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4 Compressive strength of medium calcium fly ash based geopolymer paste

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Abstract. This study discusses the effect of heating duration and curing age on compressive strength geopolymer paste made with medium calcium fly ash and potassium hydroxide solution (KOH). The hardening process was performed in an oven at a temperature of $100 \pm 5^\circ\text{C}$ in a variety of heating duration for 2, 4, and 6 hours to form a geopolymer paste with a potassium hydroxide concentration of 6 M. Flow testing on fresh paste indicates that all materials were bonded properly without segregation. After the heating process in the oven, the specimens were cured at room temperature until the age of 3, 7, and 28 days. The results of the compressive tests indicated that the heating duration and curing age were the key factors. Where there is no visible negative effect of the medium level CaO content in fly ash against compressive strength development of the hardened geopolymer paste.

1. Introduction

Concrete is known as a main material for infrastructures. The infrastructure development made from concrete will be increasingly growing with Portland cement as a primer binder (cementitious material). Producing 29 ton of Portland cement will emit about 1 ton of CO₂ gas emissions [1]. This amount shows the enormous effect of Portland cement production to atmospheric CO₂ concentrations.

On the other hand, fly ash is the residual material generated from coal fire power plant. In 2014, it was reported that the energy industry with coal fuel produced 50.4 million tons of fly ash but only 23.2 million tons had been used in various applications [2]. Fly ash which is mostly composed of silica and alumina has the potential to be used for several purposes.

One of the ways that have been introduced by researchers to reduce the amount of fly ash in the dumping areas is by converting it into another form of material called geopolymer. The geopolymer paste synthesis has two the main requirements to be able to react. First, it should have rich material source of Silicon (Si) and Aluminium (Al). Second, it has the ability to react with alkali activators such as Sodium Hydroxide or Potassium Hydroxide [3]. To achieve the same strength as mortars made of cement, geopolymer pastes need be heated at elevated temperatures within a particular time.

Alkaline activator is a substance or compound causing a substance or other element to react. In the production of fly ash geopolymer paste, the activator used is a hydrated alkaline element like potassium hydroxide (KOH). The activator is needed for the polymerization reaction of Alumina and Silicate monomers. As an activator, Alkali will dissolve precursors into monomers (SiO₄) and (AlO₄). During the curing process, the monomers (SiO₄) and (AlO₄) condense and form a cross-linked three-dimensional polymer network. Alkaline ions also act as charge neutralizers for each tetrahedron alumina

molecule (AlO_4). Alkaline Activators are classified into six classifications: (1) Caustic alkalis: MOH , (2) Non-silicate weak acid salts: M_2CO_3 , M_2SO_3 , M_3PO_4 , MF and others. (3) Silicates: $\text{M}_2\text{O} \cdot n\text{SiO}_2$, (4) Aluminates: $\text{M}_2\text{O} \cdot n\text{Al}_2\text{O}_3$, (5) Alumina-silicates: $\text{M}_2\text{O} \cdot n\text{Al}_2\text{O}_3 \cdot (2-6) \text{SiO}_2$, and (6) Non-silicate strong acid salts: M_2SO_4 [4].

Referring to ASTM C 618-05 [5], fly ash is divided into two types, namely class F and class C. In class F fly ash, its minimum content of SiO_2 , Al_2O_3 and Fe_2O_3 is 70%, while for class C it is 50% - 70%. In addition, Nataraja [23] stated that there were three large groups of fly ash called low calcium ash, medium calcium, and high calcium fly ash. It was proved that the use of low calcium fly ash produces very low strength development at early ages, and no significant pozzolonic activity, while the use of calcium medium fly ash produces a significant improvement in strength after 90 to 365 days, decreases in average diameter diameter in concrete, excellent durability against freezing and thawing. Furthermore, most of the available scientific literature discusses making geopolymers using class F fly ash (low CaO levels) [7-14]. Therefore, it is important to conduct research on the manufacture of geopolymers using medium calcium fly ash.

This research uses potassium hydroxide to activate medium calcium fly ash with curing process through heating in an oven at a temperature of $100 \pm 5^\circ\text{C}$ for 2, 4 and 7 hours. The test was conducted to evaluate the compressive strength development of medium calcium fly ash based geopolymer paste.

