Characterization of Geopolymer Paste based on Fly Ash and Bottom Ash in PLTU Kaltim Teluk using Sodium Hydroxide (NaOH)

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Abstract: Cement Industry in Indonesia has grown significantly every year. Limestone is one of the raw cement substitution material which has rapidly grown. The cement production process released carbon dioxide (CO2), which resulted in global warming. As an alternative material substitution, fly ash (FA) and bottom ash (BA) as the remained coal was generally used for cement production. PLTU Kaltim Teluk, one of the steam power plant which located in Balikpapan, East Kalimantan, produced 150 tons of FA in a day. However, FA and BA were laid out in the landfill and had not managed well. Actually, FA and BA had potential material as a substitution of cement in the paving block ingredients. Adding an alkaline activation, which consists of sodium silicate (Na2SiO3) and sodium hydroxide (NaOH), could be increased the compressive strength, especially for paste using 100% FA. The property of FA was characterized by Scanning Electron Microscope (SEM) and X-Ray Fluorescence (XRF). A compressive strength test was carried out on paste at the age of 3 and 14 days. The result showed that the highest compressive strength at 14 days was 13.93 MPa with 12 M NaOH. This research was conducted as an alternative solution to the waste of PLTU Kaltim Teluk as a substitute for cement in Balikpapan.

1 INTRODUCTION

Cement is a binder that is able to bind solid materials such as sand and stone into a compact unit when it mixed with water. The main raw materials in the process of making cement are limestone and clay; the main compounds in cement come from both of the materials (Rahadja, 2010). The ancillary materials of cement are silica sand, contributing to correct low level of silica (SiO2) in clay and iron sand as correction material of low-level Fe2O3 in main raw material (Farhan, 2016). The components of cement are lime (CaO), SiO2, alumina (Al2O3), ferric oxide (Fe2O3), magnesite (MgO), and a small number of other oxides (Rahadja, 2010).

The cement industry in Indonesia has experienced rapid development. The Ministry of Industry (2016) states that currently, the consumption of domestic cement is 62,4 million tons, with a total of national cement production reaching 68,7 million. The increased cement production causes cement raw materials needs also increasing; one of them is lime. Nur et al. (2015) stated that the use of lime to produce cement is the largest use of raw material from total raw material for 87,4% or 5.047.263,31 tons. The increasing consumption of limestone affects mining in the karst area, such as reduced water supply sources and environmental damage to the needs of the surrounding communities. On the other hand, the production process or forming karst to become cement results or releases carbon dioxide (CO2), where the CO2, itself, gas can cause the increase of global warming.

As an alternative to reduce mining in karst areas and to cope with increasingly high CO2 gas, other materials are required for replacing cement as a solution. There are various solutions or alternatives to substitute cement, including copper slag (Kadhafi, 2015), Lapindo mud (Susanto and Nugroho, 2012), rice husk ash (Anam and Sumarno, 2018), eggshell powder (Hibur, 2017), fly ash (FA), and bottom ash (BA). From all of those alternative materials, FA is able to become the best one because of the content of pozzolan as cement replacement with the highest level of SiO2, Al2O3, and Fe2O3.

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Furthermore, FA and BA able to be used in this research are the waste from the coal combustion process at Kaltim Teluk Steam-electric Power Station (PLTU). Everyday PLTU Kaltim Teluk produces FA of 150 tons (Rini et al., 2018). According to Government Regulation (PP), Number 101, the Year 2014 large amount of resulted waste in a period is categorized as specific hazardous and toxic material waste (B3). Therefore, FA and BA from PLTU Kaltim Teluk are categorized as specific hazardous and toxic waste.

So far, FA and BA of PLTU Kaltim Teluk are managed by using the third party. The increasing total amount of FA and BA in every year requires management in order to not causing environmental issues, for example, air or water pollution and decreased quality of the ecosystem. Due to that, one solution or alternative measure that is able to be conducted is to process FA and BA as substitution or replacement of cement in a mixture of pozzolan concrete, causing them to become good additive mineral for concrete (Adibroto et al., 2018).

2 MANUSCRIPT PREPARATION

2.1 Materials

Materials used in this research are FA and BA wastes, which were collected from PLTU Kaltim Teluk on February 4, 2019, solid Sodium Hydroxide (NaOH) pro analyst (PA) type from Ensure ISO brand, and solution of Sodium Silicate (Na2SiO3) from Pudak Scientific brand.

