clay soil. Sarathi Parhi reported the addition of alkali activated GGBS/fly ash to clay soil can reduces liquid limit and index plasticity due to the exchanged ions and the process of agglomeration and flocculation of the soil particles [22].

Table 1. Particle size distribution and Atterberg limits of soil, fly ash and GGBS.

No.	Parameter	Unit	Soil	Fly ash	GGBS
1.	Particle Size Distribution:				
	➢ Fine - grained (> 50% Passes No.200 Sieve).	%	52.00	94.00	96.00
	➢ Course - grained (> 50% Retained on No.200 Sieve).	%	48.00	6.00	4.00
2.	Atterberg Limit:				
	➢ Liquid Limit	%	51.20	23.40	40.73
	➢ Plastic Limit	%	28.48	Non plastic	Non plastic
	➢ Index Plasticity	%	22.72	Non plastic	Non plastic

3.2. Chemical composition analysis

The major chemical composition in the soil, fly ash, and GGBS are given in the table 2. Based on the chemical compositions of the clay soil, the silica oxide (SiO_2) and aluminium oxide ($A_{l2}O_3$) showed the most major oxides of clay soil which is most of the geopolymer source materials shows rich in silica oxide (SiO_2) and aluminium oxide (Al_2O_3), where more than 90 % was found. Other compounds also can be found in clay soil such as iron (III) oxide (Fe_2O_3). In order to allow the geopolymerization process to be happened, the primary requirement had to be fulfilled where the materials used must be rich silica (Si) and alumina (Al) minerals [23-25]. The range of Al_2O_3 and SiO_2 of soil were in between 17.00 % to 73.30 %. The presence of quartz has been proven can contribute reasonable amount of silicon to the formation of Si-O-Si bond in the geopolymer, lead to a higher compressive strength [1]. Previous researchinvestigate potential of soil as geopolymer material, where total percentage of Al_2O_3 and SiO_2 was 63.60%.

The chemical composition of fly ash which consist mainly of alumina (Al_2O_3) and silica (SiO_2), which make fly ash suitable to be raw materials for geopolymer formation. The mineral Al and Si are important in the reaction to form alkali activated composites especially in the production of the alkali activated strength. Fly ash also contained traces of other compounds such as iron (III) oxide (Fe2O3), calcium oxide (CaO), magnesium oxide (MgO). According to ASTM C618, the fly ash used in this study has mineral oxide of total sum less to 70% which is classified into Class C and is considered as a pozzolan or self-cementitious material. The high content of SiO_2 were found in fly ash and the ratio of SiO_2 to Al_2O_3 is 2.3 which is higher than the suggested ratio in producing cement binder. This proves that this fly ash is able to form alkali activated and can be used in soil stabilization application.

For the chemical composition of ground granulated blast slag (GGBS), the main constituent of GGBS was SiO_2 and Al_2O_3 which were 40.9 % of the total composition. The GGBS also contained traces of other compounds such as calcium oxide (CaO), and magnesium oxide (MgO). Ground granulated blast slag (GGBS) was favorable as a source of geopolymerization process, which fulfils the fundamental requirements of SiO_2 and Al_2O_3 in order to be activated by alkali solution. There is contradiction of the CaO content in GGBS as geopolymer materials. The existence of calcium (CaO) content in GGBS contributed to the development of compressive strength. The reaction between GGBS and alkali activator solution forms a calcium silicate hydrate (C-S-H) and calcite (CaCO3) within the geopolymer matrix. These hydration products along with aluminosilicate structure in the slag samples contributed to gain high strength significantly.

Table 2. Major chemical composition of soil, fly ash and GGBS.

Compositions	Soil (%)	Fly ash (%)	GGBS (%)
Al_2O_3	17.00	13.30	10.50
SiO_2	73.30	30.70	30.40
Fe_2O_3	6.15	23.92	-
CaO	-	22.40	50.37
MgO	-	3.6	3.2

3.3. Phase analysis

Figure 1 shows the X-ray diffractometer of the soil, fly ash, and GGBS. Figure 1.a) indicate that the mineralogical component of soil is quartz (SiO_2) (ICDD reference: 00-046-1045), kaolinite Al2Si_2O_5 $(OH)_4$ (ICDD reference: 00-029-1488) and hematite (Fe_2O_3) (ICDD reference: 00-024-0072). The clay minerals, kaolinite appear in soil as the liquid limit and plasticity index were high due to the presence of clay minerals was proven. The presence of quartz minerals can contribute reasonable amount of silicon to the formation of Si-O-Si bond in the geopolymer, thus lead to a higher compressive strength [26].

