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Research paper



Effect of Heat Curing Temperatures on Fly Ash-Based Geopolymer Concrete

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Abstract

The geopolymer concrete (GC) is a cement replaced by aluminosilicate material combined with alkaline solution to produce strong binding property and replaced function of ordinary Portland cement. The purpose of this research was to determine the relationship between alkali solution concentrations (8 M, 10 M and 12 M), heat curing temperature and heat curing duration as the influencing parameters of concrete strengths. Geopolymer sample was prepared under different target heating temperatures which were room temperature and heat curing ranges of 60 °C to 100 °C for a duration curing period of 24 hours in the oven. Then the specimens were left in a room temperature until the testing day. The mechanical properties of geopolymer concrete were determined with the compressive test. The comparison was made with concrete control specimens that cured in ambient temperature. The results were determined from 7 days specimens after the curing process. The results show that the compressive strength of GCs under heat curing condition was developed quickly when there is an increase in temperature. It can be concluded that the temperature plays an important role in accelerating the GC strength increment compared to curing in ambient temperature.

Keywords: *Curing temperature; , fly ash; geopolymer; mechanical properties; molarity.*

1. Introduction

Ordinary Portland Cement (OPC) concrete has a resistance agent good strength at the fire. However, exposure in high temperature will affect the strength of OPC concrete due to changes in chemical and physical of concrete [1]. The geopolymer concrete required heat to increase the compressive strength [2]. Geopolymer concrete does not contain OPC and the powder binder which used in industrial waste has low calcium and rich with aluminosilicate source. Geopolymer concrete is polymer-inorganic material that was synthesized with sodium silicate and sodium hydroxide solutions to produce good binder paste [3]. Geopolymer concrete has difference from alkali activated cementations composites (AACC) where geopolymer will react with alkali solution as activated material to form binder. While, AACC when it contents a lot amount of calcium materials to react with water such as fly ash combined with OPC will improve concrete properties.

The alkali activator solution plays an important role in the concrete mixture of geopolymers to dissolute of Si and Al oxides [5]. The alkali react of fly ash is a chemical process also known as polymerization process. The reaction process result was inorganic material polymer formed by chains of hydrated aluminosilicates, where a negative charge ion was balanced by alkaline ions [6]. The concentration of alkaline activator influenced the workability and mechanical behaviour of concrete give an advantage to geopolymer mortar [7].

The hardening of geopolymer concrete depends on a factor of curing temperature and time and will significantly influence the mechanical strength of fly ash-based geopolymer concrete. Previously, researchers had presented that the development of compressive strength of geopolymer concrete was influenced by concentration of NaOH, type of alkaline activator, curing time and curing temperature [4, 5]. The scanning electron microscopy (SEM) test was conducted to observe the microstructure of GC with elevated curing temperature and low alkaline molarity together with large proportions of fly ash still did not completely reacted chemically with alkaline solution. The chemical reaction also knows as a polymerization process to represent this binder. While, high concentration alkali exists pores and crack in matrix structure geopolymer which could limit the compressive strength of geopolymer concrete [8]. The geopolymer concrete curing with that high curing temperature and long curing time resulted in higher compressive strength [9]. However, the increase in curing time will not significantly increase the compressive strength after 48 hours [10].

The fly ash geopolymer shows that low reactivity at ambient curing condition and long-term to achieve target strength. However, in tropical climates with an average temperature of 30° C, geopolymer concrete strength could reach close to OPC concrete strength when it was cured in ambient temperature. The objective of this investigation is to experimentally examine the performance of fly ash-based geopolymer concrete under elevated temperatures compared with OPC concrete.

