

The Effect of Si/Al Ratio to Compressive Strength and Water Absorption of Ferronickel Slag-based Geopolymer

Mustofa¹ and Sungging Pintowantoro¹

Abstract—Geopolymer material is inorganic polymers that are generally synthesized from aluminosilicate materials, such as ferronickel slag. In this study, synthesis of geopolymer material using NaOH 7 molar and Na₂SiO₃ solutions with different Si/Al ratio to make paste. Paste is molded in a metal mold with dimensions of 5x5x5 cm³, then released from the mold and heated in an oven at 80 °C for 24 hours. The specimens that have been heated further left at room temperature for 28 days and then tested for compressive strength and water absorption. The results indicate a trend compressive strength increases and water absorption decreases with increasing the Si/Al ratio. The optimum condition is obtained when Si/Al ratio is 3.5.

Keywords—ferronickel slag, inorganic polymer, aluminosilicate material.

I. INTRODUCTION

Geopolymer is amorphous to semi-crytalline materials that synthesized from materials generally contain SiO₂ and Al₂O₃ compounds as a source of aluminosilicate. Geopolymers were synthesized by mixing at room temperature or higher (e.g., 90 °C) reactive aluminosilicate powder with alkaline activator containing alkali hydroxide, silicate, aluminate, carbonate or sulfate, or a combination thereof.

Geopolymer materials have several advantages including high initial strength, low shrinkage, freeze-thaw resistance, resistance to sulfates, corrosion, acid, fire resistance, and resistance to alkali aggregate harmless reactions. When compared with the cement, geopolymer productions required operating low temperatures and energy, low CO₂ emissions [1] and the availability of raw materials are still quite a lot. One of the materials that are used as raw materials for the synthesis of geopolymer is ferronickel slag [2] [3] [4]. Ferronickel slag is an industrial by-product generated during smelting to ferronickel used in the manufacture of stainless steel and nickel alloys [5]. Main reason for the selection of ferronickel slag as raw material geopolymer synthesis is it has the high content of silica and alumina components, where those components have an important role in the geopolymerization reaction.

Synthesis of geopolymer typically include (a) pretreatment mechanical of raw material aluminosilicate solid (crushing and grinding), (b) the preparation of the initial liquid silicate alkali phase, (c) mixing of the solid and liquid phases, (d) the moulding paste, (e) curing at low to moderate temperature (40-80 °C) for several hours, and (f) setting in the environment [6]. This study was

conducted to determine the effect of Si/Al ratio on the properties of geopolymer produced include compressive strength and water absorption.

II. METHOD

A. Material

Geopolymer samples were prepared from ferronickel slag that crushed up to size $\leq 70 \mu\text{m}$. Chemical and mineralogical analyses of slag are performed by EDX and XRD analysis, as given in Table 1. Based on Table 1 are known that have a higher silica content than the calcium oxide, magnesium oxide and alumina. Some minerals were detected in a ferronickel slag is augite [(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)₂O₆], forsterite (Mg₂SiO₄), and pyrope (Mg₃Al₂(SiO₄)₃).

B. Synthesis of Geopolymer Material

Synthesis of geopolymer material was prepared through four stages. First, making the activator solution by mixing of NaOH 7 molar and Na₂SiO₃ solutions with ratio is 2.5, and then left it for 24 hours. Second, mixing the activator solution and ferronickel slag powder with a mass ratio of the slag and the activator solution (S/L ratio) was 5.4 while stirring process is carried out to make a paste. Third, paste was molded into metal molds with dimensions of 5x5x5 cm³, and then released from the mold. Finally, the curing process is divided into two stages, namely heating in an oven at 80 °C for 24 hours and curing at room temperature up to 28 days for further hardening.

C. Analysis and Tests

The mineralogical and microstructure studies of the slag-based geopolymers were performed by means of X-Ray Diffractometry (XRD), Fourier-Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) analysis.

