

Effect of Sodium Hydroxide Concentration on the Mechanical Property of Non Sodium Silicate Fly Ash Based Geopolymer

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Abstract: Alkali activator and materials rich in Si and Al are the requirements for the geopolymer synthesis. The common activator use is sodium hydroxide with silicate solution. There is limited literature on the mechanical property of geopolymer in the absence of silicate solution. In this study, fly ash was used as the raw material to provide Si and Al and sodium hydroxide (NaOH) as the only activator for the synthesis of geopolymer. This study describes the effect of varying the sodium hydroxide concentration with respect to curing time and temperature on mechanical properties of non-sodium silicate fly ash based geopolymer. The samples were prepared by mixing fly ash with 8, 10 and 12 M of NaOH concentration and cured in room temperature and 60°C for 1, 7 and 28 days. The highest alkali concentration of 12 M showed the fastest setting time and the highest compressive strength regardless of days of curing. The highest compressive strength obtained for curing at 60°C was 59.81 MPa and 45 min was recorded for the setting time. Meanwhile, at room temperature, the highest strength obtained was 17.71 MPa and 248 min for setting time.

Key words: Fly ash, geopolymer, alkaline solution

INTRODUCTION

Geopolymer or known as inorganic polymer is a new class of synthetic alumina-silicate materials that involves a chemical reaction between alumina-silicate oxides and alkali metal silicate solutions under highly alkaline conditions yielding an amorphous to semi-crystalline three-dimensional polymeric structures that consist of Si-O-Al bonds (Davidovits, 1999). Geopolymer material has been studied to replace ordinary portland cement and coating material due to its excellent physico-chemical and mechanical properties such as low density, micro porosity or nano porosity, thermal stability, negligible shrinkage, high strength, high surface hardness, fire and chemical resistance (Panas *et al.*, 2007).

Source material from the industrial waste and/or by-product containing high Si and Al content and alkali activator are the primary requirements to undergo geopolymerization (Khale and Chaudhary, 2007). Thus, geopolymerization can be considered as an economically viable technology for the transformation of waste into valuable product.

In this study, Fly Ash (FA) was used as the source of Si and Al. In geopolymerisation, activator such as alkaline solutions play an important role for the process

of dissolution of raw material. Based on the literature, studies are done by using sodium/potassium hydroxide and sodium silicate as alkaline activator. Silicate solution is added for the purpose of dissolution of raw material. This is to form reactive precursors required for geopolymerisation and enhance the process. It is shown that sodium silicate help to increase the dissolution of the starting materials and give good mechanical properties (Rees *et al.*, 2004). To the authors' knowledge, limited research has been done using sodium hydroxide as the only activator in geopolymerization process. In this study, we have eliminated the usage of silicate solution as an effort to produce green material. The present study describes the effect of sodium hydroxide concentration without using soluble silicate on the compressive strength and setting time of geopolymer. In particular, the strength development with respect to curing time and temperature has also been investigated.

MATERIALS AND METHODS

Materials: Fly Ash (FA) of Class F type was used for the geopolymer work. The chemical composition was determined by using X-Ray Fluorescence (XRF) as shown in Table 1. Sodium hydroxide (NaOH) solution was

Table 1: X-ray Fluorescence data analysis of fly ash

Chemical component	Composition (wt %)
SiO ₂	43.34
Al ₂ O ₃	20.77
Fe ₂ O ₃	12.41
CaO	11.13
MgO	3.75
K ₂ O	1.98
SO ₃	1.45
Others	5.17

Table 2: Summary of synthesis parameters

Raw material	Fly ash
Alkali activator	NaOH concentration (8, 10 and 12 M)
Curing time (days)	1, 7 and 28 days
Curing temperature	Room temperature and 60°C

prepared by dissolving NaOH pellets in distilled water and acted as activator. NaOH solution was prepared a day earlier to ensure the heat produced from the reaction is dissipated and do not affect the setting of geopolymer.

Methods: The solid/liquid (NaOH) ratio of 3 was maintained for the geopolymer synthesis. The geopolymer synthesis was done by adding alkali activator into the fly ash and mixed well by using a mechanical mixer for 1.5 min. The fresh geopolymer mixture was quickly casted into the vicat apparatus ring and the setting time was determined at room temperature and at 60°C. Final setting time was measured using vicat apparatus according to ASTM C191. Later, the geopolymer was placed in the 50×50×50 mm mould. Samples were cured at room temperature and 60°C and cured at different curing time. Curing at 60°C was chosen as it reaches optimum compressive strength at this temperature (Al Bakria *et al.*, 2011). Summary of parameters studied is shown in Table 2.

Analysis: The compressive strength test was done by using ELE compression strength with loading rate of 0.9 kN sec⁻¹ according to ASTM C109 and average strength of three measurements was obtained. The structural bonding of raw material and geopolymer products were tested by using Fourier Transform Infrared spectroscopy (FTIR).

RESULTS AND DISCUSSION

Effect of NaOH concentration on setting time: Based on Fig. 1, the increase of NaOH concentration used results in reduction of setting time. The increase of hydroxide ions seems to accelerate the formation of aluminosilicate gel thus lowering the setting time. Shorter time is required by the samples to set when it is exposed to heat. The heat accelerates the geopolymerization process thus shorter setting time is obtained (Panias *et al.*, 2007).

