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Effect of Curing Conditions on Compressive Strength of FA-POFA-based Geopolymer Mortar

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Abstract. This paper presents the effect of curing conditions on geopolymer mortar prepared from the blend of palm oil fuel ash (POFA) and fly ash (FA) as full replacement of cement with a combination of sodium silicate and sodium hydroxide solution used as alkaline liquid. The density and strength of the geopolymer mortars with FA:POFA mass ratios of 70:30 together with sodium silicate solution-to-sodium hydroxide solution by mass is 2.5, are investigated. The concentration of alkaline solution used is 14 Molar. Tests were carried out on 70x70x70 mm cube geopolymer mortar specimens. The effects of curing conditions including the duration of heat curing, temperature of heat curing and delay time before heat curing on compressive strength of mortar are examined. The results revealed that as the temperature increased the compressive strength of geopolymer mortar are increases, the delay time before heat curing increased, the compressive strength of geopolymer mortar reduced and suitable duration for heat curing is within one to two days.

1. Introduction

The manufacturing of Portland cement is an energy intensive process and releases a large amount of green house gas, i.e. carbon dioxide (CO₂) into the atmosphere. Geopolymer technology is one of the new technologies attempting to reduce the use of Portland cement in concrete. Geopolymer concrete is a developed technology, where most of research work has been done using low-calcium fly ash as source material, because Si-Al minerals present in low-calcium fly ash trigger a substantially fast chemical reaction with alkaline solutions, consequently produce a three dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds [1, 2]. Malaysia is the largest producer of palm oil and palm products in the world., palm oil shell and husk, by-products, are used as fuel in a boiler in the palm oil mill to produce steam for electricity generating plant and palm oil extraction process. Although the increased use of palm oil fuel ash (POFA) is evident, much of the POFA is disposed in land filling. Palm oil shell and husk, when burnt, is found to contain a high percentage of silica which



is one of the main constituents in producing geopolymer. It is an agricultural waste and one of the pozzolan materials as proven by previous research and has been successfully used in the improvement of strength and durability of concrete [3-5]. Although large amount of POFA is being used in landfilling, there is still huge volume of POFA which is not fully utilized. It is estimated that total volume of solid waste generated in palm oil fuel ash industry is about 10 million a year.

Previous studies have shown the successful use of FA as a source material in manufacturing of geopolymer concrete or mortar in terms of high strength and durability performance because of low-calcium and high silica to alumina ratio in FA [6]. Manufacturing of geopolymer mortar using FA-POFA blended ash as an alternate for cement is being examined in the present study. As a relatively new construction material, there is little information available on the properties of geopolymer concrete for structural applications. Since the preparation and hardening process of geopolymer concrete is different from those of conventional concrete, the properties of geopolymer concrete entirely depend on the source material to be used. There is less information or technical data available on the development of geopolymer mortar using FA-POFA blended ash as source material. The present study deals with the manufacturing of geopolymer mortar using FA-POFA blended ash along with a combination of sodium silicate and sodium hydroxide solution used as alkaline liquid, and intends to investigate the effect of curing condition on the behaviour of this new material in terms of strength properties. The knowledge would be instrumental and lay some foundations for the future research in this field.

2. Materials and Methods

2.1. Materials

Lignite FA from Kapar power station, Selangor, Malaysia was used. The FA has a mean particle size of 45 μ m, a specific gravity of 2.2, and a percentage retained 92.9%. POFA was obtained from burning of palm oil shell and husk (in equal volume) from a Kluang, Johor. The obtained ashes were greyish and the losses on ignitions (LOI) were 0.112% for FA and 20.9% for POFA, respectively. The chemical compositions of FA and POFA are given in Table 1. The typical scanning electron microscope (SEM) photos of surfaces and those of FA are spherical as shown in Figure 1. To activate the blended ash, commercial grade sodium hydroxide (NaOH) and sodium silicate solutions were used as alkaline activator. Local crushed granite sand with a specific gravity of 2.62 was used for making mortar. The mass of NaOH solids in a solution varies depending on the concentration of the solution. The range of NaOH concentration used in this research was 14 molar where it yielded a higher compressive strength [7,8]. In order to improve the workability, a high range water reducer super plasticizer and extra water were added to the mixture.

