

# Optimization of Alkaline Activator Mixing and Curing Conditions for a Fly Ash-Based Geopolymer Paste System

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Received: June 3, 2015

Accepted: June 19, 2015

Online Published: October 31, 2015

doi:10.5539/mas.v9n12p61

URL: <http://dx.doi.org/10.5539/mas.v9n12p61>

## Abstract

This article reports the strength development and microstructure characteristics of a fly ash (FA) geopolymer system prepared with an alkaline activator consisting of sodium hydroxide (NaOH) solution and liquid sodium silicate ( $\text{Na}_2\text{SiO}_3$ ). The effect of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass mixing ratio on the compressive strength and microstructure characteristics of hardened FA geopolymers at different ages was investigated. The influence of different curing conditions on the strength development of the FA geopolymer was also explored. The experimental results revealed that the alkaline activator prepared with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratio of 1.00 provides sufficient alkalinity to promote the geopolymerization reaction and development of high-strength FA geopolymer material. The scanning electron microscopy (SEM) results showed that the dissolution rates of the FA extremely affected by the content of NaOH solution in the liquid activator. Also, the most effective curing regime was 70 °C for 24 h to produce geopolymers with optimal strength at different aging periods.

**Keywords:** geopolymer, alkaline activator, SEM, curing condition, curing temperature, compressive strength

## 1. Introduction

The production of ordinary Portland cement (OPC) is one of the industries that significantly contribute to carbon dioxide ( $\text{CO}_2$ ) emission during the calcination process of the OPC prime material. Hardjito and Rangan (2005) reported that the production of 1 ton of OPC emits approximately 1 ton of  $\text{CO}_2$  to the atmosphere because of the calcination and fuel combustion, which mainly involves fossil fuel. The demand for new infrastructures and buildings has increased because of the continuous increase in the human population worldwide; thus, the construction technology is facing a huge environmental challenge of developing green construction materials (binders) in addition to OPC. The geopolymer material is a novel green construction material has been proposed to replace OPC. In comparison with OPC, geopolymer materials can reduce  $\text{CO}_2$  emission by 80%–90% (Davidovits, 1999) and exhibit better mechanical and durability properties (Li, Ding & Zhang, 2004; Wallah & Rangan, 2006; Gourley & Johnson, 2005). The geopolymerization technology involves the alkaline activation of typical precursors to form an aluminosilicate gel structure through the polycondensation reaction at low temperatures; these precursors include metakaolin, fly ash (FA), slag, and rice husk ash, which contain abundant silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) (Duxson et al., 2007; Detphan & Chindapasirt, 2009). In this technology, a green geopolymer binder (cement) prepared from waste materials like FA is more beneficial to the environment because it diminishes waste quantities disposed in landfills. FA geopolymer materials exhibit high mechanical properties, low density, low water absorption, negligible shrinkage, and high chemical and fire resistance (Thakur & Ghosh, 2006). Given these properties, FA geopolymer materials are considered as an alternative to OPC particularly because they can reduce  $\text{CO}_2$  emissions by 80% compared with OPC (Hardjito & Rangan, 2005). Previous studies reported that FA geopolymer concrete can achieve compressive strength higher than 60 MPa after thermal curing (Kovalchuk, Fernández-Jiménez & Palomo, 2007; Duxson et al., 2005). This material also presents excellent durability to most aggressive acids (Fernández-Jiménez, Garcia-Lodeiro & Palomo 2007) and can resist sulfate attacks better than OPC mortars in reinforcement steel (Wallah et al., 2005; Song et al., 2005; Fernandez-Jimenez, Palomo & Lopez-Hombrados, 2006).

FA, a by-product of pulverized coal combustion in thermal power plants, is primarily composed of silicon ( $\text{SiO}_2$ ), aluminum ( $\text{Al}_2\text{O}_3$ ), iron ( $\text{Fe}_2\text{O}_3$ ), and calcium (CaO) oxides, with less amounts of magnesium, potassium, sodium,