The geopolymers properties studied in this work include compressive strength and water absorption. Compressive strength was measured on a universal testing machine of the Concrete and Building Material Laboratories, Department of Civil Engineering using cubic specimens with dimensions 5x5x5 cm³ according to the ASTM C109 standard test. Three specimens of each experiment were tested and the average compressive strength was reported as the result. The water absorption was determined according to the ASTM C1403 standard test.

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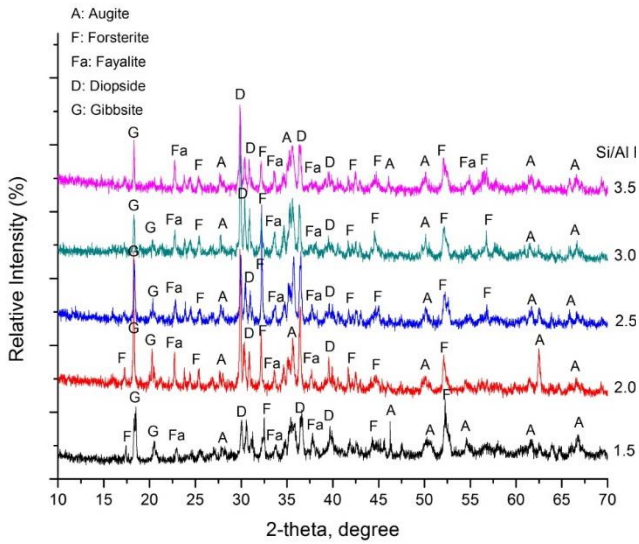


Figure 1. XRD Patterns of Geopolymer

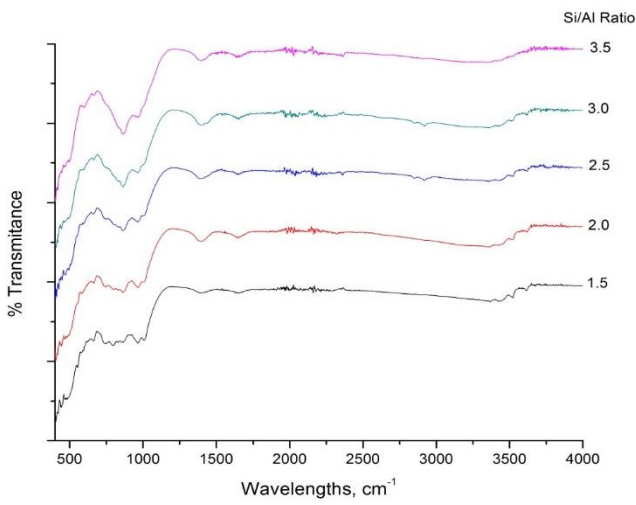


Figure 2. FTIR Spectra of Geopolymer

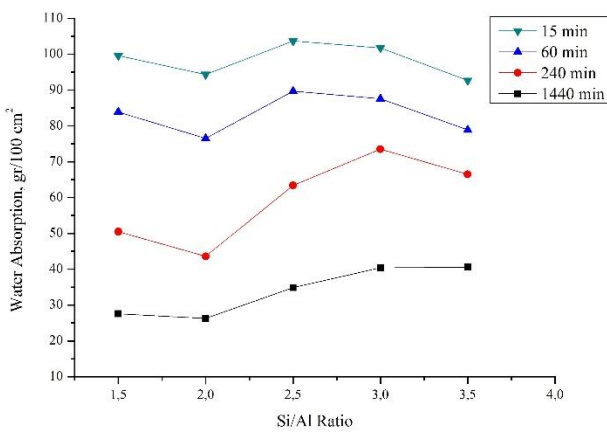


Figure 3. Water Absorption of Geopolymer.