2.2 FA Characterization

FA raw material is tested by using a Scanning Electron Microscope (SEM) of FEI type from Inspect-S50 brand and X-Ray Fluorescence (XRF) from Panalytical brand with Minipal 4 type. SEM is intended to determine shape, size, and surface structure of FA grain, meanwhile XRF is intended to analyze chemical composition and element concentrate in FA.

2.3 The Making and Vicat Test on Portland Cement Paste

In this procedure, making and Vicat test is conducted on the cement paste. The purpose of this procedure is to find out the effect of setting the time on the cement paste. In order to figure out the setting time of a cement paste, the normal consistency of a cement test should be carried out first so that the need for water is able to be determined. Steps of implementation in normal consistency of a cement paste test are in accordance with SNI 03-6827-2002 (Normal Consistency Test Method of Portland Cement Using Vicat Apparatus for Civil Work) that the object of cement test is prepared with the composition of cement and water in table 1. Afterward, cement is put in, and water is added into the mixing pan to form cement paste. After mixing has been completed, it is formed into ball-shaped by using a hand. Then, the cement paste is put into Vicat mold through its base hole until fully filled and place it on the glass plate. Next, the Vicat mold filled with cement paste is positioned on the Vicat apparatus, and then the tip of the Vicat rod is contacted to the center of cement paste surface, and the position of the Vicat rod is tightened.

Table 1: Composition of portland cement paste for the Vicat test.

Cement (gram)	Water (mL)
300	76,95

Steps of setting time tests are in accordance with SNI 03-6827-2002 (Initial Setting Time Test Method Using Vicat Apparatus for Civil Work) that 300 grams of cement and volume of water required for normal consistency in accordance with normal consistency of a cement paste test method are prepared. Next, cement is put in, and water is added into the mixing pan to form cement paste. Afterward, a vicat mold filled with cement paste is stored along with the glass plate in a humid cabinet, and the concrete thermometer is positioned on the Vicat mold and counted for 30 minutes by using a stopwatch. After that, the vicat mold filled with cement paste is positioned on the Vicat apparatus, then the tip of the Vicat rod is contacted on the center of the cement paste surface, and the Vicat rod is tightened. This process is repeated every 15 minutes for other different holes on the cement paste surface. The space between penetration points is no less than 5 mm to the inner sidewall of mold, and the space between 2 penetration points is no less than 10 mm.

2.4 The Making and Vicat Test on Geopolymer Paste

In this research, the used composition of FA and alkaline activation to form geopolymer paste is determined by the Vicat test result. The mass ratio between Na_2SiO_3 solution and NaOH solution used for alkaline activator is 3: 2. The composition of FA and alkaline activation used on the Vicat test are shown in table 2.

Table 2: Composition of FA and alkaline activation for Vicat test

Variable	FA (gram)	Alkaline Activation		FA: Alkaline Activation Ratio
		Na_2SiO_3 (gram)	NaOH (gram)	
100% FA	300	159,62	106,42	53% : 47%
100% FA	300	166,15	110,77	52% : 48%
100% FA	300	172,94	115,29	51% : 49%

In this procedure, forming and Vicat test on the geopolymer paste are conducted. The purpose of this is to figure out the effect of setting the time on geopolymer paste. Steps of implementing setting time tests are in accordance with SNI 03-6827-2002 (Initial Setting Time Test Method on Portland Cement Using Vicat Apparatus for Civil Work) FA and alkaline activation mass are prepared in accordance with table 2. Afterward, FA is put in, and alkaline activation is added into mixing pan to form a paste. Then, the geopolymer paste is put into Vicat mold. The top surface of geopolymer paste is leveled with one movement using the scoop and smooth off the surface of geopolymer paste using the tip of scoop without applying pressure on geopolymer paste. After that, the vicat mold filled with a geopolymer paste is stored in the humid cabinet along with a glass plate, and the concrete thermometer is positioned on the Vicat mold and count for 30 minutes using the stopwatch. And after that, Vicat mold filled with geopolymer paste is positioned on the Vicat apparatus, then the tip of the Vicat rod is contacted on the center of the geopolymer paste surface, and Vicat rod is tightened. This process is repeated every 15 minutes for other different holes on the geopolymer paste surface. The space between penetration points is no less than 5 mm on the inner sidewall of mold, and the space between 2 penetration points is no less than 10 mm. Every time this penetration is carried out, the Vicat needle shall be cleaned and always align, and there should be no vibration. The steps are repeated for Vicat mold with a composition ratio of FA and alkaline activation.