titanium, and sulfur oxides (Hardjito & Rangan, 2005). Accordingly, a highly alkaline activator is required to dissolve the acidic oxides of FA; therefore the usage of normal water in the activation process of a FA source is inappropriate. The common alkaline activator used to prepare FA geopolymers is a liquid combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) or potassium silicate ( $\text{K}_2\text{SiO}_3$ ) (Kong & Sanjayan, 2008; Kong & Sanjayan, 2008; Rukzon, Chindaprasirt & Mahachai, 2009; Nazari, Riahi & Bagheri, 2012). Many studies used Na-based activators to prepare FA geopolymers because of their economic advantages (Rashad, 2014; Temuujin, van Riessen & MacKenzi, 2010; Nematollahi & Sanjayan, 2014; Ryu et al., 2013). The content and concentration of liquid alkaline activator play a fundamental role in the development of high-strength FA geopolymers at low temperatures (Ryu et al., 2013); these factors also determine the thermal durability or fire resistance of FA geopolymers exposed to temperatures higher than 100 °C (Abdulkareem et al., 2013; Abdulkareem et al., 2014).

According to Glukhovsky, (1981), the mechanism of alkali-activation is composed of conjoined reactions of destruction-condensation, that include the destruction of the prime material like FA into low stable structural units, their interaction with coagulation structures and creation of condensation structures. The authors estimated that the first steps consisting of breakdown of covalent bounds of Si-O-Si and Al-O-Si, which happened when the pH of the alkali solution rises, so those groups are transformed to a colloid phase. Then an accumulation of the destroyed products occurs, which interacts among them to form a coagulated structure, leading in a third phase to the generation of a condensed structure.

Alvarez-Ayuso et al., (2008) reported that the geopolymerization reaction occurs in the alkaline solutions with aluminosilicate oxides and silicates (either solid or liquid) as reactants. In addition, the mechanism involving the dissolution of the aluminum (Al) and silicon (Si) species from the surface of the source material as well as the surface hydration of undisclosed particles. Afterward, the polymerization of active surface groups and soluble species take place to form a gel, generating subsequently a hardened structure.

Thakur & Ghosh, (2009) reported a description for the geopolymerization of FA geopolymers, as the main reaction product in the alkali-activated FA is an alkaline silico-aluminate gel and the  $\text{OH}^-$  ions acts as a reaction catalyst during the activation process, while the alkaline metal ( $\text{Na}^+$ ) acts as a structure-forming element. The structure of the pre-zeolite gel chains Si and Al tetrahedral randomly distributed along the polymeric chains that are cross-linked so as to provide cavities of sufficient size to accommodate the charge balancing hydrated sodium ions (Xu & Deventer, 2000). Furthermore, the development of the compressive strength of the hardened FA geopolymers is attributed to the resulted aluminosilicate gel phase in the geopolymer matrices.

This work aims to evaluate the effect of alkaline activator concentration on the strength development and microstructure of FA geopolymers. The geopolymers were prepared with the selected activator/FA ratio of 0.59, various mixing ratios of the activator solution constituents, and different curing regimes. The prepared geopolymers were used to determine the role of activator constituents in the geopolymerization reaction and strength development of FA geopolymers.

## 2. Experimental Procedure

### 2.1 Source Materials

FA was provided by the Manjung Power Station, Lumut, Perak, Malaysia. FA was stored under shade at ambient temperature and covered by a thin plastic layer to protect from ambient humidity and maintain clean powder. FA was analyzed prior to mixing, and its chemical composition was determined using X-ray fluorescence (Table 1). FA contained high  $\text{Fe}_2\text{O}_3$  (30.13%) and 21.6% CaO, and thus classified as class C according to ASTM C618 (2008). With a specific surface area of 0.463  $\text{m}^2/\text{g}$ , about 90 % of FA presented particle size smaller than 40  $\mu\text{m}$  as indicated in the particle size analysis results of Figure 1. Figure 2 presents the XRD diffractogram of the FA showing that the FA is mainly an amorphous material with the appearance of a typical broad hallow at 16 to 38  $2\theta$ . However, the FA diffractogram is also contains some of crystalline phases of quartz ( $\text{SiO}_2$ ) at 21, 26.6 and 65  $2\theta$ , mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) at 17.1 and 28.3  $2\theta$  and hematite ( $\text{Fe}_2\text{O}_3$ ) at 24, 35 and 41  $2\theta$ .

Table 1. Chemical composition of fly ash using XRF.

Chemical	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{TiO}_2$	CaO	MnO	CuO	$\text{K}_2\text{O}$	$\text{P}_2\text{O}_5$	$\text{SO}_3$	SrO	LOI*
%	26.4	9.25	30.13	3.07	21.6	0.27	0.14	2.58	0.67	1.3	1.57	3.02

\*LOI: loss on ignition.