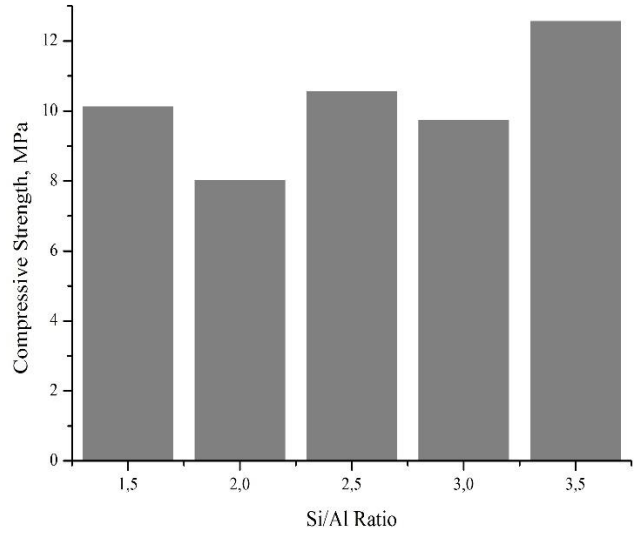


Figure 4. Compressive Strength of Geopolymer

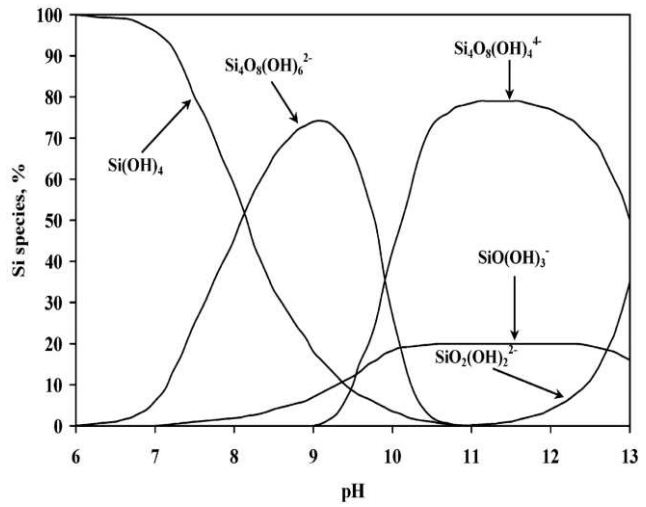


Figure 5. Speciation Diagram of Si in Aqueous Solution [8]

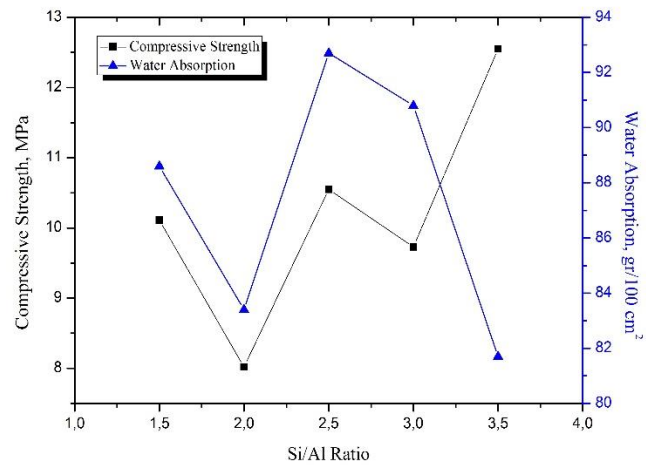


Figure 6. Compressive Strength versus Water Absorption (after immersion for 24 hours) of Geopolymer

