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Effect of Alkali Concentration on Fly Ash Geopolymers

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Abstract. This paper presents the effect of NaOH concentration on fly ash geopolymers with compressive up to 56 MPa at 12M. The physical and mechanical on fly ash geopolymer are investigated. Test results show that the compressive strength result complied with bulk density result whereby the higher the bulk density, the higher the strength. Thus, the lower water absorption and porosity due to the increasing of NaOH concentration.

1. Introduction

Geopolymer was developed by Davidovits [1] in 1978 describing a family of mineral binders which have amorphous to semi-crystalline polymeric silicon-oxygen-aluminum framework structure. Davidovits [2] also proposed a terminology of polysialates as shown in Figure 1 by taking into consideration the Si-to-Al ratio defined as the following: "geopolymer is a polymer which is not original polycondensation, but by so-called "geopolymerization" reaction as result of the alkali activation of aluminosilicate materials.

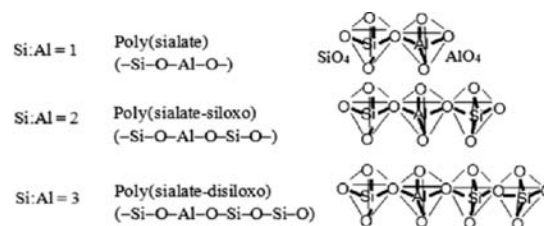


Figure 1: Davidovits terminology on geopolymer [2].

A three-dimensional aluminosilicate network created by the empirical formula $\text{Na}_n\text{M}_z(\text{Si}_n\text{Al}_m)_z \cdot n\text{wH}_2\text{O}$ where M is K, Na or Ca cation, n is the degree of poly-condensation and z is the 1, 2, 3 or more than 3 is formed. The network is configured of SiO_4 and AlO_4 tetrahedrons united together by oxygen bridges. Chains or rings united by Si-O-Al bridges are formed. Positive ions (Na^+ , K^+ , Ca^{2+}) must compensate the negative charge of Al that is present in the IV-fold coordination [2].



Geopolymer can be categorized as an inorganic polymer that created by the reaction between sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3) with solid aluminosilicate materials [4]. A strong alkaline solution induces the dissolution of those solid raw materials producing aluminate and silicate species which linked by means of a polycondensation reaction to form the three-dimensional-cross-linked polysialate structure $([-(\text{Si-O})_z\text{-Al-O-}]_n)$ [5]. It is necessary to dissolve and release the certain amounts of silica and alumina as to increase the surface hydrolysis the particles of the raw materials [6]. Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) are commonly used in the studies.

According to previous studies [7], it was found that with the higher concentration of NaOH solution that is above 10M, a lower rate of polymerization took place and the strength decreased. In addition, higher alkaline activator content gave a higher compressive strength. Molarity of NaOH solution plays a vital role in the strength of geopolymer. The NaOH concentration will affect the formation of aluminosilicate geopolymer network and change the geopolymer structure of the material since some losses in mechanical properties may occur due to the free OH^- in the alkali-activated matrix and consequently changes the properties of the end product [6, 8]. Previous studies [3, 9, 10] reported that the type of activator affected the properties of its alkali-activated and recommended sodium hydroxide since it is commonly used. Thus, in this study, the effect of NaOH concentration on the physical and mechanical properties of fly ash geopolymers was investigated.

2. Experimental Method

2.1 Raw Materials

Fly Ash (FA) was used as aluminosilicate source. It was collected from a local coal power plant Manjung, Perak. The chemical composition of fly ash as determined by X-Ray Fluorescence (XRF) analysis is given in Table 1. It was a class F fly ash due to the calcium oxide (CaO) content was less than 10% according to the ASTM C-618.

Table 1. Chemical composition of fly ash (FA).