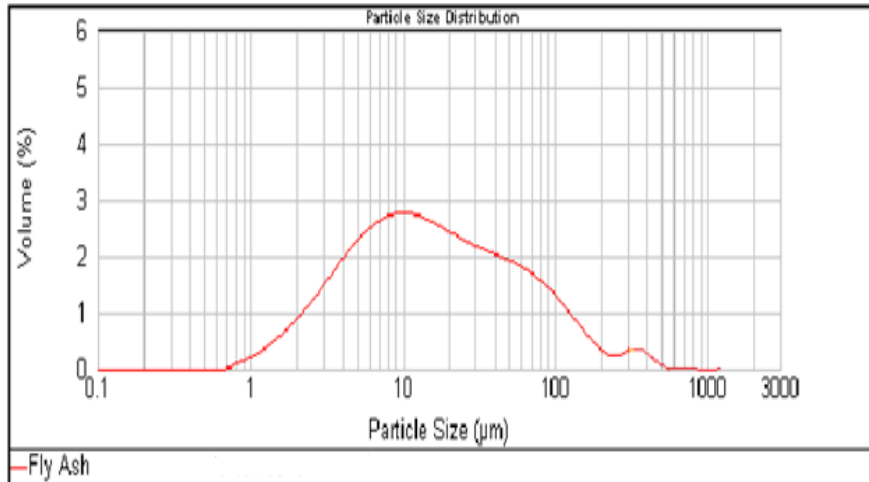


Figure 1. Particle size distribution of the FA

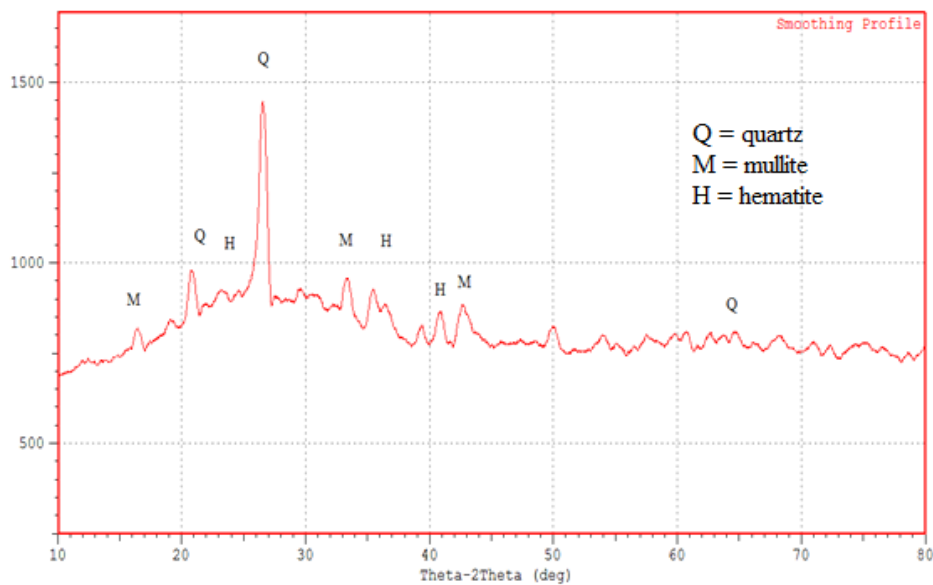


Figure 2. XRD diffraction pattern of the FA.

FA was activated with an alkaline activator, which was prepared by mixing technical-grade  $\text{Na}_2\text{SiO}_3$  (Sigma Chemical Ltd.) and NaOH solution. The chemical composition of  $\text{Na}_2\text{SiO}_3$  was 30.1%  $\text{SiO}_2$ , 9.4%  $\text{Na}_2\text{O}$ , and 60.5%  $\text{H}_2\text{O}$ , with a modulus ratio ( $M_S$ ) equal to 3.2 ( $M_S = \text{SiO}_2/\text{Na}_2\text{O}$ ).

