Characterization of Fly Ash on Geopolymer Paste

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Characterization of Fly Ash on Geopolymer Paste

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Abstract.

The effects of loss of ignition, specific gravity, fineness, specific surface area and soluble fly ash to compressive strength of geopolymer paste are presented. Six fly ashes from two different sources and different time of collections were evaluated. Sodium hydroxide and sodium silicate were used as alkali activator. Concentration of sodium hydroxide and mass ratio of sodium hydroxide to sodium silicate were fixed at 14M and one, respectively. The result indicated that the improvement in compressive strength of paste was more influenced by fineness, specific surface area and soluble content of fly ash. Soluble content of fly ash greatly affected the compressive strength of geopolymer paste compared to the compressive strength of cement paste with 20% fly ash replacement.

Introduction

This paper presents the effect of physic and chemical properties to compressive strength of geopolymer paste on various of fly ash and compares with cement paste that uses the same fly ash.

Geopolymer is a new and environmental friendly material that is very popular among practitioners and researchers. Portland cement is not used in geopolymer material and entirely replaced with substitute materials that are pozzolanic materials and high content of silica (SiO_2) and alumina (Al_2O_3).Waste material such as fly ash and rice husk ash are usually used as raw material. Alkaline solution is added into fly ash to produce a binder. The physical, chemical and mechanical properties of geopolymer are strongly influenced by the properties of raw material such as fly ash [1, 2]. However the properties of fly ash vary depends on the quality of coal, temperature and process of combustion [1, 2, 3, 4]. Therefore the characteristics of fly ash are required.

Fly ash properties have been investigated by some researchers. Six fly ashes from South Africa and Australia had been investigated by Jaarsveld et al. [1] in order to evaluate the effect of using different fly ashes on properties of geopolymer paste. The result showed that the setting time and properties of hardened geopolymer paste were influenced by zeta-potensial of fly ash particle and calcium content. Jiménez & Palomo [5] examined chemical, physical, mineralogical and microstructure of Spanyol fly ash to determine the reactivity of fly ash. The percentage of reactive silica (SiO_2) was the most important factor in chemical properties of fly ash. They recommended that the reactive silica content was around 40-50%. Furthermore Somna, et al. [6] evaluated effect of class C fly ash which retained on 45μm sieve less than 2% to compressive strength of paste geopolymer on different NaOH concentration. The result indicated that the smaller particle showed better strength than the coarser particle fly ash.

Some investigations had been conducted by some researchers to evaluate the quality of Indonesian fly ash [7, 8]. The physical and chemical properties of fly ash were studied related to mechanical properties to find out the reactivity of fly ash. A strength activity index (SAI) was used to determine pozzolanic activity of fly ash. SAI is percentage of compressive strength of mortar with fly ash replacement over compressive strength of mortar control. The result showed that compressive strength of mortar was more affected by particle size, CaO and soluble content of fly ash. However, these researches were carried out on fly ash replacement cement paste between 10-20%. That was why the characterization of Indonesian fly ash on geopolymer paste was required. In this study, the evaluation on six Indonesian fly ashes had been conducted to evaluate the effect of fly ash characteristic on the strength of geopolymer paste. As well as on research above [8], the characteristics of fly ash are expected to give influence to the strength of geopolymer paste.

Materials

Six fly ashes that were collected from two industries in East Java Indonesia to be used in this research. The name of fly ash was based on their sources. Fly ash SB was from PT. Surya Beton Indonesia and SI was from PT. Semen Indonesia. While sodium silicate solution with 18% Na_2O, 36% SiO_2 and 46% H_2O and sodium hydroxide were used as the alkaline activator.

Mix Composition and Procedures

All fly ashes were mixed with the same geopolymer paste compositions. 560gr of NaOH flakes were mixed with distilled water to produce one liter NaOH 14M solution and prepared for 24 hours before mixing. Then sodium silicate and sodium hydroxide solution were mixed prior to blend with fly ash. Mass ratio of sodium silicate to sodium hydroxide was one while mass ratio of alkaline activator to fly ash was 0.35.