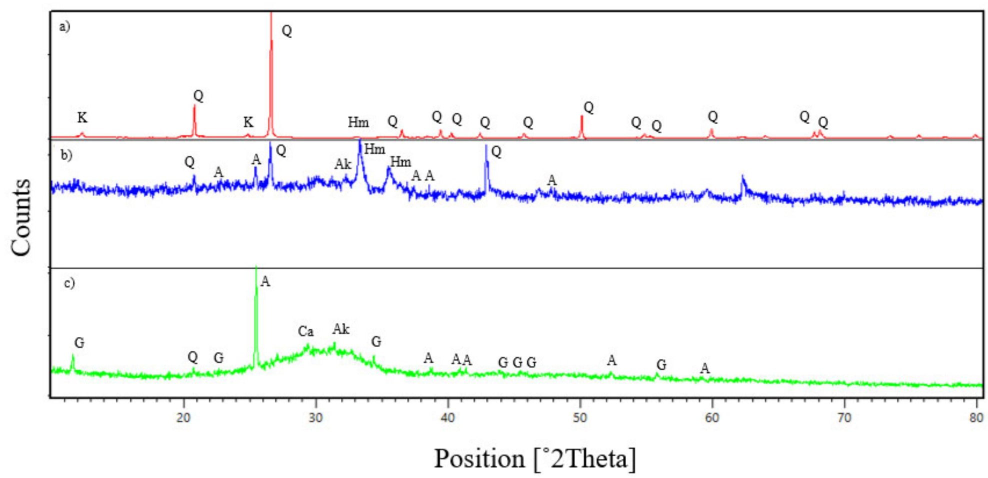

Figure 1. XRD patterns of a) soil, b) fly ash and c) GGBS (Q: Quartz, K: Kaoline Hm: Hematite, G: Gypsum, A: Anhydrite, Ak: Akermanite and Ca: Calcite).

Figure 1.b) indicate that the mineralogical component of fly ash is quartz (SiO_2) (ICDD reference:00-0461-045), hematite (Fe_2O_3) (ICDD reference: 00-024-0072), anhydrite ($CaSO_4$) (ICDD reference: 00-037-1496), and akermanite (Ca_2Mg [Si_2O_7]) (ICDD reference: 00-035-0592). The existence hematite mineral in fly ash lead to the chemical bond by in reaction components geopolymer become stronger. The existence of anhydrite ($CaSO_4$) and akermanite (Ca_2Mg [Si_2O_7]) in fly ash contribute to maintain the volume expansion in clay soils. The volume expansion fills the void surface area of the clay soil efficiently, making the stabilized soil more compact and increase the compressive strength of the clay soil [29]. The presence of quartz (SiO2), mullite ($2Al_2O_3SiO_2$), hematite (Fe_2O_3), and magnetite (Fe_3O_4) in the fly ash XRD pattern. The existence hematite mineral in fly ash lead to stronger chemical bond of geopolymer. Thus, lead to increase on compressive strength [16, 28, 29].

Figure 1.c) indicate the mineralogical component of GGBS which are anhydrite ($CaSO_4$) (ICDD reference: 01-086-2270), gypsum ($CaSO_4 \cdot 2H_2O$) (ICDD reference: 00-037-1496), quartz (SiO_2) (ICDD reference:00-0461-045), calcite ($CaCO_3$) (ICDD reference: 01-089-0387) and akermanite (Ca_2Mg [Si_2O_7]). The presence of the quartz (SiO_2), anhydrite ($CaSO_4$), gypsum ($CaSO_4 \cdot 2H_2O$), calcite

($CaCO_3$), and akermanite (Ca_2Mg [Si_2O_7]) with high intensity are due to the original source of GGBS which is driven from high calcium (Ca), silica (Si) and low magnesium (Mg) content. Another research done investigated the microstructural evolution of alkali activated binder based on GGBS, the XRD pattern GGBS revealed the presence of gypsum ($CaSO_4 \bullet 2H_2O$) and calsite ($CaCO_3$). The presence of mineral gypsum has an effect to increase the strength of soil [30].