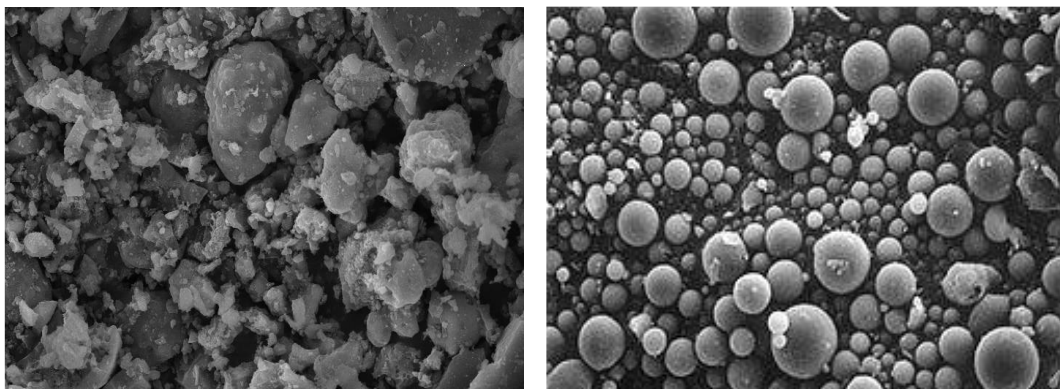


Figure 1. Morphology of POFA and FA using SEM

Table 1. Chemical composition of POFA and FA

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	P ₂ O ₅	LOI
FA	46.7	35.9	5.0	3.92	0.8	0.6	0.5	0.4	0.112
POFA	53.5	1.9	1.1	8.3	4.1	1.3	6.5	2.4	20.9

2.2. Preparation of geopolymer mortar specimens

All geopolymer mortar specimens were prepared with sand to blended ash ratio of 3:1, whereby the sand was prepared to saturated surface dry condition. The concentration of sodium hydroxide (NaOH) to solution was 14 molar. The ratio (by mass) of sodium silicate to sodium hydroxide is 2.5. The blended ash and the aggregates were first mixed together dry in 80-litre capacity pan mixer to about three minutes. The alkaline liquid was mixed with super plasticizer and the extra water was added to maintain the workability of geopolymer mortars. The liquid component of the mixture was then added to the dry materials and mixed for another four minutes. The mixing was carried out in a room temperature of approximately 28°C. The mix proportion are given in Table 2. The flows of geopolymer mortars were determined in accordance with ASTM C124 [9] and were fixed in the range of 130 ± 5. The test specimens 70x70x70 mm cubes were prepared. The specimens were compacted in three layers with tamping as described in ASTM C109 [10]. Additional vibration of about 10s was applied using vibrating table. The specimens were wrapped with vinyl sheets to protect moisture loss and kept in the room temperature for ambient curing and placed in the oven for heat curing.

Table 2. Mix proportion of geopolymer concrete (kg/m³)

FA	POFA	NaOH	Sodium silicate	Sand	Super plasticizer	Liquid /(FA+POFA)
369	158	68	169	1586	5.27	0.45

2.3. Curing conditions

The method of curing the geopolymer mortars is different from the ordinary Portland cement mortars. There are two types of curing conditions which are heat curing for 24 hours (90°C and 60°C) and room temperature (28°C) curing. After vibrating process, the moulds with mortar specimens were wrapped with plastic sheet to prevent moisture loss. The specimens were then cured until the testing ages. After that the specimens were left in the laboratory to cool down. Demoulding process was done the next day and then kept in the room temperature, 28°C. The compressive strengths of mortar were tested at the age of 7 days to determine the strength development of geopolymer mortar curing in heat curing and room temperature curing. The reported strengths were the averages of the set of three specimens for each type.

The water curing was not helpful in geopolymer mortars because in geopolymerization process, water is just for increasing the workability only [9]. The geopolymerization shows that water was released during the chemical reaction during geopolymers form. The water expelled during the curing process left behind discontinuous nano-pores in the matrix which would provide benefits to the performance of geopolymer mortar. The effects of curing that in delay time, temperature of heat curing and period of heat curing were investigated. Optimum mixed proportions of geopolymer mortar was used.

Delay time is the time duration before placing the specimen in the oven. In this test, 0, 1, 3, 6, 24, 120, 168 and 216 hours were used. After the delay time, the specimens were placed in the oven at 90°C after casting and wrapping them with plastic sheet to avoid the evaporation of alkaline liquid. In this test, the periods of heat curing were 24, 48 and 72 hours to determine the longer curing effect on geopolymer mortar. The specimens were placed in the oven at 90°C. Geopolymer mortar showed higher compressive strength at 90°C heat curing because of complete polymerization between binders and alkaline solution. The curing temperatures of 60°C and 90°C were assigned. The specimens were placed in the oven for 24 hours after casting and wrapping with plastic sheet.