The procedure of mixing was to put fly ash into mixing bowl and poured the alkali activator then mixed them for two minutes. The material which was stuck on the side of the bowl was cleaned and then mixed again for one minute. The fresh paste poured into cylinder mold specimen with diameter of 2mm and height of 4mm as shown in Fig. 1a. All specimens were kept in the room temperature for 24h before being removed from the mold and stored in a closed container until the days of testing (Fig. 1b).

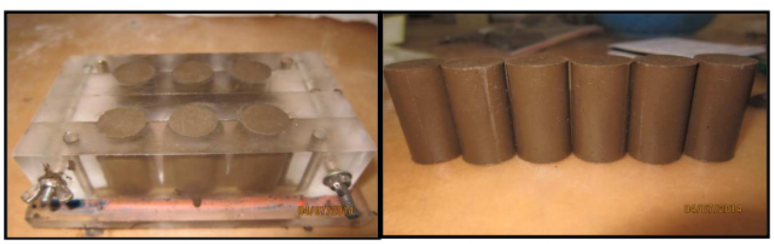

Fig. 1. (a) mold specimens; (b) specimens

The characteristics of fly ash such as loss of ignition (LOI), specific gravity, fineness and specific surface area were examined in this research. These examinations were based on ASTM C311-04 [9]. Scirocco machine and mastersizer 2000 software were used to determine and analyse the specific surface area. X-Ray Fluoressence (XRF), X-Ray Difractometer (XRD) and insoluble test were also conducted to analyse the chemical composition oxides, mineral and soluble content, respectively in all fly ashes. While the compressive strength of geopolymer paste were determined at 3, 7, 14, 21, 28 and 56 days. Universal testing machine type AU-5 with 5 ton capacity was used to determine the compressive strength of paste.

Meanwhile the same sources and date collections of fly ashes were also used for cement blend paste with 20% cement replacement. Two types of fly ash finenesses were used, an original fineness or unsieved fly ash (UFA) that was used in geopolymer paste previously and sieved fly ash (SFA). SFA was obtained by sieving the UFA with sieve no.200 (75μm). The weight ratio of fly ash to cement was 1:4 with normal consistency. The fresh paste were cast into 50mmx100mm cylinder molds. After casting, all specimens were cured in steam curing chamber for 2h in 20°C then increased the temperature into 80°C in 15 minutes. Kept the temperature constant for 3h and then decreased temperature into 20°C in 15 minutes. At the end of curing system, the specimens were removed from the mold and kept in room temperature until the day of testing. The compressive strength of UFA and SFA were determined at one day.

Result and Discussion

The chemical composition oxides of all fly ashes with XRF analysis are listed in Table 1. Different date of fly ash collections are indicated by the number on the fly ash code as shown in Table 1. Based on ASTM C618 [10] all fly ashes are categorized as class F fly ash except SI2 is classified as class C fly ash. It is because the amount of SiO_2, Al_2O_3 and Fe_2O_3 a bit less than 70%. The chemical composition of fly ashes from the same source but different batches SB1, SB2, SB3 and SB4 are similar on SiO_2 and Al_2O_3 content. High difference occurs on CaO and Fe_2O_3 content. While the composition of fly ashes SI1 and SI2 have significantly different on SiO_2, Al_2O_3 and CaO content. Only two of six fly ashes have CaO less than 5%. SB1 and SI1 are the highest and the lowest of CaO content, respectively. All fly ashes have Fe_2O_3 more than 10% whereas Jiménez & Palomo [5] suggested Fe_2O_3 less than 10% and low content of CaO to obtain maximum strength.