3.4. Functional group analysis

The present of Si and Al structure bond of soil, fly ash, and ground granulated blast slag (GGBS) were also confirmed using FTIR analysis as presented in figure 2. The presence aluminosilicate functional group was demonstrated on wavenumber range 800 cm^{-1} to 1100 cm^{-1}. The asymmetric Si-O-T/T-O-Si stretching (T represent either Si or Al) of soil, fly ash and GGBS were illustrated at a wavenumber of 1014.57 cm^{-1}, 958.06 cm^{-1} and 860.88 cm^{-1}. Moreover, spectra peak in the range 676.89 cm^{-1} to 784.11 cm^{-1} is the bending vibration mode of Si-O-T bonds. The presence of peaks in the range 3685.02 cm^{-1} to 3737.20 $cm^{-1}$are assigned to the O-H stretching vibration, which is indicative of moisture in the raw materials. The band around 1600 cm^{-1} are assigned to the Mg-O bonds, indicated to magnesium mineral in the raw materials of fly ash and GGBS. This finding was supported by the X-ray Fluorescence (XRF) analysis, which clearly indicates the presence of MgO (3.2% and 3.6%) in the raw materials of fly ash and GGBS. The band 1490.38 cm^{-1} was identified in the fly ash and GGBS is assigned to symmetric stretching mode of O-C-O bonds of carbonate group subjected to superficial weathering of fly ash and GGBS during storage. In addition, the band at around 1490.38 cm^{-1} was characteristic of CO_3 stretching mode suggested the presence of calcite as a result of the reaction between excess calcium oxide with atmospheric carbon dioxide [31].

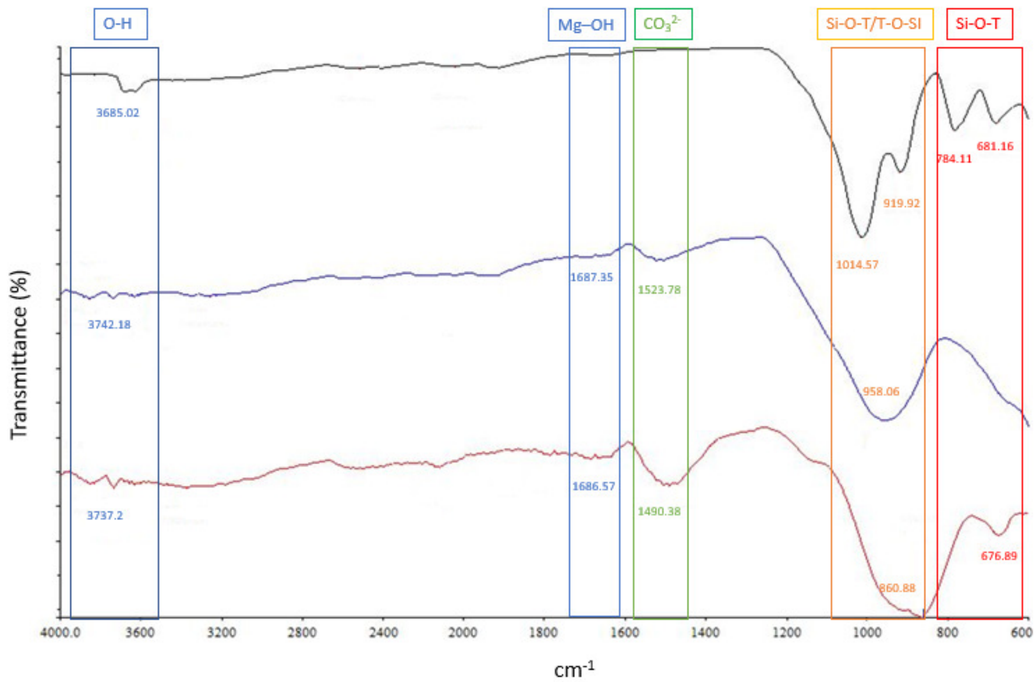

Figure 2. FTIR spectra of (a) soil, (b) fly ash, and (c) GGBS.

3.5. Morphology analysis

The morphology of the soil, fly ash and ground granulated blast slag (GGBS) were analyzed by using scanning electron as can be seen in figure 3**.** Based on figure 3(a), the microstructure of soil consists of number of flaky-like particles. Flaky particles of clay soil provided larger surface area and availability of more contact area with water. The large surface area allows the soil to hold a greater quantity of water, which explained the high values of liquid limit and plasticity index of the clay soil. The existence of several rounded particles with high sphericity in the microstructure indicated the presence of large voids between their particles or loose morphology due to the inexistence of cementing compound that could bind the soil particles together possessed low value of strength [31]. The geopolymerization reaction to soil also can combined the particles of soil thus, led to the change of the morphology from having large voids to a dense appearance [1].