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2. Experimental work

2.1. Materials characterization

Two types of local aggregate used were coarse and fine aggregates, in saturated surface dry condition. The coarse aggregate consist of crushed basalt aggregates which were produced from a local site. The fine aggregates from the river having a water absorption percentage of 0.18%, specific gravity of 2.61 and fineness modulus of 2.7. The aggregate size distribution for coarse aggregate range from 20 mm to 10 mm and the maximum size distribution of the sand is 4.75 mm was measured in accordance with ASTM C136 (2001).
The fly ash was provided by Energy Venture Power Station, Kapar, Selangor, Malaysia and it is equivalent to ASTM C 618. The fly ash was made by product of coal burning which was used to generate electricity that was supplied by thermal power plant. The fly ash used in this research was class F based on given standards by ASTM C 618 to classify the fly ash.
The fly ash chemical composition was determined by X-ray fluorescence (XRF) as listed in Table 1. The particle size of fly ash retained on 45 µm sieve was reported as 71.21% based on research by MD. Maruf Hossain (2015). In general, the main composite for fly ash is the oxides of silicon (SiO_2), aluminium (Al_2O_3), iron (Fe_2O_3), and calcium (CaO), where magnesium, potassium, sodium, titanium, and sulphur are present in less amount in fly ash. The content of unburnt carbon remaining in the ash was measured by loss on ignition (LOI).
The ratio for silicon oxide (SiO_2) and aluminium oxide (Al_2O_3) by mass should be in the range of 2.0 to 3.5 to affecting the compressive strength geopolymer concrete [11]. The ration value of SiO_2 / Al_2O_3 were 2.7, this indicates that fly ash has the properties of cementitious and pozolan which increase the compressive strength. If, the properties of cementitious less than 2.0 leads to lower high compressive strength and will be hydrated when exposed to moisture.
Generally, a combination of sodium silicate (Na_2SiO_3) and sodium hydroxide solution (NaOH) were used as activators of silica and aluminium in the source material (fly ash) [12]. Na_2SiO_3 was in the form of a solution from factory and NaOH in pellet form with 97% purity, supplied by R&M Chemicals, Malaysia. In order to make sodium hydroxide solution, sodium hydroxide pallets were dissolved in distilled water at 24 hours duration.

Table 1: The Chemical Composite of Fly Ash

Chemical Properties	Percentage (%)
SiO_2	59.92
Al_2O_3	22.39
Fe_2O_3	5.97
TiO_2	1.42
CaO	3.87
Na_2O	0.18
K_2O	1.63
P_2O_5	0.73
SO_3	0.61
MnO	0.16
MgO	0.67
LOI	3.33

2.2. Method of mixing

A solution of sodium hydroxides (NaOH) was prepared separately. To prepare NaOH solution, NaOH pallets was dissolved in one litre of distilled water for three different concentration of NaOH. The solution was left for 24 hours before mixing it with sodium silicate (Na_2SiO_3). The activator preparation was made by adding Na_2SiO_3 solution into the NaOH solution and then stirred manually for 3 to 5 minutes. Alkaline activator with the combination of NaOH and Na_2SiO_3 was prepared just before mixing it with fly ash.
The ratio of fly ash/alkaline activator and Na_2SiO_3/NaOH is 1.5 and 2.5 respectively and were fixed for all mixture. The ratio used was based on previous researcher finding [18]. The fly ash and alkaline activator were mixed together in the mixer until the homogeneous paste was obtained. Superplasticizer with a dosage of 1.5% by mass of the fly ash was added into geopolymer paste mixture to improve the workability geopolymer concrete.
Table 2 shows the mixture proportions of the geopolymer concrete. The mixture proportion of geopolymer concrete contains coarse aggregate, fine aggregate, fly ash, sodium silicate soluteon and sodium hydroxide (NaOH) solution and water is added if necessary. All sample used same mixture with elevated of temperature for curing process. The geopolymer concrete prepared by 60 specimens cube and each of 18 specimens of cylinder and beam for tensile were examined. All material quantities were kept constant to avoid the effects of replacement of FA on the compressive strength. The minimal required amount of extra water was added into the mixing to achieve a suitable workability.
The curing mode condition for all specimen of geopolymer concrete and normal concrete was used two (2) types of curing condition and listed in Table 3.