Compound	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	SrO	SO_3	K_2O	MnO	PbO	NiO
Mass (wt%)	52.3	25.9	10.52	2.92	4.90	0.654	0.05	1.81	0.032	0.046	0.027

The NaOH used was caustic soda flakes with the brand name of Formosoda-P purchased from the Formosa Plastic Corporation, Taiwan. The technical grade liquid Na_2SiO_3 was purchased from South Pacific Chemicals Industries Sdn.Bhd. (SPCI), Malaysia. The liquid Na_2SiO_3 is colourless and dissolves readily in water. It has SiO_2/NaO modulus of 3.20. Stock solution of NaOH (4M-14M) was prepared and allowed at room temperature. The NaOH and sodium silicate were mixed at ratio 2.5 to prepare the alkali activator. This activator solution was prepared 24 hours before use.

2.2 Preparation of Fly Ash Geopolymers

The fly ash was mixed with alkali activator at ratio of 2.0 to produce the fly ash geopolymers paste. The fly ash geopolymers were then cured at room temperature for 24 hours, followed by 24 hours at 60°C . After curing, the geopolymers samples were kept in room temperature until the day of testing at 7 and 28 days.

2.3 Testing and Analysis Methods

Before conducting the compressive strength, the bulk density was measured by measuring the dimension and weight. For water absorption and porosity testing, three samples from each series of NaOH concentration were oven dried at a temperature of 105°C for 24 hours and its weight was determined as weight dry (W_d). The samples were then immersed in water for 24 hours as the saturated weight in water (W_w) and its saturated surface dry weight as the saturated weight in air (W_a). The following equations (1) and (2) were used to calculate the water absorption and porosity values, respectively, in percentage.

$$\% \text{ Porosity} = \frac{(W_a - W_d)}{(W_a - W_w)} \times 100\% \quad (1)$$

$$\% \text{ Water Absorption} = \frac{(W_a - W_d)}{W_d} \times 100\% \quad (2)$$

The compressive strength of fly ash geopolymers was tested using Mechanical Tester as according to ASTM C 109/C 109M-08. Three samples were tested to obtain an average value of strength. All the tests were conducted after 7 and 28 days. A JSM-6460LA model Scanning Electron Microscopy (JEOL) was used to analyze in the morphology of fly ash and fly ash geopolymers. For microstructural analysis of fly ash geopolymers, fly ash geopolymers were the fracture surfaces after the compressive test.

3. Results and Discussion

3.1 Bulk Density, Porosity and Water Absorption

The bulk density, water absorption and total porosity values of the fly ash geopolymers at different NaOH concentration after 7 and 28 days are presented in Figure 2. The density of fly ash geopolymers varied from the range 1.63 until 1.89 g/cm³. In general, the bulk density did not change significantly with varying NaOH concentration. Even so, it could be clearly seen that the highest bulk density was recorded for NaOH concentration at 12M, the bulk density which is 1.89 g/cm³ after 7 days and 1.87 g/cm³ after 28 days. The bulk density slightly dropped with increasing NaOH concentration up to 14M as achieved the optimum concentration at 12M as shown in Figure 3. The concentration of 14M shows a clearer difference between 7 and 28 days due to a reduction in the resulted weight. Increasing the NaOH concentration may result in paste of a lower density compared to other molarity due to the mixing problems. Density is known to affect the mechanical strength of geopolymers [11].

From Figure 2 (b) and Figure 2 (c), the results showed that water absorption and porosity values decrease when using the higher NaOH concentration. In particular, with NaOH concentration was increased up to 12M, the lowest porosity percentage of 24.64% after 7 days and 22.74% after 28 days were recorded. The porosity is related to the structure and water absorption of the geopolymers [10]. At 4M, fly ash geopolymers showed the highest water absorption of 32.22% after 7 days and at 12M, the lowest water absorption of 12.51% after 7 days and 11.89% after 28 days were collected. The results complied with the bulk density measurement in Figure 2 (a). It is observed that the bulk density of 4M and 8M almost the same density values although the porosity shows the quite difference. This is due to the low strength and porous morphology of the fly ash geopolymer which reduce the porosity at 8M. The higher bulk density samples had lower porosity and thus lower water absorption. The result showed that higher NaOH concentration in the mix gives better reactivity with fly ash resulting in denser microstructure [12].