For figure 3(b) shows the microstructure of GGBS. The microstructure of GGBS particles the consist several of irregular-shaped and sharped-edged with rough surface. Moreover, the shape of GGBS particles was influenced by the processing approach such as air mill, ball mill, and vibro mill. SEM micrographs of the ground-granulated blast furnace GGBS shows a massive stone-shaped morphology as well as angular irregular particle texture with particle size ranging from 1230 nm to 2390 nm. During GGBS is active with alkali activators are due to the high rate of hydration reaction formed a desirable early strength that suitable for construction application [32, 33].

Figure 3(c) shows the microstructure of fly ash. The microstructure of fly ash consists of a series of spherical vitreous particles of different sizes, but with a regular smooth texture. These particles are usually hollow, and some spheres may contain other particles of a smaller size in their interiors. Another research reported the geopolymerization reaction of the fly ash to soil make the discrete soil particles appear more closely bound and dense texture in the stabilized material with the void seemingly filled.

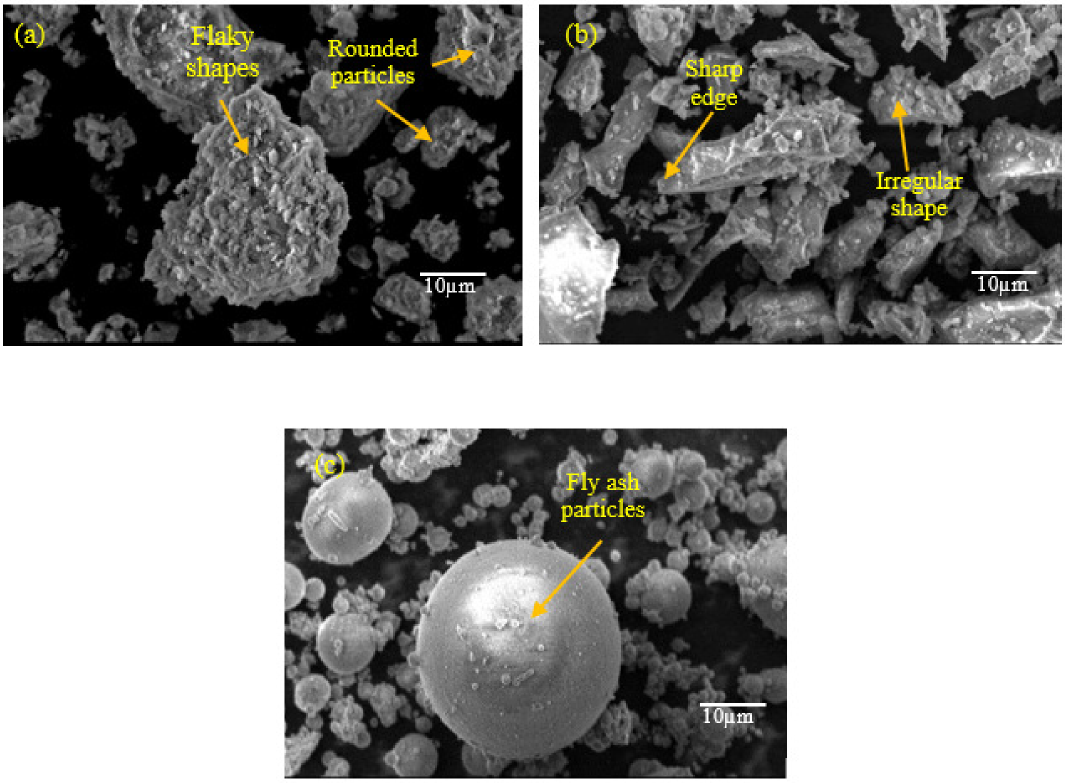

Figure 3. Microstructure of (a) clay soil, (b) GGBS, and (c) fly ash.

4. Conclusions

Based on the result form this study, soil, fly ash and GGBS have potential as materials for soil stabilization by using geopolymerization process. The percentage of fine-grained particles in soil exceeded 50% were classified as fine-grained soil. According to unified soil classification system, soil was classified as clay soil with high plasticity (CH). The composition of Al, Si, and Ca in Soil, fly ash and GGBS make this material to be utilized as alkali activated. Even though soil contain kaolinite, has flaky structure that will cause low reactivity of geopolymer gel, geopolymerization process is still possible and addition of other geopolymer material (Fly ash and GGBS) as reactive filler most likely will fasten the process and leads to higher compressive geopolymer strength.

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