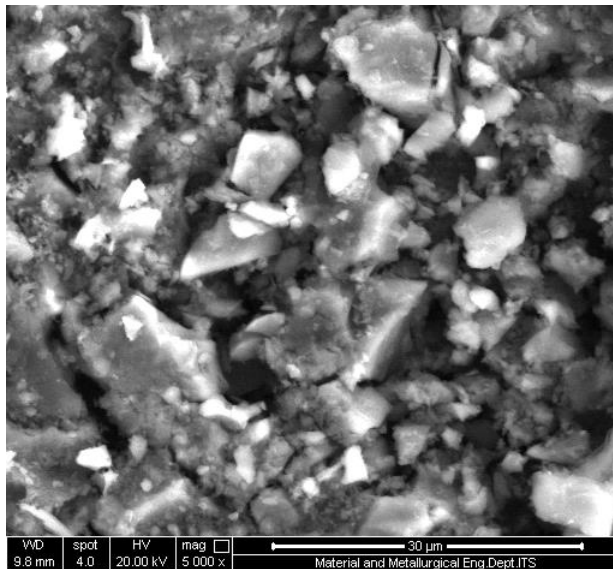


Figure 7. SEM Micrograph of Geopolymer with Si/Al Ratio is 1.5

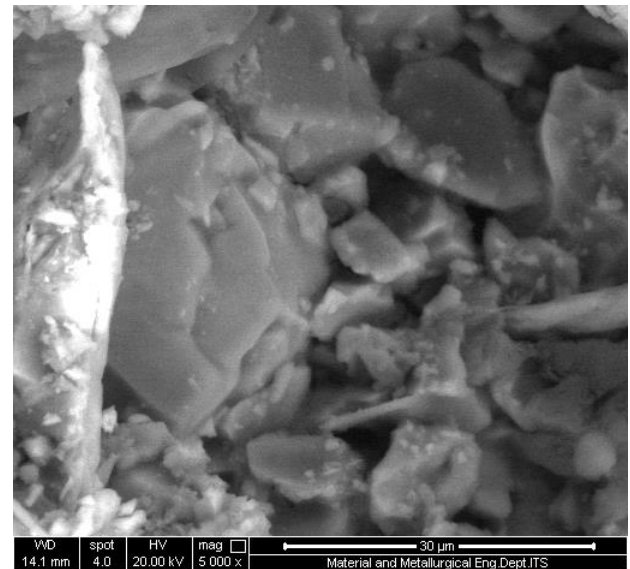


Figure 8. SEM Micrograph of Geopolymer with Si/Al Ratio is 2.5

TABLE 1.
 CHEMICAL COMPOSITIONS OF FERRONICKEL SLAG

No	Compounds	Compositions (% wt)
1	SiO ₂	43.08
2	Al ₂ O ₃	9.13
3	FeO	8.43
4	MgO	18.67
5	CaO	20.69

TABLE 2.
 MINERAL COMPOSITIONS OF FERRONICKEL SLAG-BASED GEOPOLYMER

No	Group	Mineral Name	Formula	Reference Code
1	Pyroxene	Augite	(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al) ₂ O ₆	01-088-0849
		Diopside	CaMgSi ₂ O ₆	01-083-1821
2	Olivine	Forsterite	Mg ₂ SiO ₄	00-001-1290
		Fayalite	Fe ₂ SiO ₄	01-071-1668
3		Gibbsite	Al(OH) ₃	01-074-1775

III. RESULTS AND DISCUSSION

A. Mineralogical and Structure of Geopolymer Material

Mineralogical transformation that occurs during the geopolymerization process of ferronickel slag and minerals contained in each of the geopolymer material can be seen in Figure 1 and Table 2. Generally, the peaks produced has a sharpness decreases with increasing Si/Al ratio, especially at $2\theta = 17.25, 20.42$ and 52.11 . This indicates that the geopolymerization process produce amorphous phase structure of the aluminosilicate.

The new peak is formed for all products geopolymer at $2\theta = 18.34$ and 20.42 is characteristic of gibbsite. This is confirmed by previous studies, where in the alkaline solution, Al(OH)₃ experienced the dissolution to forming aluminate species [7]. The presence of gibbsite at $2\theta = 20.42$ has a peak decreases with increasing Si/Al ratio. This characteristic can be attributed to the formation of geopolymer structure is more complex because of the increased concentration of Si so that more aluminate species is used for the dissolution of silicate. Characteristic peaks at $2\theta < 20^\circ$ is a phenomenon polycondensation that lasted geopolymerization of liquid

phase aluminosilicate as the portion dissolution of the amorphous phase in ferronickel slag.