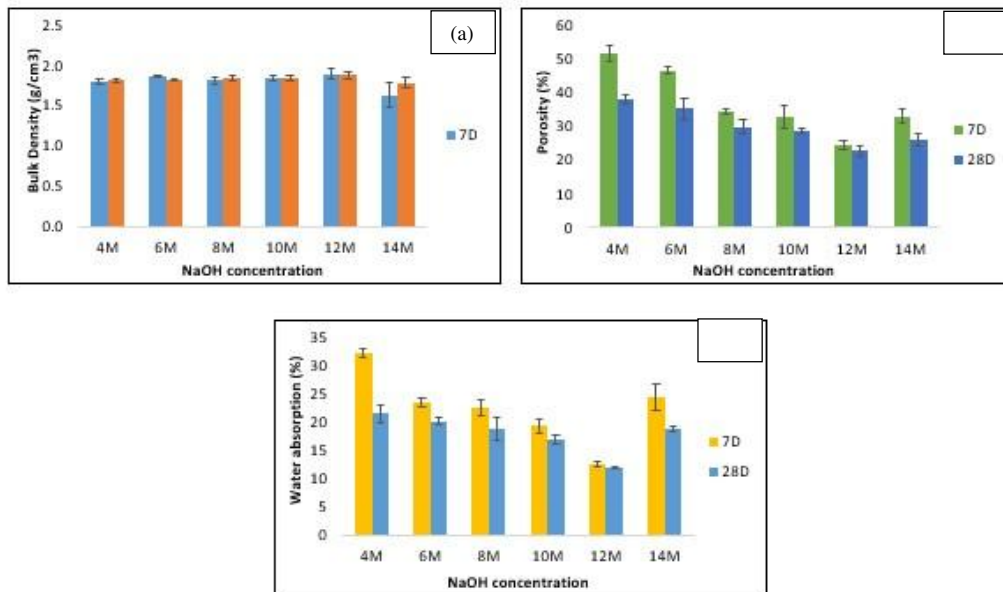


Figure 2: Bulk density of fly ash geopolymers at varying NaOH concentration, (b) porosity (%) of fly ash geopolymers after 7 and 28 days, (c) water absorption (%) of fly ash geopolymers.

3.2 Compressive Strength

Figure 3 presents the compressive strength of fly ash geopolymers at different NaOH concentration in the range of 4M to 14M after 7 and 28 days. Initially, compressive strength gain of fly ash geopolymers for all NaOH concentration slightly fluctuated after 7 days. At 10M, the compressive strength was recorded the highest (29 MPa) after 7 days. This might due to the geopolymers were still at the early stage of strength development [13]. After 28 days, the compressive strength development showed an increasing trend with increasing NaOH concentration up to 12M (56 MPa) due to the high concentrations of NaOH can increase the dissolving rate of silicon and aluminium atoms in raw materials [14]. Beyond 12M, the strength reduced (52 MPa for 14M). The strength reduction at 14M was most probably due to a low of geopolymerization formation [15].

Palomo et al. [16] also proposed that alkaline activator with 12M NaOH solution indicated better result compared to 18M NaOH. At low NaOH concentration of 12M, it meant that there are sufficient Na⁺ and OH⁻ ion for complete geopolymerization reaction and hence the highest compressive strength. The increased strength is related to the structure and density of the geopolymers. The compressive strength result complied with bulk density, porosity and water absorption values. Higher bulk density, lower porosity and percentage of water absorption contributed to higher compressive strength [12]. Thus, the compressive strength with respect to the increase of NaOH concentration was the factor that controls the performance of the fly ash geopolymers. The increased of strength at high NaOH concentration was supported by Pimraksa et al. [17] and Bakkali et al. [18]. Bakri et al. [15] observed the molarity of NaOH is one of the factors that affect the early age compressive strength of fly ash geopolymers.

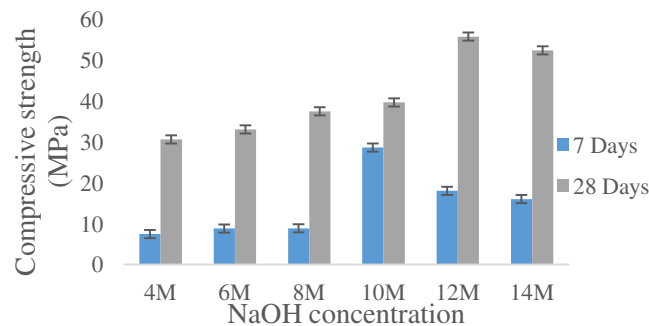


Figure 3: Compressive strength (MPa) of fly ash geopolymers after 7 and 28 days.