Table 1. Chemical Composition of Fly Ash [mass %]

Fly Ash Code	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	P_2O_5	SO_3	$SiO_2+Al_2O_3+Fe_2O_3$
SB1	37,45	24,49	15,73	11,79	1,13	0,32	0,99	2,52	0,80	73,72
SB2	38,76	29,43	4,05	18,43	0,88	0,01	0,88	2,19	0,67	86,61
SB3	39,16	31,03	10,26	11, 60	0,46	0,72	0,68	2,27	0,70	81,79
SB4	39,11	30,92	10,04	11,93	0,47	0,62	0,69	2,30	0,69	81,96
SI1	41,17	34,48	1,89	11,54	1,05	1,74	0,96	2,04	0,65	87,19
SI2	34,65	22,55	15,24	12,52	1,31	6,82	1,58	3,23	1,17	69,72

X-ray diffraction patterns of all fly ashes are shown in Fig. 2. The peak of quartz intensity of fly ash SI2 is the lowest while the highest belongs to SI1. SI2 is more reactive as compare with other fly ashes. Mineral mullite is found on SB2, SB3, SB4 and SI1.

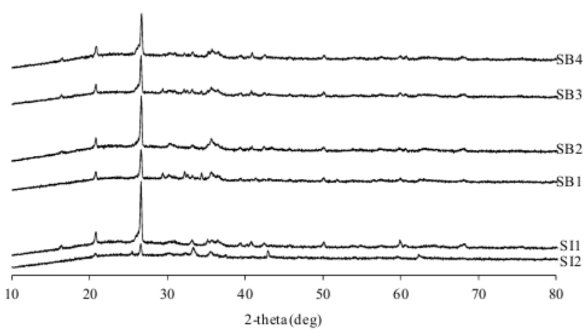

Fig. 2.XRD patern of six type of fly ash

Physical and chemical properties of six types of fly ashes are listed in Table 2. It shows a different of both physical and chemical properties of fly ash even though they were collected from the same source.

Table 2. Physical and chemical fly ash properties

Code Fly Ash	LOI (%)	Specific Gravity (gr/cm^3)	Fineness (%)	Spesific Surface Area (m^2/g)	Soluble Fly Ash (%)
SB1	0.62	2.21	15.4	0.989	41.39
SB2	0.86	2.31	17.2	0.966	40.04
SB3	1.41	2.53	15.8	0.854	40.23
SB4	0.82	2.42	19.0	0.863	42.75
SI1	0.47	2.28	17.6	0.851	47.03
SI2	0.29	2.60	10.6	1.110	54.50

Reactivity of fly ash depends on the amount of LOI that is unburned material [5, 11]. All fly ashes have LOI less than 6% as recommended by ASTM C618 [10] while Jiménez & Palomo [5] proposed less than 5%. Fly ash SI2 has the lowest content of LOI. While specific gravity of fly ashes between 2.21 to 2.60 gr/cm^3 and fineness between 10.6 to 19.0%. The fineness is the percentage of particle fly ash retain on the 45μm (#325) mesh sieve. ASTM C618 [10] suggests the fineness maximum is 34%. Fly ash SI2 has the lowest fineness only around 10.6%. Furthermore fly ash SI2 has the highest specific surface area and percent of soluble fly ash as given in Table 2. The effect of these properties to compressive strength of geopolymer paste would be discussed next.

The compressive strength of geopolymer paste at different ages is illustrated on Fig. 3. The compressive strength value is the average of six samples. It shows compressive strength increase with ages of paste. SI1 and SI2 were collected from the same source but different batches. The hardened geopolymer paste at 3 days reach the higher compressive strength at about 50.9MPa for SI2 compare to SI1 just around 38.3MPa. The compressive strength SI1 and SI2 at 28 days are 64.7MPa and 108.8MPa respectively. As mention previously that large difference of chemical compositions (SiO_2, Al_2O_3 and CaO) occur between these two fly ashes. The percentage of CaO content is higher 13.35% in SI2 (Table 1). Higher content of calcium increase the compressive strength of geopolymer paste [1, 2].