## 2.2 Specimen Preparation

### 2.2.1 Preparation of FA geopolymer Paste with Different Mass Ratios of $\text{Na}_2\text{SiO}_3/\text{NaOH}$

An alkaline activator was prepared by mixing 12 M NaOH solution with  $\text{Na}_2\text{SiO}_3$  at six  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mixing ratios ranging from 0.5 to 3.0 with 0.5 increment to investigate the effect of mass ratio on the strength development of FA geopolymer paste. The mixture proportions of geopolymer pastes are listed in Table 2. Geopolymer paste specimens were synthesized by gradually adding the alkaline activator to dry FA at an activator/FA ratio of 0.59. After 5 min of hand mixing, the fresh homogeneous geopolymers were poured into 50 mm × 50 mm × 50 mm plastic molds and placed on a vibration table for 2 min to remove entrapped air bubbles. The molds were then wrapped using a thin plastic sheet to prevent water evaporation and cured in a furnace at 60 °C for 24 h. After curing, the molds were removed from the furnace and cooled at room temperature before demolding. The specimens were again sealed from ambient conditions and aged at targeted aging periods of 3, 7, and 28 days.

Table 2. Mix proportioning of the geopolymer paste

Mix no.	FA(g)	Activator/FA	Na <sub>2</sub> SiO <sub>3</sub> /NaOH	Na <sub>2</sub> SiO <sub>3</sub> (g)	NaOH(g)
1	450.00	0.59	0.5	88.00	177.00
2	450.00	0.59	1.0	132.75	132.75
3	450.00	0.59	1.5	153.90	102.60
4	450.00	0.59	2.0	177.00	88.00
5	450.00	0.59	2.5	189.64	75.85
6	450.00	0.59	3.0	199.12	66.37

### 2.2.2 Preparation of FA Geopolymer Paste with Different Curing Regimes

Mixture no. 2 (Table 2) was selected to prepare FA geopolymer paste specimens. The specimens were cured using different curing regimes to examine the effect of curing conditions on the strength development of FA geopolymer paste. In the first condition, geopolymer paste specimens (50 mm × 50 mm × 50 mm) were cured at different curing temperatures of 60 °C, 70 °C, 80 °C, and 90 °C for a fixed curing period of 24 h. In the second condition, geopolymer specimens (50 mm × 50 mm × 50 mm) were cured at 70 °C for different curing times of 6, 12, 18, 24, and 30 h. Under both curing conditions, the specimens were wrapped with a plastic sheet to prevent water evaporation during curing and aged for 3, 7, and 28 days.

### 2.3 Compressive Strength Evaluation

The compressive strength of 50 mm × 50 mm × 50 mm specimens was tested according to EN 12390-3 (2009). The compression test was performed using a speed rate of 50 mm/min by using the Shimadzu Universal Testing Machine (Japan) with a maximum loading of 1,000 kN. A minimum of three specimens was tested to evaluate compressive strength.

### 2.4 Scanning Electron Microscopy (SEM) Measurements

The microstructures of the geopolymers were investigated through SEM using JSM-6460 LA Jeol (Japan). The specimen fragments were mounted in an epoxy resin, vacuumed, and coated with a thin platinum layer by using JFC-1600 auto-fine coater Jeol (Japan). The test was conducted using secondary electrons imaging (SEI).

## 3. Results and Discussion

### 3.1 Effect of Na<sub>2</sub>SiO<sub>3</sub>/NaOH Mass Ratio on the Strength Development of FA Geopolymers

The FA geopolymers prepared with various Na<sub>2</sub>SiO<sub>3</sub>/NaOH mass ratios display dissimilar strength developments at 3, 7, and 28 days (Figure 3). The lowest NaOH content is detected from the samples with Na<sub>2</sub>SiO<sub>3</sub>/NaOH mass ratio of 3.0. This ratio produces the lowest strength development among the other ratios at all tested ages and thus presents the lowest geopolymerization reaction. While, at the highest NaOH solution from the samples with a Na<sub>2</sub>SiO<sub>3</sub>/NaOH mass ratio of 0.5, the geopolymer exhibits relatively poor strength development at the tested ages. The strength development in the specimens with Na<sub>2</sub>SiO<sub>3</sub>/NaOH mass ratios of 0.5 and 3.0 implies the complexity of the geopolymerization reaction and the significance of the concentration of the alkaline activator constituents.