Table 2: Mix Design Proportions

Materials	GPC (Kg/m³)
Fly ash	639.4
Fine agg.	639.4
Coarse agg.	959.4
NaOH	121.8
Na_2SiO_3	304.5
SP	25.6
Water(if)	-

Table 3: Curing Conditions of Sample

Curing Condition	Temperature	Time/Days
GPC	Room temperature	28 days
	60 °C, 70 °C, 80 °C, 100 °C, Room tempt.	1 days, 28 days

2.3. Casting and curing

The fresh geopolymer concrete was cast in 150 mm x 150 mm x 150 mm cube mould and remould after 24 hours of casting. Specimen for the tensile and flexural test was cast in cylinder size 100 mm x 200 mm and beam size 100 mm x 100 mm x 500 mm. The moulds were opened according to standard practice by ASTM C192/C 192M-02. The specimens are cured in room temperature condition until testing day and half of the sample heat cured condition at 60 °C, 70 °C, 80 °C and 100 °C temperature for 24-hour duration. After removing the specimen from the oven the specimens were maintained at ambient temperature: 26ºC. For ambient curing conditions, specimens were placed in the shade outside the laboratory. Meanwhile, the specimens cured at heat condition for 24 hours and ready to be released at ambient condition and allowed to room temperature until test day. There were 90 specimens of geopolymer concrete was prepared for testing various mode curing condition to determine the mechanical strength.

2.4. Testing

The compressive strength test was performed on geopolymer mortar according to ASTM C109 by using mechanical testing. The specimens were loaded with 10 KN and then utilized with a speed rate of 1 KN/min. Before testing, the top surface of cube mortar specimens was sanded to be flat and parallel. The compressive strength value was determined by the average of 3 specimens testing for each different types of mixture. The compressive strength for paste was being tested for 7 and 28 days.
To observe the effect of temperature curing on fly ash geopolymer concrete specimens, the 24 hours geopolymer concrete specimens was placed under heat curing conditions. After 24 hours, the specimens was taken out from the oven and de-mould for curing under an ambient condition with 26 ºC room temperature until the days of the test. The experiment results of fly ash geopolymer concrete are presented in Table 4.

Table 4: Effect of Duration and Temperature on Compressive Strength

Sample	Compressive strength, fc (MPa)		
	Temperature	7 -D	28 - D
GPC	Room tempts.	18.6	31.2
	60 °C	19.9	37.4
	70 °C	31.6	36.0
	80 °C	32.4	51.2
	100 °C	19.2	35.0

3. Result and discussion

3.1. Effect of alkaline activator solution and concentration

The concentration variation in NaOH between 8 M to 12 M had effects on the compressive strength of fly ash geopolymer mortar. Figure 1 shows the results of compressive strength. Fly ash-based geopolymer mortar with concentration NaOH 12 M cured in ambient conditions provide the highest compressive strength with the value of 32.10 MPa at 28 days. This value was 35% greater than the mix with 8 M and 27% greater than the mix with 10 M at ages of curing at 28 days. The compressive strength was improved with the increase in the molar concentration of sodium hydroxide solution. As it can be seen in figure 1, the optimum molarity for development of geopolymer concrete was at 12 M NaOH solution where it shows highest compressive strength than the other molarity. Besides, development strength of geopolymer concrete under ambient curing was slowly increased with time and achieved target strength at ages 28-day, similar to OPC concrete.
A higher concentration in term of molarity of NaOH influenced the geopolymer mortar compressive strength results [13]. Furthermore, the strength development was closely related to the NaOH solution concentration used in concrete hydration products [6]. High concentration will be effect on compressive strength of geopolymer concrete because the ions OH- will high dissolution fly ash chemical chain. The fly ash geopolymer concrete mixed with a higher concentration of sodium hydroxide will become more cohesive but reduced flowbility. This will improved workability of fresh concrete.

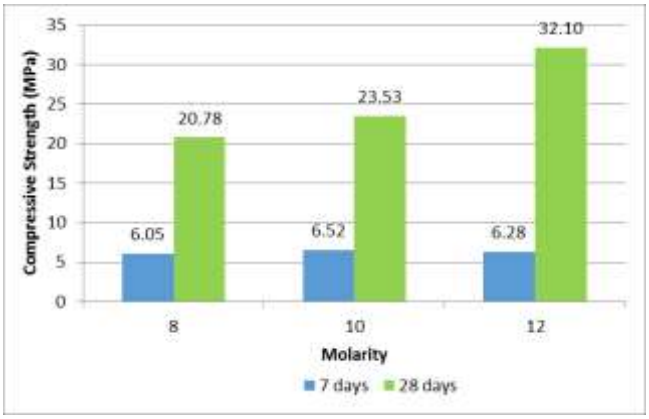


Fig. 1: Effect of Molarity of Alkali Solution on Compressive Strength of Geopolymer Concrete. All sample curing under ambient condition.