The compressive strength of SB2 drops at 3 and 7 days only around 5.6MPa and 16.3MPa, respectively. The lowest compressive strength on SB2 is affected by highest content of Fe_2O_3 (18.43%) and lower content of CaO (4.05%). Fernández and Palomo [5] recommended Fe_2O_3 content less than 10% to achieve maximum strength. CaO content of fly ash SB1 is the highest as listed in Table 2, however its compressive strength just around 26.47MPa. The reason of lower strength of SB1 will be discussed later. In spite of the percentage of CaO higher indicates higher compressive strength but the setting time is very fast. On the other hand, the percentage of CaO less than 5% decreases compressive strength. So that Diaz et.al. [2] suggested that CaO content just between 5 to 15% which could be used as geopolymer material.

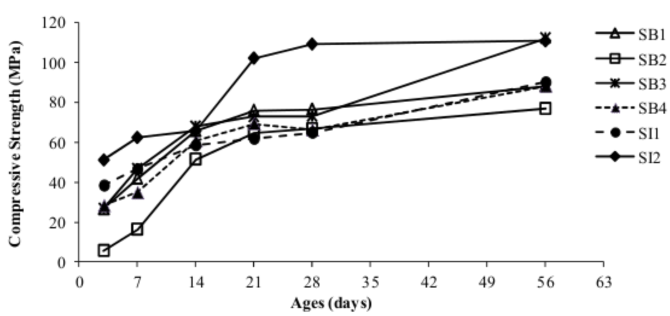

Fig. 3. Compressive strength geopolymer paste at different ages

Effects of fly ash characteristics which are given in Table 2 to compressive strength at 28 days are illustrated on Fig. 4 to Fig. 7. Fig. 4 shows the compressive strength decreases with increasing percentage of LOI. The carbon content in fly ash that is indicated as LOI is unreactive particle and porous [1, 5, 8]. The higher specific surface of this material requires the higher ratio of liquid/solid and lower the pozzolanic reaction [2, 5, 11]. Higher LOI means higher content of unreactive particle so that effect of high of LOI content will reduce the compressive strength. However in this research, the effect of LOI to compressive strength of geopolymer paste is not significant.

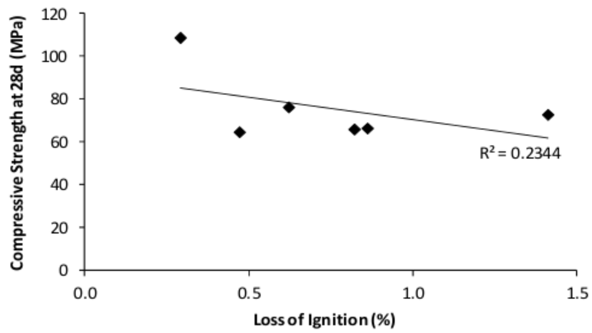

Fig. 4. Relationship between LOI to compressive strength

Fig. 5 illustrates that the compressive strength of geopolymer paste is affected by specific gravity of fly ash. The compressive strength increases as increasing the specific gravity of fly ash. Fly ash SI2 has the highest spesific gravity 2,6 gr/cm^3 and the highest compressive strength at 28 days of 108,8MPa. Higher specific gravity means more dense the particle of fly ash so that it increases the compressive strength. However specific gravity is not significant influence the compressive strength of geopolymer paste.