After the Vicat test on geopolymer paste is conducted, the result which is close to the result of the Portland cement Vicat test, the ratio of FA and alkaline activation are acquired with the percentage of 51 %: 49%. The making of paste is carried out to find out the maximum compressive strength acquired on concentrate variation of NaOH solution. The composition of making geopolymer paste for six Vicat molds is shown in table 3.

Table 3: Composition of making geopolymer paste

Vicat Mould Code	FA (51%) (gram)	Alkaline Activation (49%)	
		Na2SiO3 (gram)	NaOH solution (gram) (molarity)
P-8	600	345,87	230,58 (8 M)
P-9	600	345,87	230,58 (9 M)
P-10	600	345,87	230,58 (10 M)
P-11	600	345,87	230,58 (11 M)
P-12	600	345,87	230,58 (12 M)

2.5 Curing Geopolymer Paste

In this research, after a geopolymer is removed from the mold, it is continued by curing the geopolymer paste. The type of curing that is used is a process of treatment by placing a geopolymer paste in open space with the specified time. Curing is conducted on normal curing temperatures for 3 days and 14 days.

2.6 Compressive Test

On the paste compressive strength test, a compressive test machine is used. The first step of the compressive paste test is to clean the test object from any attached dirt using a wet cloth. Afterward, the paste is weighed to determine the mass of the test object. After that, it is placed on a compressive test machine centrally. Then, the compressive test machine is turned on and compress the test object until it is cracked or splinter. After that, record the maximum load of the test objects displayed on the compressive test machine. And then, calculate the paste compressive strength, which is the load per metric unit in N/mm2 or MPa.

3 RESULTS AND DISCUSSION

3.1 XRF Test on FA

XRF test is conducted to find out the chemical composition in FA.

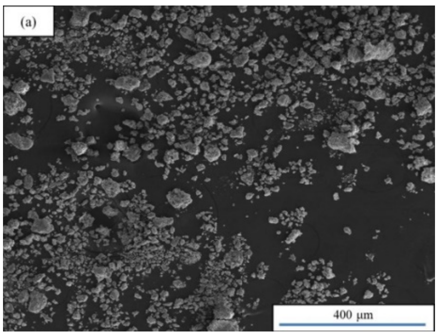
Table 4: The result of the XRF test on FA from PLTU Kaltim Teluk

Element	Concentrate (%)	Compound	Concentrate (%)
Al	5,60	Al2O3	7,80
Si	11,50	SiO2	18,00
S	1,60	SO3	2,80
Ca	27,10	CaO	24,70
Fe	42,30	Fe2O3	35,80

In research, Akinyemi and Mugera (2018) specified that class C FA has SiO_2 + Al_2O_3 + Fe_2O_3 with the amount of 50% - 70%, a maximum of 5% of SO_3, and more than 10% CaO. In Table 5, the result of the XRF test on FA of PLTU Kaltim Teluk has SiO_2 of 18%, 7,8% of Al_2O_3, and 35,8% Fe_2O_3. This indicates the total of SiO_2 + Al2O_3 + Fe_2O_3 for 61,6%, 2,8% of SO_3, and 24,7% of CaO. Thus, it can be inferred that the FA at Kaltim Teluk can be classified as class C FA.

3.2 SEM Test on FA

SEM is conducted to acquire an overview of morphological conditions on FA, which is used as a basic material in synthetizing geopolymer paste activated by an alkaline activation solution. In research conducted by Akinyemi and Mugera (2018), it is stated that generally, the FA particle has irregular and porous microscopic structures. The result of observation on the FA particle of PLTU Kaltim Teluk using the SEM test in figure 1 with a magnification value of 250x and 10.000x shows that FA of PLTU Kaltim Teluk has an irregular particle. In research conducted by Sinaga (2018), it is said that irregular FA particles will decrease workability because FA is unable to enter through the paste, causing the occurrence of pores or tiny holes on paste and slowing down the hydration process.