2. Material and Methods

2.1. Fly Ash

Fly ash is the result of the combustion of coal which forms powder. The particles size of fly ash have a diameter of 1-150 micrometers or pass sieve of 45-micrometre. In general, the main chemical compounds of fly ash silica (SiO_2), alumina (Al_2O_3), ferric oxide (Fe_2O_3) and calcium oxide (CaO). The CaO content of fly ash for class N and F is relatively smaller than class C. In Class C, the content of CaO is more than 20% [15]. Refer to the results of the study [6] classification for low calcium fly ash containing 53-63% SiO_2 , 0.6% CaO , medium calcium fly ash containing 39-52% SiO_2 , 3-18% CaO , and for high calcium fly ash containing 30-40% SiO_2 , 16-29% CaO . Table 2 shows the chemical content of fly ash based on XRF test that the CaO content of fly ash is 12.74% and SiO_2 is 34.63%, so this study determined that the fly ash used was medium calcium fly ash.

Table 1. Physical characteristic of fly ash used in this study.

Physical Characteristic	Result
Density, kg/m^3	2.65
Filter and Hydrometer Analysis, passed sieve No.200	< 30%
Particle size, less than $75 \mu\text{m}$	90%

Table 2. Chemical content of fly ash (% by mass).

Oxides (%)	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	TiO_2	Cr_2O_3	MnO	Fe_2O_3	SrO	BaO	Pr_6O_{11}	Nd_2O_3
	8.10	19.16	34.63	1.80	1.33	12.74	1.26	0.07	0.25	19.96	0.13	0.21	0.05	0.07

2.2. Potassium Hydroxide (KOH)

Several studies have indicated that after being activated at a particular heat temperature over a period, an alkaline activator in the form of potassium hydroxide provides good bond strength to fly ash based-geopolymers. Therefore, the concentration of potassium hydroxide used in this study was 6 M KOH meaning that for every 1 liter (1000 ml) this solution contained 6 moles of KOH (or 336 grams KOH) calculated according to Equation (1).

$$M = \frac{gr}{Mr} \times \frac{1000}{V \text{ ml}} \quad (1)$$

M = Molaritas (mol/L)

gr = gram of solution

Mr = molar mass

V = volume of the solvent (ml)

Before being used as an activator in a geopolymer paste mixture, it takes ± 30 minutes to dissolve KOH using distilled water until the temperature of solution is equal to room temperature then ready to be mixed with fly ash.

2.3. Mix Design of Geopolymer Paste

The composition of geopolymer paste was designed using fly ash and potassium hydroxide as alkaline activator. The concentration of potassium hydroxide solution is 6 Molar for 1500 gram of fly ash, Geopolymer paste mixture composition is provided in Table 3. Geopolymer paste specimens were heated for 2, 4, and 6 hours under oven temperature of $100 \pm 5^\circ\text{C}$, then they were cured at room temperature for of 3, 7, and 28 days.

Table 3. Geopolymer paste mixture composition.

KOH Concentration (Molar)	KOH (gram solution)	Fly Ash (gram)	Water (gram)
6	174.72	1500	520

2.4. Preparation of Specimens

This research aimed to find out the effect of heating duration of geopolymer paste with a $5 \times 5 \times 5$ cm cube mould. The mixing methods used in this research are as follows:

preparing geopolymer paste samples, Fly ash is mixed in a mixer. An alkaline activator solution is added to the fly ash, and is further mixed for one minute at low speed, followed by another minute at high speed mixing. After casting, fresh paste in a cube mold is left for 24 hours and then placed in an oven maintained at $100 \pm 5^\circ\text{C}$. After heating for 2, 4, and 6 hours in the oven, geopolymer paste is removed, cure specimens for 3, 7, and 28 days in conditions of air curing and then demoted. Work flow tests are performed for each mixture, giving flow measurement results.

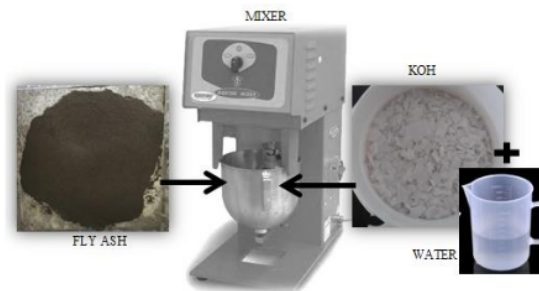




Figure 1. Procedure to make specimens.

2.5. Testing Method

2.5.1. Flow Test

After the mixing process is complete, the flow measurement is continued. Flow measurement is conducted by inserting the fresh mixture into the troum conique in the middle of the flow table. For the first layer, the mixture is put half of the volume of the troum conique then compacted using tamper as much as 20 times the pressure. Then proceed to the second layer. After the mortar mixture is flattened, then the troum conique is lifted slowly and immediately the flow table handle is rotated at a rate of 25 beats within 15 seconds until the mortar base is enlarged. The diameter of the base of the enlarged mortar is then measured and recorded. The required mortar flow is at least 110%. Flow test is tested based on SNI 1972-2008 [16]. Figure 2 shows flow table test setup.