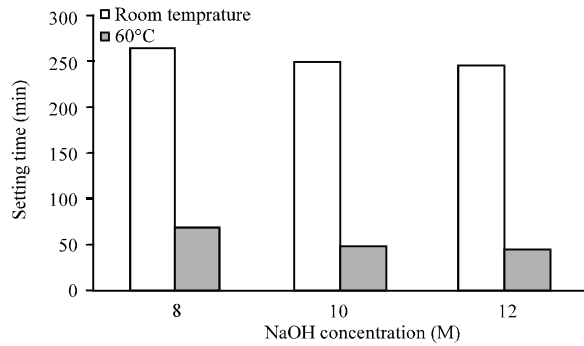


Fig. 1: Effect of NaOH concentration and curing time on compressive strength

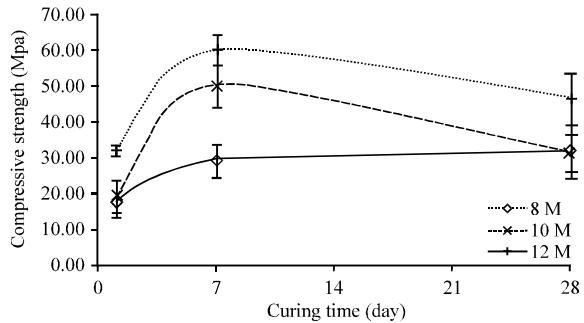


Fig. 2: Compressive strength vs. curing time with variation of NaOH concentration

Figure 2 shows that the compressive strength development for various concentration with respect to curing time at 60°C. It is observed that the highest compressive strength obtained is by using the highest concentration of sodium hydroxide which is 12 M regardless of curing time even though sodium silicate is not used. The result shows similar trend with the reported research by using both sodium hydroxide and sodium silicate that act as activator. It is reported that the high use of molarity of NaOH could accelerate dissolution and hydrolysis and thus develop high compressive strength (Zuhua *et al.*, 2009). In addition, the strength increased with the increased NaOH concentration that may be due to the leaching process of silica and alumina (Chindapasirt *et al.*, 2009). It shows that NaOH concentration also play significant role in affecting mechanical property of geopolymer produced. The highest compressive strength obtained at room temperature was 17.71 MPa for 28 days of curing. Meanwhile, the highest compressive strength for curing at 60°C was 59.81 MPa for 7 days of curing.

Based on Fig. 2, the compressive strength of the samples tends to decrease after 7 days of curing at 60°C for all mixtures. It is reported that for long curing time, at elevated temperature it weakens the material structure

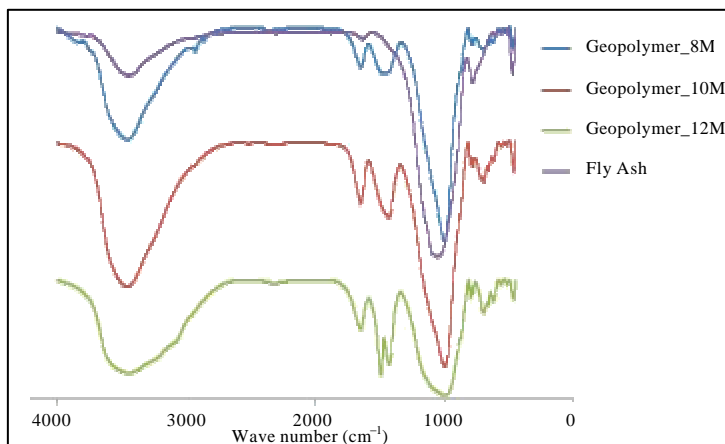


Fig. 3: Infrared spectrum of raw material and geopolymer products cured at 60°C for 1 day

Table 3: Characteristic of FTIR bands of fly ash and geopolymer product

Wave No. (cm ⁻¹)	Assignment
3500-3000	Stretching vibration (-OH, HOH)
1650-1620	Bending vibrations (HOH)
1460-1420	Stretching vibration (O-C-O)
1090-990	Asymmetric stretching vibration (T-O-Si, T = Si or Al)
550-750	Symmetric stretching vibration (Si-O-Si and Al-O-Si)
460-470	Bending vibration (Si-O-Si and O-Si-O)

and result in decreasing of strength. This result shows that curing condition is also an important parameter for development of mechanical properties.

The structure and bonding of geopolymer with different concentration used is shown in Fig. 3. The sample for 1 day cured was taken for the analysis. The main FTIR absorption bands of fly ash and geopolymer sample is summarized in Table 3. The difference that can be observed between FTIR spectrum of fly ash and FTIR spectrum of geopolymer is coherent to the asymmetric stretching vibration of Si-O-Si and Al-O-Si. This band appears at 1051 cm⁻¹ and shifted to lower frequency (~999 cm⁻¹). This indicates that the formation of new product is the amorphous aluminosilicate gel phase. This also can be related with the dissolution of the fly ash in the alkali activating solution. The most shifted band shows the faster dissolution and is showed by using 12 M concentration of NaOH. The faster dissolution can be associated with the shortest setting time and the increase of high mechanical strength as shown in Fig. 1 and 2. The appearance of band at frequency ~750-550 cm⁻¹ indicates the symmetric stretching vibration Si-O-Si and Al-O-Si. This is also referred to the formation of amorphous to semi crystalline aluminosilicate materials. The band in the region of 3500-3000 cm⁻¹ is assigned to stretching (-OH) and bending (H-O-H) vibration of water molecule. Similar result was reported using both sodium hydroxide and sodium silicate (Paniias *et al.*, 2007).

CONCLUSION

This study has shown that without using soluble silicate, high compressive strength can be obtained. The shortest setting time recorded was 45 min at 60°C by using 12 M of NaOH concentration. The mechanical property of geopolymer was affected by the concentration of NaOH used, curing time and curing temperature. The highest compressive strength was achieved by using high concentration of NaOH of 12 M. In addition, the highest strength of 59.81 MPa was obtained when cured at 7 days at 60°C.

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