3. Results and Discussion

3.1. Effect of Duration of Heat Curing on Compressive Strength

The results of heat curing duration were shown in Figure 2. In this test, the specimens were put in the oven at 90°C immediately after casting process with no delay time. It is shown that the compressive strength decreased with the increasing of duration of the heat curing process. The rate of increase in strength was rapid up to 24 hours and beyond that, the strength was only moderate and even lower. The compressive strength for 24 hours curing was 31.46 MPa and slightly increases to 32.1 MPa for 48 hours curing condition. An increase in the duration of heat curing is expected to improve the strength properties of geopolymer mortars. However, it has been suggested that curing for a long duration of time will only weaken the microstructure of the minerals in geopolymer mortars which decreases to 29.67 MPa which similar to the previous study [11] Therefore, heat curing duration should not be more than 24 hours in the practical applications.

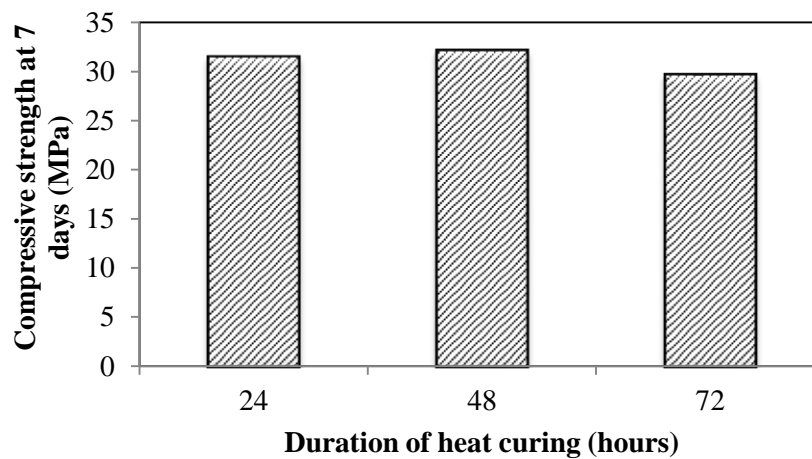


Figure 2. Effect of duration of heat curing on compressive strength of geopolymer mortars

3.2. Effect of Delay Time before Heat Curing on Compressive Strength

This test was done to the optimum mix proportion of (70:30) PFA:POFA and 2.5:1 alkaline solution ratio. The delay time is the time interval before putting the geopolymer mortars into the oven for heat curing process. The time delay was 0, 1, 3, 6, 24, 120, 168 and 216 hours before putting in the oven for 24 hours. The effect of delay time on compressive strength of geopolymer mortars was shown in Figure 3. It is shown that compressive strength without delay time is about 31.5 MPa. When the delay time was 1 hour, the strength was slightly increased to a maximum of 33 MPa. This is because the delay time allows the dissolving of the main ingredients for the alumino-silicate geopolymerization which is alumina and silica. However, increase in the delay time more than 24 hours result in significance change in strength. Furthermore, a slight delay in the start of heat curing has increased the compressive strength of geopolymer mortars. This may be due to the complete geopolymerization that occurs prior to the start of heat curing. In this research, the delay time up to 24 hours does not give much adverse effect thus some delay time before heat application is generally needed in the manufacturing of geopolymer mortars.