Results of FTIR analysis can be seen in Figure 2, shows absorption bands at wavelengths between 400-4000 cm^{-1} . The new peaks were generated occurs at wavelengths range 400-500, 700-1100, 1300-1750 and 3300-3700 cm^{-1} . Broadly, the FTIR spectra are divided into two ranges, namely 370-1300 cm^{-1} related to vibration mode of Si-O-Si unit and 1600-4000 cm^{-1} related to the vibration mode of water [6]. This reflects the fact that the range of 1600-4000 cm^{-1} geopolymer product containing free water absorbed from the environment and water molecules bound in the structure of geopolymer network of the crystallization process and the absorption of the reaction product [8] [9] [10]. Peaks at 1300-1600 cm^{-1} related to the absorption band which is owned by carbonate units [11] [12] [13] [14].

The peaks of the range of 400-1200 cm^{-1} indicates the structure of Si-O-X (X = Si, Al) in geopolymer system. At a wavelength around 768-1200 cm^{-1} shows the geopolymerization process [6]. The smaller the ratio of Si/Al leads to the formation of new peaks in the structure of geopolymer. New peak began to look at the ratio of Si/Al = 3, especially at a wavelength around 1009.63 cm^{-1} to more sharply with the smaller ratio of Si/Al. This relates to the asymmetric stretching vibration of Si-O units in SiO_4 tetrahedral structure in the aluminosilicate. asymmetric stretching vibration and shifting tape reveals early dissolution of the solid material into a strong alkaline solution and the establishment of new products during geopolymerization. The new products generated from these events is an amorphous aluminosilicate phase [2] [13]. Absorption band at 664.34 cm^{-1} relating to the incorporation of AlO_4 in the aluminosilicate caused by the formation of a new phase of amorphous zeolites [2].

B. Effect of Si/Al Ratio to Water Absorption

The purpose of the water absorption test is to determine how easily water flows into the geopolymer material. Water absorption is a very important parameter in the manufacture of geopolymer for showing permeability and rate of reaction of the geopolymer. The low water absorption will be low permeability. Generally, high rate of geopolymerization leads to geopolymer matrix has a pore and low permeability [15]. Based on the results obtained is known that the water absorption tends to increase with the increase of immersion time and the ratio of Si/Al (Figure 3). Geopolymer with a Si/Al ratio = 2 has the lowest water absorption. Besides that, the water absorption decreasing in 240 and 1440 minutes of time with increasing Si/Al ratio from 2.5 to 3.5.

In this study specimens heated in an oven for 24 hours at ± 112 °C. The purpose of this method is to obtain a mass constant of specimen. That is, the test specimen is completely dry in order for water testing occurs efficiently. The values obtained water absorption is still relatively very high. One of the factors that influence it is the level of dryness of the specimen. The specimens were dried at room temperature have a lower water absorption than the specimens are dried in the oven until a constant mass. Specimens are dried under saturated humidity after 28 days at 20 °C have a lower water absorption than the specimens are dried at 105 °C [16].

Another study describes the influence of pressure in the manufacture of geopolymer to water absorption. Increased water absorption with the increased pressure causes higher amounts of NaOH solution is squeezed out [17]. In such conditions, the geopolymerization reaction blocked so geopolymer gel formed slightly and then produce a high porosity of the geopolymer matrix. In other words, the higher geopolymerization process was inhibited so that the structure of geopolymer produced less homogeneous (Figure 7) as a result of the slag that is not involved in the reaction. After 1440 minute immersion, water absorption of geopolymer is decrease with increasing of Si/Al ratio. This results show that the increasing of Si/Al ratio role on the forming homogeneous microstructure so that decrease water absorption.