3.2. Effect of elevated curing temperature on compressive strength and curing condition

Geopolymer concrete still not hardened yet after 1 day of curing in room temperature and it took 3 days before specimens can be remoulded. Fly ash required high temperature for the activation of fly ash with an alkaline solution. Therefore, four elevated temperatures

of curing were to be compared with room temperature curing condition. According to a previous study [11], higher elevated temperature resulted in higher rate of the polymerization process, which accelerates hardening of geopolymer concrete. Figure 2 shows that at 7 days of curing, the compressive strength of specimens cured at 80 °C was approximately similar to the specimens which cured at 28 days but in the room temperature. The specimens cured at 80 °C have a compressive strength value of 32.40 MPa. The lower strength value about 50% in the specimens which cured in ambient temperature was caused by a slow polymerization process in low temperature. The early condition curing period within the first week from mixing to casting process will result in a lower strength of fly ash geopolymer concrete caused by low chemical reaction activity and after 28 days of curing, the strength development started to active and stable [15].

In ambient curing condition, the specimens' strength increased steadily with the times up to days 28 where the specimens achieved their target strength. It can be observed in figure 2, the compressive strength was increasing up to the temperature of 80 °C but then at 100 °C, the strength decrease. A similar result was found in specimens cured in ambient temperature. It proved that at very high temperature, geopolymer phase integrity will be affected. The compressive strength at 28 days for all specimens whether curing in ambient temperature or elevated temperature, approximately achieved similar compressive strength except in the specimens curing at 80 °C. the Hou et al. [14] reported that the heat curing temperature was influencing the development strength of geopolymer concrete.
Figure 2 shows that the specimens concrete strength of heat curing at day 3 was approximately similar to the specimens cured in ambient condition at day 28. However, after heat curing specimens were exposed to the ambient condition, the strength decreases about 35% at ages 7 days. The strength starts increasing again up to 28 days curing and achieved its target strength. This proved that the curing conditions were among the important factor that influenced the process of the hardening and strength development of geopolymer concrete [14]. The temperature using as curing condition on geopolymer concrete to accelerate the geopolymerization process of chemical reaction and process of hardening concrete, but the temperature not only the main factor involved in strength development of geopolymer concrete [6].

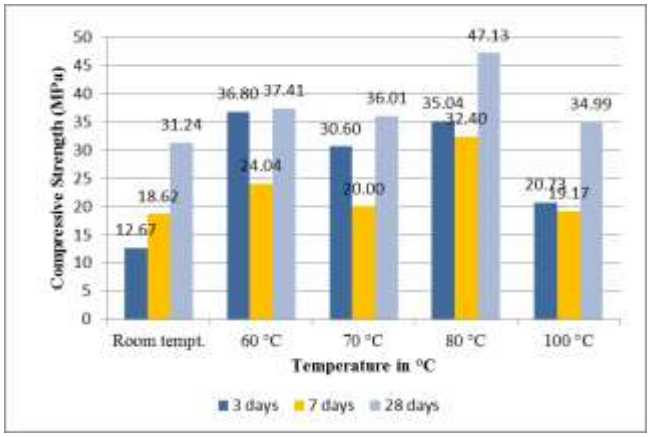


Fig. 2: Effect of Elevated Curing Temperature on Compressive Strength of Geopolymer Concrete at ages 3 days, 7 days and 28 days.

3.3 Conclusion

The alkaline activator molarity and the curing condition play an important role in the development of geopolymer concrete. The study can be concluded as follows,

a) Fly ash-based geopolymer concrete with 12 M NaOH concentration obtained higher compressive strength in both curing conditions
b) Curing temperature and duration is important in the activation of geopolymer concrete to produces higher compressive strength. The optimum curing temperature to be found is at 80 °C. According to research by Fareed Ahmad Memon et al. [19], compressive strength of geopolymer concrete decreased with the increase the temperature beyond 80 °C caused a negative effect on the physical properties of the geopolymer.
c) The rate gain of strength is slow at ambient temperature but consistent. In the heat curing condition, compressive strength develops rapidly at day 3; however, start to decrease at day 7 and then it starts to increase again up to day 28.

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