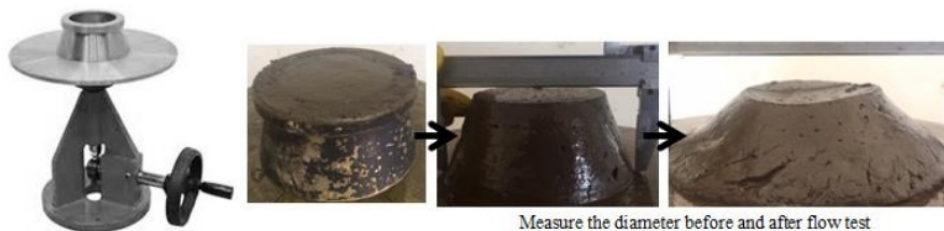


Figure 2. Flow Table Test Setup

2.5.2. Compressive Strength Test

The compressive strength of hardened paste is the maximum compressive strength that can be performed by specimen per unit area. The compressive strength of geopolymer paste has grown with increasing curing age of the mortar. The compressive strength of specimen was calculated by dividing the maximum compressive load obtained by the specimen during testing by the cross-sectional area. Figure 3 shows compressive strength method. Based on SNI 03- 6825-2002 [17], the compressive strength of mortar is calculated according to Equation (2).

$$f'c = \frac{P}{A} \quad (2)$$

$f'c$ = Compressive strength of hardened paste (N/mm²)

P = Ultimate Load (N)

A = Cross-sectional area (mm²)

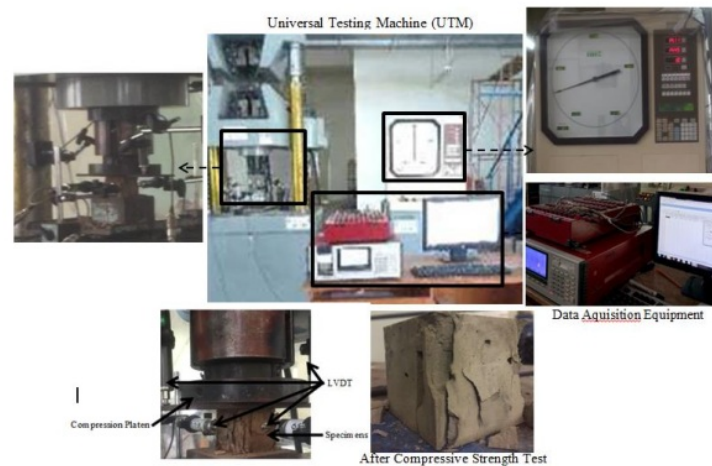


Figure 3. Compressive Strength Method.

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3. Results and discussion

3.1. Slump flow

The results show the geopolymer paste mixture has a slump flow value of 110%. This value indicates that geopolymer has a good level of workability and all materials of geopolymer paste can be bond properly without segregation.

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3.2. Compressive strength

Figure 4 shows compressive strength of the hardened geopolymer paste made with medium calcium fly ash and potassium hydroxide. Visual observation of all specimens was carried out before conducted the compressive strength test. It was seen that all specimens made from medium calcium fly ash and KOH had no hair cracks. The geopolymerization process of the test specimens made with medium calcium fly ash and KOH went well, where from the curing age of 3 days to 28 days no cracking occurred. This shows that CaO content of fly ash 12.74% did not cause hair cracks after being heated at a temperature of $100 \pm 5^\circ\text{C}$ with a heating duration of 2, 4, and 6 hours.

The compressive strength test results show the maximum compressive strength of geopolymer paste made with medium calcium fly ash and potassium hydroxide activators continuing to increase from the curing time of 3 days to 28 days. The second factor that increases the compressive strength of geopolymer paste is the heating duration, which also increases the compressive. The results show the compressive strength of fly ash-based geopolymer paste influenced by curing and heating time. The highest compressive strength value was at 28 days were at heating time 2 hours, 4 hours, and 6 hours attained 3.82, 5.84, and 7.33 N/mm² respectively.

From the compressive strength test using UTM that have been conducted, as shown at Figure 4, the increase in compressive strength of geopolymer paste was directly proportional to the addition of heating time of 2 hours, 4 hours, and 6 hours.