C. Effect of Si/Al Ratio to Compressive Strength

Figure 4 shows the compressive strength increasing with increasing ratio of Si/Al although there are deviations in Si/Al ratio = 2.0 and 3.0, respectively. However, they have a tendency compressive strength increase with increase of Si/Al ratio too. These results were confirmed by previous studies that an increase in the compressive strength with increasing concentration of Si although the increase was not linear [18].

Hardening process of specimens occurs because geopolymerization reactions that include condensation reactions that depend on the type of aluminate and silicate produce silicate and aluminate monomers. Silicate and aluminate monomers produced as a hydrolysis reaction or dissolution of Al^{3+} and Si^{4+} components form $[\text{Al}(\text{OH})_4]^-$, $[\text{SiO}(\text{OH})_3]^-$ and $[\text{SiO}_2(\text{OH})_2]^{2-}$. This process is influenced by the concentration of NaOH solution is used as the activator solution. At high concentrations of NaOH (8 molar), the early stages of the condensation reaction starting from a stable oligomer formation as a dimer result of condensation between $[\text{Al}(\text{OH})_4]^-$ and $[\text{SiO}_2(\text{OH})_2]^{2-}$ according to Equation 1. The resulting structure of Equation 1 is a stable dimer structures within the framework of polisialat (Si: Al = 1).

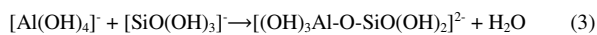
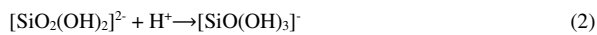


The low compressive strength is one factor that the delayed of geopolymerization process for low Si/Al ratio. In this condition, known simply occurred early high dissolution of Al. Low concentrations of Si (Si/Al ratio is low) yielding a poly(sialate) structure [7] as a result of the replacement of Si by Al. That is, at the low Si/Al ratio most of the aluminum is present in the form of $[\text{Al}(\text{OH})_4]^-$ monomer. The high $[\text{Al}(\text{OH})_4]^-$ monomer causes high alkalinity so that the reaction product in Equation 1 tends to react with $[\text{Al}(\text{OH})_4]^-$ condenses further aluminosilicate trimer [19]. Although this type of dimer and trimer produced were dominant, but in these conditions a higher polymerization is difficult that disrupts the hardening process of geopolymer. The high aluminate is then easily formed bond is the bond of Si-O-Al weaker than the bond Si-O-Si in geopolymer system. Theoretically, the bond between Si-O-Si is stronger than the Si-O-Al and Al-O-Al, which means that the strength increases with increasing Si/Al ratio.

The low ratio of Si/Al affects the loss of compressive strength associated with the low $[\text{SiO}(\text{OH})_3]^-$ structure to

form the larger geopolymer structure than the $[\text{SiO}_2(\text{OH})_2]^{2-}$ structure as a result of the high aluminate in the system. Equilibrium and stability of both types of silicate structure depends on the pH [20]. In speciation diagram of Si (Figure 5), the structure of $[\text{Si}(\text{OH})_3]^-$ is more stable than the structure of $[\text{SiO}_2(\text{OH})_2]^{2-}$ on high alkaline condition.

Increasing the ratio of Si/Al is useful to provide an silicate extra component in the form $[\text{SiO}(\text{OH})_3]^-$ monomer according to the reaction in Equation 2 [16]. Availability $[\text{SiO}(\text{OH})_3]^-$ monomer complete the process of condensation reaction (Equation 3) with $[\text{Al}(\text{OH})_4]^-$ and self-polymerization between groups silicate produced in the generation of silicate oligomer.



Further condensation process between the reaction product (Equation 3) with $[\text{SiO}(\text{OH})_3]^-$ to form a more stable products until all the $[\text{Al}(\text{OH})_4]^-$ monomer used in the reaction. In a system with a high concentration of Si (Si/Al ratio is high), the condensation process begins with the formation of oligomer silicates. Finally, the formation of a polymer network is composed [19].