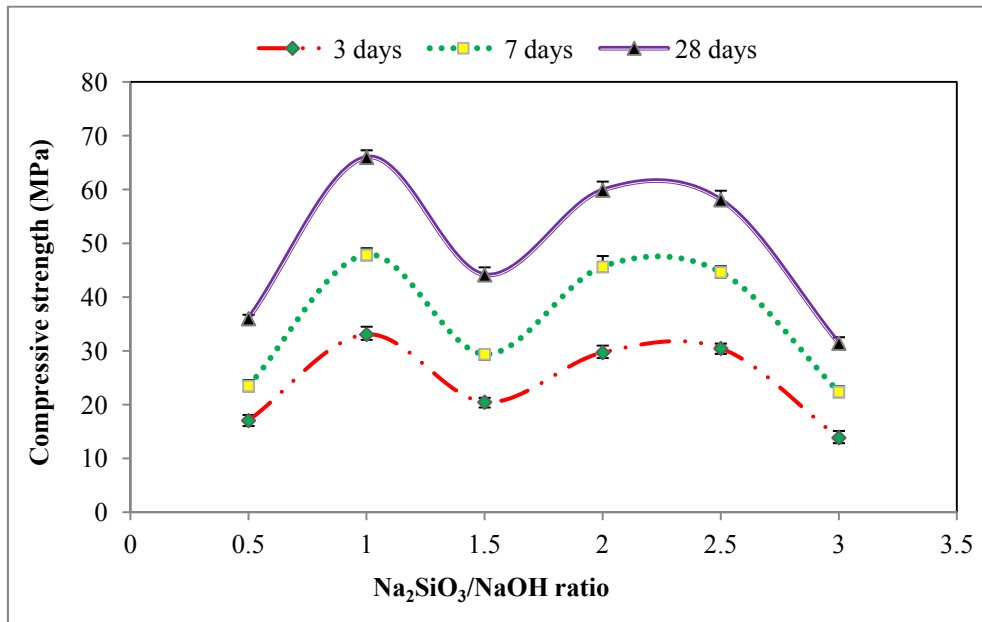


Figure 3. Effect of different  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratios on the compressive strength development of the FA geopolymers paste

Furthermore, the specimen with a  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 1.0 exhibits the highest compressive strength at all ages and thus presents the highest geopolymerization reaction. Chindaprasirt, Chareerat & Sirivivatnanon (2007) reported that the optimum  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio ranges from 0.67 to 1.0 produce FA (class C)-based geopolymers with optimal strength; this finding confirms the results shown in Figure 1. Although the specimen with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 1.5 produces low strength development, the specimens prepared with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratios of 2 and 2.5 produce almost similar strength developments at all tested ages (Figure 3); such strengths were approximately parallel to the strengths obtained with the optimum  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratio of 1.0 particularly at 3 and 7 days. The strength gaining behavior of the specimens with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratios of 2 and 2.5 is advantageous for large-scale production of FA geopolymer binders. Their significance is reflected in terms of economic efficiency because silicate-based activators are cheaper than hydroxide-based compounds.

Moreover, Figure 4 shows the SEM micrographs of the 28 days age FA geopolymers prepared with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratios of 0.5, 1.00, 2.00 and 3.00. At the highest NaOH content of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 0.5 of Figure 4a, the SEM micrograph indicates the high dissolution of the prime material (FA) by the absence of the unreacted FA microsphere similar to the microstructural characteristics of the geopolymer prepared with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 1.0 of Figure 4b. However, the strength development for the  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 1.0 is significantly higher than that of 0.5 and this finding can be attributed to its higher  $\text{Na}_2\text{SiO}_3$  content that entered into the reaction, considering that high content of silicate species may result in high strength development (Palomo, Grutzeck & Blanco, 1999).

Figure 4c shows the SEM micrograph of the geopolymer prepared with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 2.0 indicating the appearance of unreacted FA microsphere portion in the microstructure. The SEM micrographs of Figure 4d shows the high content of unreacted FA microspheres in the microstructure of the FA geopolymer prepared with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 3.0. This result is significantly related to the low dissolution rates of Si and Al species of FA that entered into the reaction caused by the insufficient alkalinity of the alkaline activator prepared at this mixing ratio. The insufficient alkalinity of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 3.0 resulted in the lowest strength development than other ratios as illustrated in Figure 3.