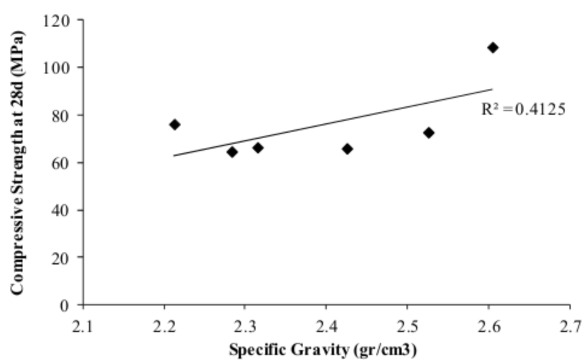

Fig. 5. Effect specific gravity to compressive strength

Influence of fly ash fineness (percentage particle retained on sieve no. 325) on compressive strength of geopolymer paste can be seen in Fig. 6a. The graph indicates that the compressive strength drops when the fineness increases. Lower fineness means the finer particle and vice versa. The biggest particle fly ash SB4 has lower compressive strength of 66MPa at 28days. On the other hand the smallest particle of fly ash SI2 indicates the highest compressive strength around 108.8MPa at 28 days. This is connection to the highest surface area of fly ash SI2 than other fly ashes as given in Table 2. Low percentage of particle fly ash retained on 45µm sieve shows higher specific surface area and higher reactivity of fly ash and subsequently increases the compressive strength of paste [2, 12]. Similar result occurs on cement paste which partially replaced with fly ash [13]. The decrease amount of fly ash retained on 45µm sieve cause the decrease of pore size and total porosity so that increases the density, durability and compressive strength of paste. It occurred because fly ash more reactive on smaller particle compare on bigger particle of fly ash [13].

Somna, et al. [6] evaluated effect the fineness of fly ash particle to compressive strength of paste geopolymer on different NaOH concentration. The result showed compressive strength of UFA (unsieved fly ash) only around 12MPa for 14M NaOH at 28 days while compressive strength of SFA (sieved fly ash) around 23MPa increase about 91.7% [6]. It was revealed that the smaller particle fly ash produced the higher compressive strength of paste. However this compressive strength was less than compressive strength in present research. Somna, et al. [6] used only NaOH as alkali activator while in this research NaOH and Na_2SiO_3 were used as alkali aktivator. The present sodium silikat in aktivator increases the compressive strength because the reaction in geopolymer occurs faster than using only sodium hidroksida [14, 15].

Fig. 6b describes the compressive strength of geopolymer paste that is affected by specific surface area of fly ash. Specific surface area is defined as the surface area per mass unit (m^2/g). It can be seen that compressive strength increases with increasing the specific surface area of fly ash. The same result was obtained on geopolymer paste which conducted by Kumar and Kumar [16].

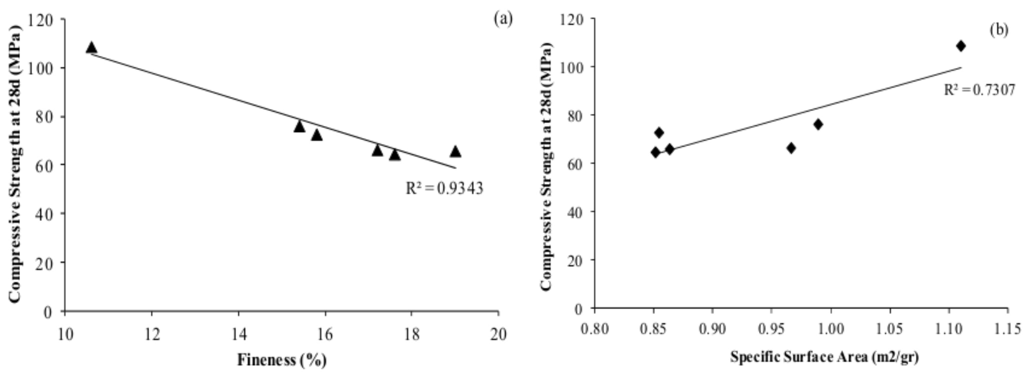

Fig. 6. Effect of fineness (a) and specific surface area (b) to compressive strength

Fig. 7 describes relationship between the percentage of soluble fly ash and compressive strength. Soluble fly ash indicates reactive fly ash. It reveales that increasing soluble fly ash is followed by increasing compressive strength geopolymer paste at three days and cement paste at one day. The percentage soluble fly ash SI2 is 54.5% (Table 2) that exhibits the highest compressive strength. As mention before, the compressive strength of SB1 is only 26.47MPa eventhough the CaO content as high as 15.73%. It occurs because the soluble content is only 41.39%. It should be noted that soluble content is more influence to compressive strength than CaO content.