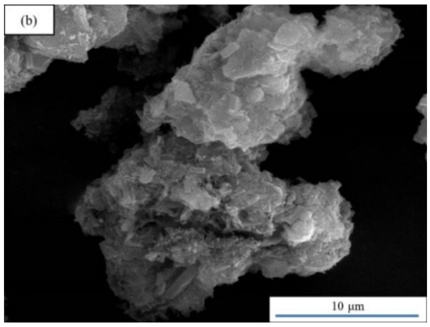


Figure 1: SEM of FA (100 wt%) with magnification value of (a) 250x and (b) 10.000x (Writer, 2019)

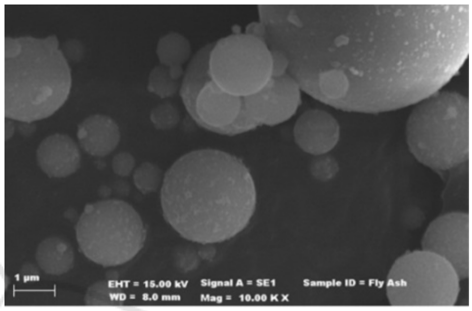


Figure 2: SEM of FA (Ilmiah, 2017)

In research conducted by Rommel, et al. (2014), as well as Ilmiah (2017), displayed in figure 2, also specified that the smoothness of the particle is able to affect the hydration process and setting time. Reactions between pozzolan and water are commenced from the pozzolan particle surface, so the more area of particle surface is, the quicker is its hydration process. This means that a smooth particle will strengthen and generate faster hydration heat than the rougher one. In addition, the spherical particle will increase workability resulting in the reduced need for water, easily bind each other, and also narrow the space between mixed materials. In conclusion, the best shape of FA farticle to be used for making geopolymer paster is spherical and possess a smooth surface.

3.3 Normal Consistency Test and Vicat Test

The steps of normal consistency test of a cement paste are in accordance with SNI 03-6827-2002 (Normal Consistency Test Method of Portland Cement Using Vicat Apparatus for Civil Work) which is conducted in order to make the requirement of water in forming cement paste on setting time test is able to be determined. Cement paste reaches normal consistency when the depth of cement paste penetration is 10 ± 1 mm.

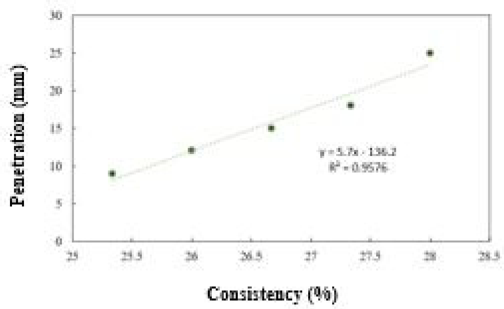


Figure 3: Graphic of normal consistency on cement paste (Writer, 2019)

It is showed in figure 3 that the linear equation that can be used as a measurement of required water to reach normal consistency of a cement paste with a penetration of 10 mm. In normal consistency, cement paste, when penetrated 10 mm, is 25,7%. The total amount of water required in setting the time test of a cement paste is 76,9 grams. In accordance with SNI 03-6827-2002 (Initial Setting Time Test Method Using Vicat Apparatus for Civil Work), the setting time test is conducted to find out the effect of initial setting time and final setting time of a cement paste. Initial setting time is the time required for a cement paste to change its property from liquid to solid, while final setting time is the time where penetrated Vicat needle is visually visible. The initial setting time is at least 45 minutes, and the final setting time is 375 minutes in maximum. Initial setting time is reached when the Vicat needle enters into the paste in 30 seconds with penetration of 25 mm. Meanwhile, the final setting time is reached when the Vicat needle does not come into the paste in 30 seconds or does not penetrate the surface of paste at all.

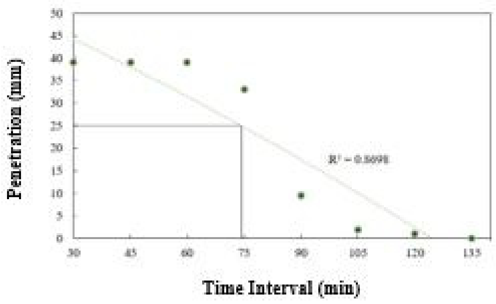


Figure 4: Graphic of Vicat test result on cement paste (Writer, 2019)

In figure 4, during the penetration of 25 mm, the initial setting time is acquired in the 74th minute, and the final setting time is in the 135th minute. Setting the time on a cement paste is used as a reference in creating geopolymer paste (Manuahe et al., 2014).