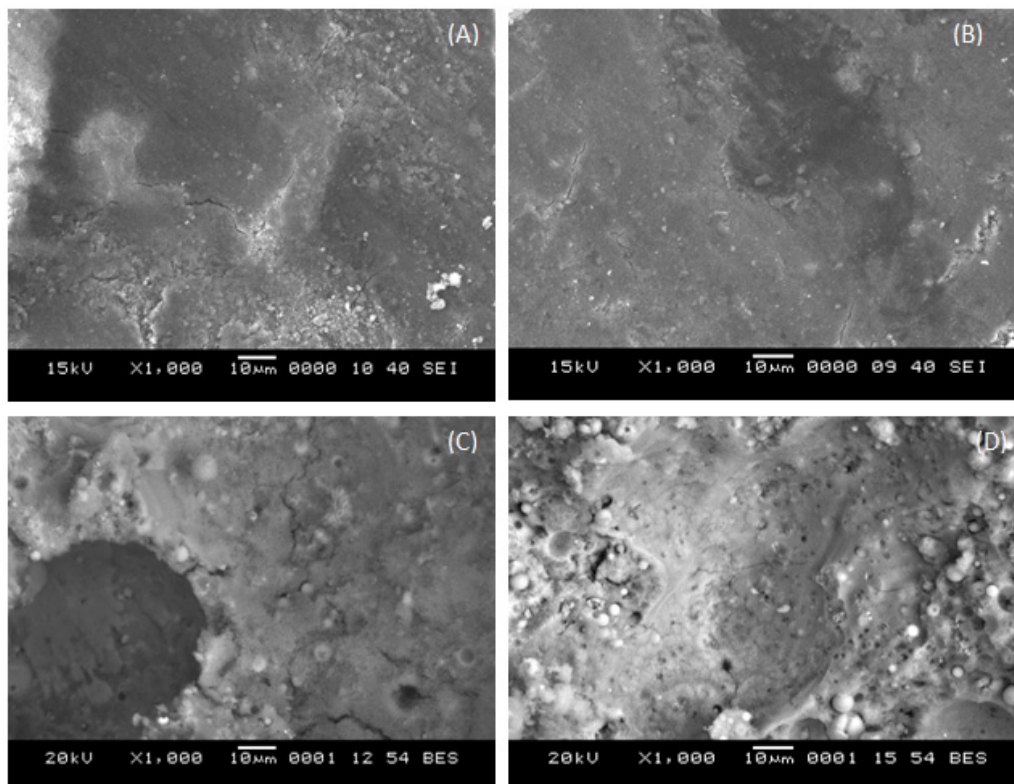


Figure 4. SEM micrographs of the FA-based geopolymers prepared at different  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratios at age of 28 days (A) 0.5, (B) 1.0, (C) 2.0, (D) 3.0.

### 3.2 Effect of Curing Temperature on the Strength Development of FA Geopolymers

The mechanism of the geopolymerization reaction is based on the fast dissolution and polycondensation of Si and Al oxides of FA in the presence of sufficient  $\text{Na}^+$  and  $\text{OH}^-$  species of the alkaline activator to form a three-dimensional polymeric Si-O-Al-O gel (Ryu et al., 2013). The hardening of the geopolymeric material is attributed to water liberation from the fresh aluminosilicate gel structure. Therefore, curing the fresh geopolymers at temperatures higher than the ambient is preferred to accelerate the geopolymerization reaction and decrease the setting time (Bakharev, 2005). Figure 5 shows the compressive strength development of the FA geopolymers pastes cured at different temperatures ranging from 60 °C to 90 °C with 10 °C increment at aging periods of 3, 7, and 28 days. Although the highest compressive strength at all tested ages is detected at 70 °C, the varied curing temperatures produce comparable compressive strengths at 3 and 7 days. The specimens cured at 80 °C and 90 °C and aged for 28 days show relatively lower strength development than geopolymers cured at 70 °C. The loss of moisture at curing temperatures of 80 °C and 90 °C may cause the reduction in strength because the geopolymerization reaction requires the presence of moisture to develop good strength (Chindaprasirt, Chareerat & Sirivivatnanon 2007).