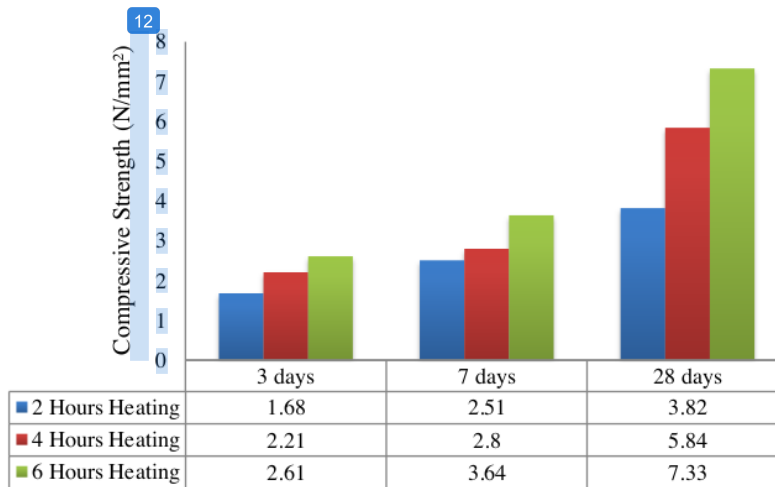


Figure 4. Compressive strength of geopolimer.

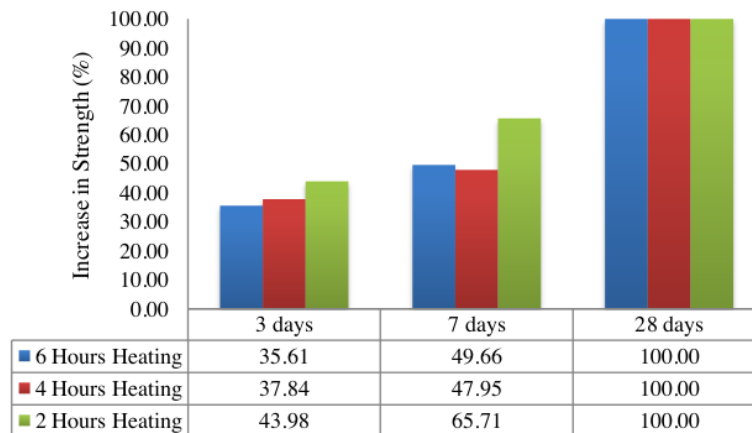


Figure 5. Percentage increase in strength.

The percentage increase in strength from the curing time of 3, 7, and 28 days based on the heating duration is shown graphically in Figure 5. For heating duration 2 hours, the specimens aged 3 days and 7 days have compressive strengths 43.98% and 65.71% of the 28 days compressive strength, respectively. For duration heating 4 hours, specimens aged 3 days and 7 days have compressive strengths 37.84% and 47.95% of the 28 days compressive strength. Whereas for heating duration 6 hours, at the age of 3 days and 7 days have a compressive strength of 35.61% and 49.66% of the 28 days compressive strength, respectively.

This shows that at the duration of 2 and 4 hours, geopolimer mortar does not get enough heat to complete the geopolimer reaction at a younger age, therefore the reaction occurs at a slower age at a slower rate. For specimens that are heated for 6 hours, the reaction continues to increase from 3, 7 days to 28 days.

seen that the compressive strength value increases with increasing curing time. Lack of soluble silicate which was not used as an additional solution in this paste showed that the fly ash was not reactive enough to induce more silicaalumino bound during geopolymerization in early strength [17]. However, the results proved that the medium calcium-fly ash was a potential material as a low-carbon binder.

4. Concluding remarks

Medium calcium fly ash can be mixed with activator to produce geopolymer paste, which compressive strength test results with a curing temperature of $100 \pm 5^\circ\text{C}$ presented the compressive strength continuing to increase along with the increasing duration of heating and curing time. The compressive strength of the geopolymer paste increases with the addition of the heating duration from 2 hours, 4 hours to 6 hours. At the age of 3 days to 28 days the highest compressive strength of the fly ash-based geopolymer paste was 6 hours of heating duration, respectively 2.61, 3.64, and 7.33 N / mm². with a heating duration of 2 hours, the compressive strength of geopolymer mortar increases up to 49.4% for 7 days from 3 days, and 127.3% for 28 days from 3 days. It is showed that eventhough the fly ash required heat at a certain time to develop the paste strength, it was a promising material as a geopolymer binder to contribute in infrastructures .

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