3.3 Microstructure Analysis

Figure 4 reveals the SEM micrographs of fly ash whereas Figure 5 shows the SEM micrograph of fly ash geopolymers with various NaOH concentration after 7 and 28 days. Fly ash particles are spherical in shape with smooth surface. After alkali activation, the fly ash particles dissolved and participated in the geopolymerization reaction. Some fly ash particles can still be clearly seen in the geopolymer matrix. However, the smooth surface could no longer be seen with some agglomerated particles on its surface due to alkali attack. According to Bakri et al. [19], fly ash reacted with the NaOH concentration produced the roughness of the surface.

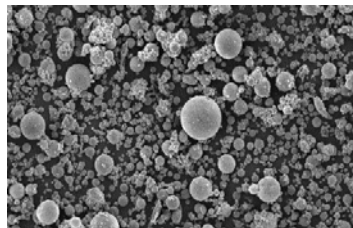


Figure 4: SEM micrograph of fly ash.

Moreover, images of fly ash geopolymers showed the combination of unreacted fly ash particles and also heterogeneous elements. It showed in Figure 5 that the materials are heterogeneous and contain a large proportion of unreacted fly ash after 7 days. However, images of fly ash geopolymers showed the smooth geopolymer matrix and less the unreacted fly ash particles after 28 days. The smooth geopolymer matrix presented in different shapes of the pure fly ash and the fly ash geopolymers. Besides, the various NaOH concentration resulted in presence of pore and less the unreacted fly ash since the spherical shape rarely existed. With increasing NaOH concentration, the unreacted fly ash particles were less abundant and the matrices looked rather dense. This was because of the better ability to dissolve fly ash particles which resulted in better geopolymerization [7].

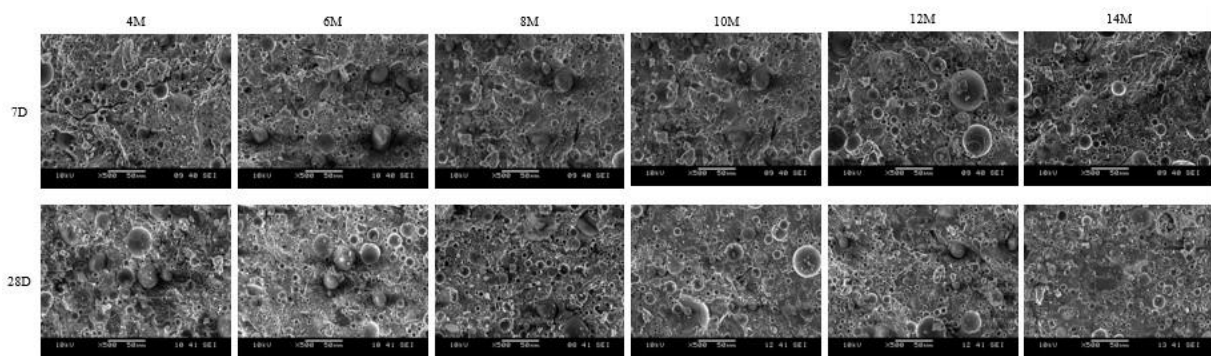


Figure 5: SEM micrograph of fly ash geopolymers after 7 and 28 days with varying NaOH concentration.

4. Conclusion

The molarity concentration of sodium hydroxide (NaOH) in fly ash geopolymers gives a major effect on the geopolymerization process and on the strength of the resulting geopolymer. When the NaOH concentration increased up to 12M, the highest strength of 56 MPa was achieved thus lowering the water absorption and porosity. In SEM images, the higher concentration of NaOH solution showed less the unreacted fly ash particles and smooth geopolymer matrix with fewer pores.

Acknowledgments

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