The reaction continues until leads to the formation geopolymer structures of poly(sialate-siloxo), -Si-O-Al-O-Si-O- or poly(sialate-disiloxo), -Si-O-Al-O-Si-. The higher concentration of dissolved silicate source plays a role in increasing the amount of Si-O-Si bonds and compressive strength [21] [22]. In addition, the formation of geopolymer network depends on the concentration of $[\text{SiO}(\text{OH})_3]^-$. In the Si/Al ratio is low, the silicate monomer that resulted is $[\text{SiO}_2(\text{OH})_2]^{2-}$ and $[\text{Al}(\text{OH})_4]^-$, where the condensation reaction that occurs between the two tend to form small oligomers, such as dimers and trimers [19]. With increasing Si/Al ratio, the concentration of $[\text{SiO}(\text{OH})_3]^-$ also increased, where the condensation reaction between $[\text{SiO}(\text{OH})_3]^-$ with $[\text{Al}(\text{OH})_4]^-$ forming the large oligomers and lead to the establishment of the complex polymer network so that increasing the strength.

D. Relationship between Water Absorption and Compressive Strength of Geopolymer

Generally, there is a relationship between water absorption and compressive strength of geopolymer, where the higher the compressive strength indicates that the microstructures produced more homogeneous and dense so that tends to lower water absorption. At a ratio Si/Al = 1.5 to 3.0, water absorption is directly proportional to the compressive strength of up to 3.5 water absorption is inversely proportional to the compressive strength (Figure 6). Although there are deviations in the ratio Si/Al = 1.5 to 3.0, but at the ratio of Si/Al = 3.5 provides information that increases the compressive strength will cause decreases the water absorption as a result of homogeneous geopolymer produced and solid and has a low porosity. The optimum condition is obtained when the geopolymer material has high compressive strength with low water absorption, where the condition is achieved when the geopolymer cement has a ratio of Si/Al = 3.5.

E. SEM Analysis of Geopolymer Material

Durability of the structure of the construction material in its application is closely dependent on the physical-mechanical properties and its transport, such as permeability and ion diffusivity controlled by microstructural characteristics of hardening to a certain extent of the binder. Porosity and pore size distribution is a vital component of the microstructure of the material. It determines the permeability of binder gel and penetration rate by an aggressive agent [23]. The permeability property with regard to other physical properties, such as water absorption, which is one aspect to be considered in the application, especially in conditions that are susceptible to water. Changes in microstructure and porosity associated with lability of silicate species which control the rate of structural reorganization during geopolymerization, where the structure of $[\text{SiO}_2(\text{OH})_2]^{2-}$ more labil than $[\text{SiO}(\text{OH})_3]^-$. High lability promote the occurrence of large interconnected porosity as a result of low concentrations of Si [21].

SEM analysis performed one of which is to determine the microstructure and morphology of geopolymer product, especially regarding an overview of the porosity and size of the particles formed. Increasing the ratio of Si/Al contribute in the formation of microstructures that are more compact, dense and homogeneous. Geopolymer material with a ratio Si/Al = 2.5 is more homogeneous than the ratio of Si/Al = 1.5 (Figure 7 and 8). This is possibly due to the insoluble particles of slag. The insoluble of particles caused interface bonds with the binder is a sensitive area that reveal failures during the testing of compressive strength [2]. Thus, the higher of ratio Si / Al caused the formation of the structure of Si-O-Si and silicate species are more stable. This stability is formed by a further geopolymerization process leading to the formation of more complex network and homogeneous of geopolymer thus increasing strength.

IV. CONCLUSION

There is a tendency of increasing the compressive strength and reduced water absorption by increasing the ratio of Si/Al. The optimum condition was obtained when the ratio of Si/Al = 3.5. There is a possibility of increased compressive strength with Si/Al ratio of more than 3.5.

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