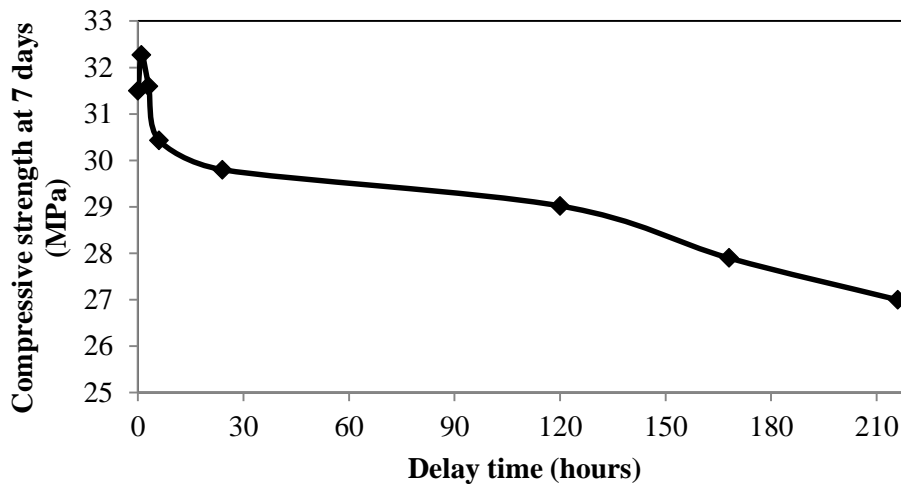


Figure 3. Effect of delay time on compressive strength of geopolymer mortars

3.3. Effect of Curing Temperature on Compressive Strength

The geopolymer mortars were cured at 60°C, 90°C heat curing and ambient temperature as the control specimens. There was no delay time for this test and the duration of oven curing was 24 hours. Figure 4 shows the effects of curing temperature on compressive strength of geopolymer mortar. Heat curing is important for the activation of fly ash, because of the activation barrier that needs to be overcome for the reaction to take place in geopolymerization process. A higher curing temperature will increase the compressive strength of the geopolymer mortar. This is because energy required for dissolution of fly ash is an endothermic process [12]. The compressive strength of geopolymer curing at room temperature is too low compared to geopolymer mortars curing in oven. This is due to the incompletely polymerization between alkaline solution and the blended ash in early age. Besides, heat curing was also required to produce a fast geopolymerization process to achieve an acceptable strength within very short periods where it is one of the important conditions for the synthesis of geopolymers. [13-15].

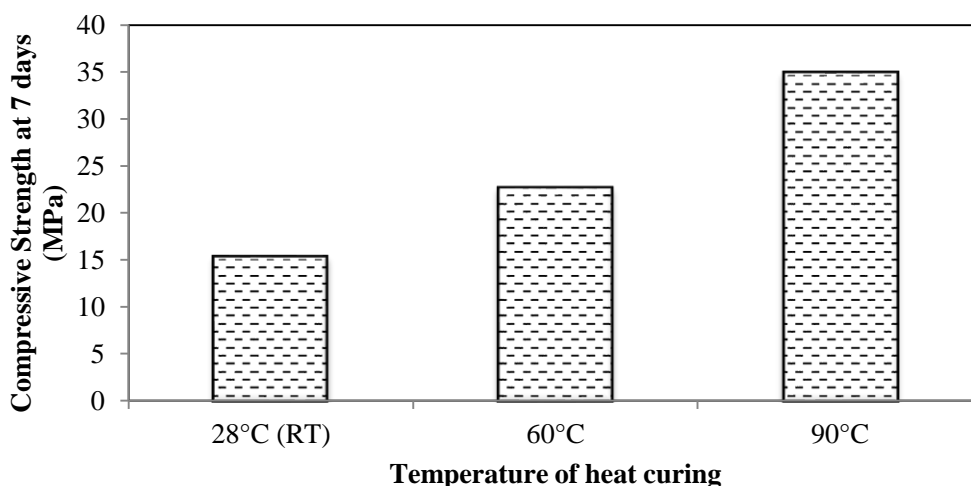


Figure 4. Effect temperature of heat curing on compressive strength of geopolymer mortars

However, the samples normally experience the loss of moisture content with high curing temperature whereby geopolymerization required the presence of moisture to achieve a good

compressive strength. Therefore, based on previous research, it is reported that curing temperature more than 90°C reduced the compressive strength of geopolymer mortars. This is because that the small specimen with high surface to volume ratio is easier to the loss of moisture in heat curing process compare to large specimen. It is shows that when the curing temperature is higher than 90°C, the specimen would experience moisture loss and would result in deterioration of strength. Therefore, 90°C oven curing can be considered as the suitable curing condition that give high strength to geopolymer mortars. Besides, when the geopolymer cured at high temperature, the samples do not have enough moisture in order to develop better strength [15].

4. Conclusions

Based from this research, it can be concluded that the suitable temperature for heat curing for FA-POFA based geopolymer mortar was 90°C. The results revealed that as the temperature increased the compressive strength of geopolymer mortar are increases, the delay time before heat curing increased, the compressive strength of geopolymer mortar reduced and suitable duration for heat curing is within one to two days. However, higher temperature than 90°C will reduced the compressive strength of FA-POFA based geopolymer mortar. Therefore, the results can be used to design curing condition for FA-POFA based geopolymer mortar.

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