Synthesis and characterization of geopolymer from bottom ash and rice husk ash

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Abstract. All Geopolymer (GP) has been synthesized from bottom ash and rice husk ash. This research aims to determine the effect of Si/Al ratio on geopolymer synthesis. Geopolymer was synthesized with various Si/Al ratio of 2, 3 and 4. The characterization result using XRD and SEM indicated that by using a different ratio of Si/A, it will produce geopolymer with varied structure and morphology. Diffractogram result shows that polymerization has been done for all samples (GP2, GP3, GP4) with the presence of hump peak at 2θ = 27-35°. In GP4, no peak at 2θ = 18° indicating sodalite phase forming. Besides that, the morphology of geopolymer with a varied ratio of Si/Al shows that higher ratio will produce geopolymer with higher particle size. The highest compressive strength of geopolymer was obtained at a ratio of Si/Al = 4, with a maximum load of 12866 kgf.

1. Introduction

The contamination of heavy metals become attention because it has a negative impact on the environment. Heavy metals waste are produced by several industrial activities such as electroplating, battery, printed circuit board (PCB), metal coating treatment [1], mining and melting of metalliferous ores, municipal wastes, fertilizers, pesticides, and the use of pigment [2]. Rochayatun (2006) states that the content of Pb, Ni, Cu, Zn and Cd in Cisadane river are high, and even exceed the limit that published by KepMen LH/51/1995 about the standard of heavy metals waste.

The method that can be used for heavy metal treatment namely waste treatment using high pressure and temperature, the use of aerobic bacteria for degradation, combustion of the waste under high pressure and catalytic oxidation [4]. Yet, those methods have disadvantages, such as the limits ability to decrease the high concentration of waste, requiring extreme conditions, and limiting as each catalyst.

The use of porous materials is currently noted as an adsorbent. The use of porous material focuses on zeolite, carbon, geopolymer and the other materials. Geopolymer is amorphous, three-dimensional aluminosilicate binder material, that can be synthesized by mixing aluminosilicate reactive materials and strong alkaline solutions, followed by curing it at room temperature [5-6]. Geopolymer has potential applications in immobilization of heavy metal, reduce the emissions of CO₂ associated with cement manufacturing, high mechanical strength, flexibility, and durability. Based on that information, in this work, we report the synthesis of geopolymer using bottom ash and rice husk ash as aluminosilicate source. The compressive strength of geopolymer was measured after 28 days. The structure and morphology of this material were observed with XRD and SEM, respectively.
2. Experimental Section

2.1 Material and Instrumentation
The materials used in this experiment were rice husk ash from Madiun, bottom ash from PLTU Paiton, Na$_2$SiO$_3$, NaAlO$_2$, NaOH pellet (99% pa) as alkali activator, and distilled water. Powder X-ray diffraction (XRD) patterns were collected using CuK$\alpha$ radiation to identify crystal phase. Scanning Electron Microscope images were collected to identify the morphology of geopolymer. The compressive strength of geopolymer was investigated after 28 days in Physical Laboratorium of PT Semen Indonesia.

2.2 Procedure
Geopolymer samples were synthesized with mole ratio composition of NaOH/rice husk ash. All procedures were conducted at room temperature to minimize the effect of it. Geopolymer samples were synthesized by mixing bottom ash with NaOH as activating solutions for 5 minutes. Rice husk ash was added to the mixture and stirred for 5 minutes. The final composition of each geopolymer sample was cast in 5x5x5 cm$^3$ cube mold. Final geopolymer was dried for 24 hours at room temperature. After that, this geopolymer was dried at 75°C for 48 hours and cooled to room temperature. The compressive strength of each geopolymer was measured after 28 days.

3. Result and Discussion

3.1 Effect of Si/Al Ratio Towards the Structure of Geopolymer
The XRD characterization was used to determine the phase of geopolymer. It is because, the polymerization reaction can form multiple crystal phases. Crystalline phase were characterized by the presence of sharp diffractogram peaks, whereas the amorphous phase were characterized by the presence of hump with irregular intensity [11]. XRD characterization was performed on three samples of geopolymer (GP 2, GP 3 and GP 4) as seen in Figure 1. X-ray diffraction pattern of all geopolymer samples with Si/Al ratio variation showed the formation of some phase such as quartz, mullite, magnetite, and sodalite. Kim (2012) suggests that the presence of hump peak at 2$\theta$ = 27-35° indicates the formation of geopolymer has been done.

GP 4 has high intensity peak at 20 = 27° that confirms as quartz phase formation. In addition, GP 2 and GP 3 have a peak at 20 = 18° that confirm as sodalite phase formation. According to the Kusumastuti, (2012) experiment, sodalite phase causes the product of geopolymer become fragile and the compressive strength of this material becomes low.

![Figure 1](image_url). The XRD pattern of (a) GP 2 (Geopolymer with Si/Al ratio = 2), (b)GP 3 (Geopolymer with Si/Al ratio = 3), GP 4 (Geopolymer with Si/Al ratio = 4)
3.2 Effect of Si/Al Ratio Towards the Morphology of Geopolymer

Xu et al. (2000) state that geopolymer product does not have a stoichiometric composition and the mixture of amorphous to semi-crystalline structure and crystalline Al-Si particles through nano-crystalline that disperse in an alumino-silicate gel. Geopolymerization process includes leaching, diffusion, condensation and hardening step. Figure 2 showed the morphology of geopolymer with Si/Al ratio of 2, 3 and 4. A higher Si/Al atomic ratio corresponded to the more crystalline aluminosilicate gel and the increase in crystalline indicated increasing in compressive strength [7]. Figure 2 showed that the higher Si/Al ratio can produce higher crystallinity of geopolymer particle.

![Figure 2](image)

Figure 2. The morphology of (a) GP 2 (x500 and x1000); (b) GP 3 (x500 and x1000); (c) GP 4 (x500 and x1000)

3.3 Effect of Si/Al Ratio Towards the Compressive Strength of Geopolymer

Geopolymerization process occurs under an alkaline condition with rapid reaction. Minerals containing silica and alumina will react to produce chain and ring that contain three-dimensional polymers with Si-O-Al-O bond [9]. The source of silica and alumina for geopolymer synthesis can be derived from a natural source or commercial source. Chindaprasirt (2009) has been synthesis geopolymer from fly ash and bottom ash because these materials contain high silica content.

The compressive strength of geopolymer was investigated after 28 days. Tavor et al. (2007) state that the compressive strength of geopolymer will increase by time, up to 28 days. This means that polymerization process requires 28 days. The result of compressive strength test was showed in Figure 3. When the Si/Al ratio were higher, the compressive strength of geopolymer increases. This result occurs while higher ratio affects the increase of geopolymer crystallinity. This result is appropriate to the XRD result. Figure 3 shows that the higher Si/Al ratio of geopolymer sample, the higher the maximum load that can be detained. Davidovits (1991) states that there is a relationship between
mechanical strength and silica content. While silica content in geopolymer increases, it will improve the amount of Si-O-Si bond. This type of bond is stronger than Si-O-Al and Al-O-Al bonds. In other cases, if the ratio of Si/Al too high, will decrease the mechanical strength of geopolymer, due to the mixture of geopolymer producing the material that cannot react [12].

Figure 3. Maximum load that can be detained by geopolymer with Si/Al ratio variation

4. Conclusion
Geopolymer has been synthesized from bottom ash and rice husk ash with various ratio of Si/Al at 2, 3 and 4. The XRD and SEM result shows higher Si/Al ratio producing geopolymer with better structure and higher particle size, respectively.

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