The soluble fly ash more influences to geopolymer paste than cement paste with 20% fly ash replacement. It should be noted that geopolymer paste consists of 100% fly ash. So that soluble fly ash has significant effect to mechanical properties of geopolymer rather than cement paste. So it can be concluded that fineness, specific surface area and soluble content are strongly affected to compressive strength on geopolymer paste.

Fig. 8 shows the comparison between compressive strength of geopolymer paste at three days and cement paste with 20% fly ash replacement at one day for UFA and SFA. The compressive strength of SFA paste is slightly higher than UFA because the particle size of SFA is less than 75µm. The reactivity increases as the particle size of fly ash decreases [6, 13]. The graph indicates that compressive strength of geopolymer is significant higher than cement paste. The compressive strength of fly ash SI2 is the highest. However geopolymer compressive strength of fly ash SB2 has lower compressive strength than cement paste. This may be caused by the highest Fe_2O_3 content (Table 1) and the lowest content of soluble fly ash (Table 2) which is only about 40%.

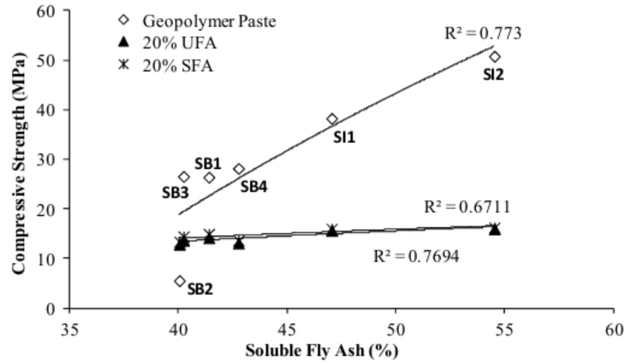

Fig. 7. Effect of soluble fly ash to compressive strength

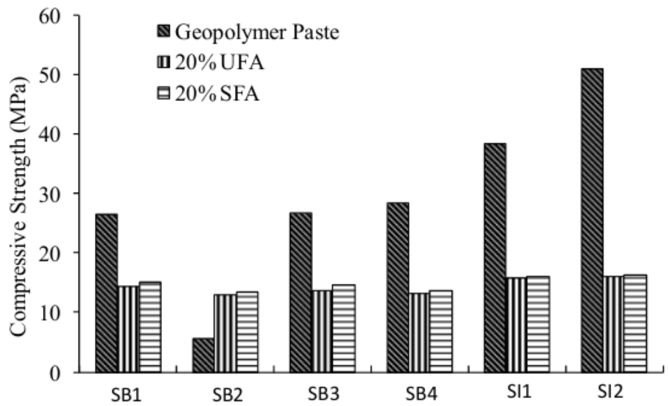

Fig. 8. Compressive strength of all fly ashes

From the discussion above, it can be observed that the fineness, the specific surface area and soluble of fly ash are significantly affected to the compressive strength of geopolymer paste.

Conclusion

1. Fly ash SI2 is the best performance compare to other fly ashes.
2. The compressive strength of geopolymer paste increases whenever decreases the loss of ignition (LOI), increases the specific gravity, decreases the fineness (% retained on 45μm sieve), increases specific surface area, increases the soluble content of fly ash.
3. The compressive strength of geopolymer paste is influenced by physical and chemical characteristic of fly ash, however the compressive strength of geopolymer paste is more affected by the fineness, specific surface area and soluble content.
4. The physical and chemical characteristics of fly ash affected significantly on geopolymer paste compares to on cement paste 20% replacement fly ash because the geopolymer contains 100% fly ash.

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