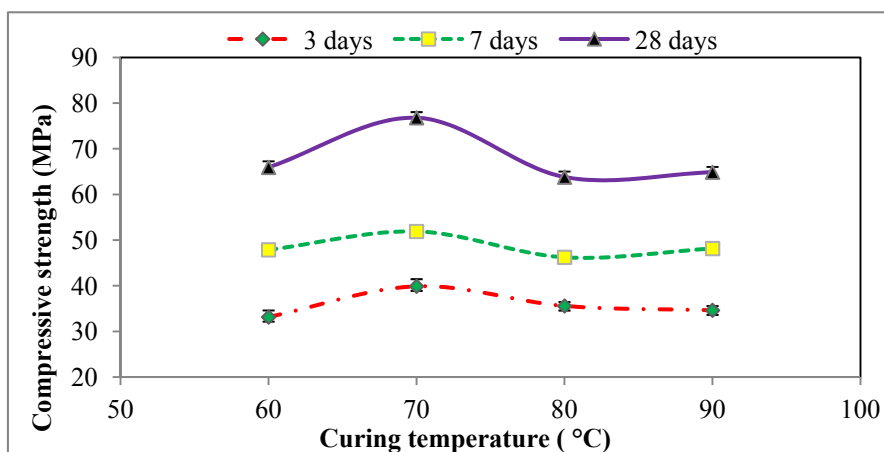


Figure 5. Effect of different curing temperatures on the compressive strength development of the FA geopolymer paste

### 3.3 Effect of Curing Period on the Strength Development of FA Geopolymers

Figure 6 presents the effect of curing periods from 6 h to 30 h with increment of 6 h on the compressive strength development of the FA geopolymer paste specimens. The strength development of the resultant geopolymers increases with increasing curing period up to 24 h. The specimen cured for 24 h exhibits the optimum compressive strength at all testing ages compared with those cured for 6, 12, and 18 h. This finding demonstrates that the highest geopolymerization reaction rate can be obtained at long curing time. However, curing FA geopolymers for more than 24 h up to 30 h negatively affects the strength development. The prolonged curing of more than 24 h may result in the breakdown of the gel structure of the geopolymer matrix, leading to low resultant strength (van Jaarsveld, van Deventer & Lukey, 2002).

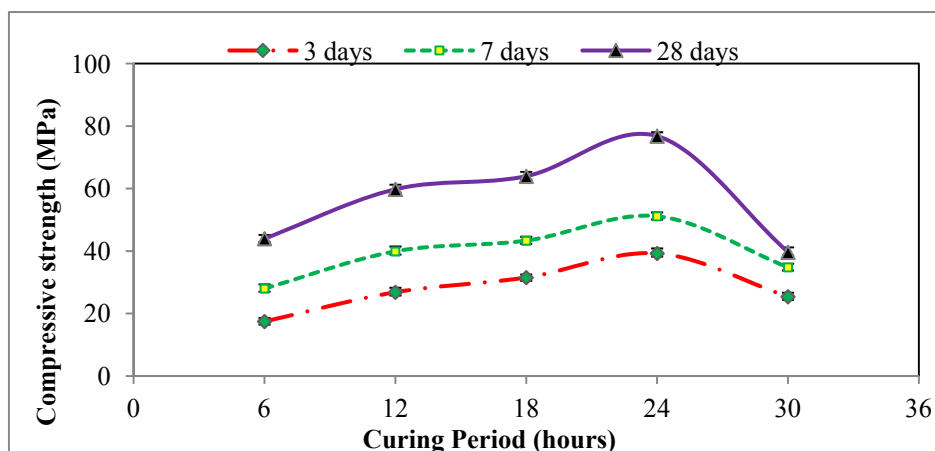


Figure 6. Strength development of the geopolymers paste at different curing periods

## 4. Conclusions

The following conclusions are established in this paper:

- The mixing ratio of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  affected the development of high-strength FA geopolymers. With sufficient alkaline contents and Si species available in the geopolymerization reaction, the strength development of the hardened geopolymer continued to increase with aging time. Although the  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratio of 1.00 showed the highest strength development,  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratios of 2 and 2.5 were apparently more applicable for large-scale production of FA geopolymer binders in terms of economic aspects.
- SEM results showed the effect of NaOH solution content on the dissolution of the prime materials (FA). The higher content of NaOH solution the higher dissolution of FA. However, with low content of  $\text{Na}_2\text{SiO}_3$  in the alkaline activator as the  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratios of 0.5 resulted in relatively low strength development



at different ages.

- The optimum curing temperature for FA geopolymers was 70 °C, and curing temperatures of 80 °C and 90 °C adversely affected the strength development after aging for 28 days. This finding presented another advantage in terms of decreasing the cost of curing process.
- Long curing periods produced high-strength geopolymers. However, extending the curing period for more than 24 h negatively affected the strength development at different testing ages.

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