3.4 Compressive Strength Test on Geopolymer Paste

The purpose of this compressive strength test on the geopolymer test is to find out the concentrate of NaOH solution optimally required. In figure 5, it is showed that the compressive strength of geopolymer paste with the concentrate of 12 M NaOH has a maximum compressive strength value of 9,51 MPa on the 3rd day and 13,93 MPa at the 14th day. It means that there is an increased value of 31,78% for 14 days. From the compressive strength test on geopolymer paste showed in figure 5, then the optimal NaOH concentrate required is 12 M with the ratio of alkaline activation solution of 3: 2.

Similarly with researches conducted by Setiadji (2011), Yuanda, et al. (2015), Qomaruddin, et al. (2018), Abdullah, et al. (2011), and Irawan, et al. (2015) stated that the higher is the concentrate of NaOH solution, and the increased age of the paste is, the higher is the resulted compressive strength. And also, the concentrate of the NaOH solution produces the highest compressive strength of 12 M. These are caused because of the effect of FA on the paste compressive strength. Reactions between calcium hydroxide (Ca(OH)2) with SiO2 in FA form calcium silicate hydrate (CSH) compound with the property of increasing strength.

$$3Ca(OH)_2 + 2SiO_2 + H_2O \rightarrow 3CaO.2SiO_2.3H_2O$$

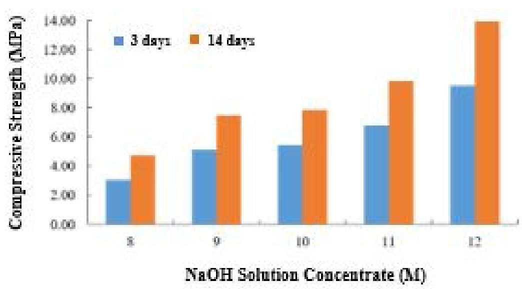
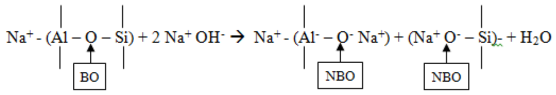


Figure 5: Graphic of NaOH solution concentrate on compressive strength (Writer, 2019)

Ferdy (2010) and Ren (2015) explained that on FA geopolymer and alkaline activation solution would synthesize, forming solid material through the polymerization process. In this polymerization process, there are 3 phases, where there is the dissolution of Si and Al at the first phase.



In accordance with the reactions, NaOH solution in polymerization has a role in dissolution Si and Al of FA, and as a supplier of Na+ to make the charge remains balance. On high concentrate, NaOH solution has more Na+ and is able to cause flash setting, because of high alkalinity and prompt dissolution of Ca-Si. The Na+ cation strikes the surface of the solid phase to dissolve Si and Al. High OH concentrate in NaOH solution is able to accelerate the dissolve of Si and Al in FA to make it more optimal. Alkaline activation solution of aluminosilicate involves chemical reaction where an atom of bridging oxygen (BO) in the structure of aluminosilicate is transformed into non-bridging oxygen (NBO), which leads to Si and Al isolation.

Adding Na_2SiO_3 also helps to slow the setting on a paste, providing more time for the Si and Al dissolution. Zhang et al. (2015), said that Si on FA is dissolved by NaOH solution and becomes one of Si sources. Then, other sources of Si are provided by Na_2SiO_3 directly. Moreover, Na_2SiO_3 in alkaline activation solution accelerates the polymerization.

The second phase is the hydrolysis of s and Al, where this process causes the forming of Si-OH and Al-OH bond.

$$Al_2O_3 + 3H_2O + 2OH^- \rightarrow 2[Al(OH)_4]^-$$
$$SiO_2 + H_2O + OH^- \rightarrow [SiO(OH)_3]^-$$
$$SiO_2 + 2OH^- \rightarrow [SiO_2(OH)_2]^{2-}$$

In this stage, there is the decomposition of Si and Al in FA to become monomer, then followed by a polycondensation process from monomer into polymer structure with a three-dimensional molecule network. On a high ratio of Si/Al, there will be condensation between silica and will form polymer silicate with Al. Adding Na_2SiO_3 into a geopolymer mixture will help increase the ratio of Si/Al inactivation phase. When the polycondensation process is ongoing, which is endothermic, where the heat is acquired from the curing process determined by temperature. In this process, a geopolymer will absorb the aluminosilicate compound.

4 CONCLUSIONS

This research shows that the highest compressive strength on geopolymer paste is acquired when the alkaline activation solution is used with a NaOH solution of 12 M concentrate with a compressive strength of 13,93 MPa at 14 days.

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