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Correlation of Microstructure and Flexural Strength of Fly Ash Geopolymer of Varying Molarities

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Abstract. The present work studies and compares the flexural properties of fly ash geopolymer with varying molarity of sodium hydroxide (NaOH). Different molarities of NaOH solution (6M, 8M, 10M, 12M and 14M) are used to prepare the alkali activator (a mixture of NaOH and sodium silicate). The fly ash geopolymer were synthesised by using the solid/liquid ratio of 2.5 and sodium silicate/NaOH ratio of 2.5. The samples were cured at 60°C for 6 hours. The flexural test and microstructural analysis of fly ash geopolymer are performed after 28 days. Generally, the flexural strength of fly ash geopolymer increases when the molarity of NaOH increases. The 12M geopolymer has the highest flexural strength and a more densified microstructure is observed.

1. Introduction

Geopolymer is an inorganic aluminosilicate polymer where it is synthesised via the alkali activation process of mixing the solid aluminosilicate base materials with the mixture of an alkali metal hydroxide and silicate solution [1, 2]. The types of solid aluminosilicate base materials could be divided into natural materials such as kaolin and metakaolin [3] or industrial by-products such as ground-granulated blast furnace slag (GGBS) and fly ash [4]. Geopolymers are sustainable as they are ecological friendly with low carbon footprint and low energy consumption production process [5].

Geopolymers are amorphous in phase with the three-dimensional structure arranged in short-range order [6]. This three-dimensional framework of SiO₄ and AlO₄ gives geopolymer ceramic-like characteristics and exhibits a wide range of properties such as high mechanical strength, low shrinkage, low water absorption, fast setting time, acid resistance, fire resistance and low thermal conductivity [6, 7]. Thus, it has captured the interest of researchers in various application such as construction and building sector, fire resistance, toxic metal immobilization and waste management [8].

Geopolymerisation involves several stages including the alkali activation of SiO₄ and AlO₄ as precursor materials to yield geopolymer gel followed by hydrolysis where the bond breaking of SiO₄ and AlO₄ tetrahedral units by water molecules occurred [9]. These units reorganize and link to form the polymeric precursors. The geopolymer gel solidified and a three-dimensional aluminosilicate network was formed [10].

The mechanism of geopolymerisation for geopolymer formation is influenced by various parameters such as raw materials, solid/liquid ratio, alkali activator ratio, curing regime and molarity of alkali



solution used. Consequently, this parameter would also indirectly affect the properties of the geopolymer. Among these parameters, the molarity of alkali solution plays a significant role as the cation (Na^+ , K^+) contributed from the alkali solution balances the charge and maintains the neutrality of the framework of geopolymers [11]. Also, alkali solution promotes the dissolution of aluminosilicates and hence increases the degree of geopolymerisation reaction [12]. In this study, a relationship between the molarity of sodium hydroxide with the flexural properties and microstructure and fly ash geopolymer was investigated.

2. Experimental

2.1. Materials

Fly ash collected from Cement Industries of Malaysia Berhad (CIMA) Perlis Plant, Malaysia was used as the source of aluminosilicate. The chemical composition of the fly ash obtained through XRF analysis is tabulated in Table 1. Referring to Table 1, the fly ash used could be classified as Class F fly ash according to ASTM C618 as it contains total composition of SiO_2 and Al_2O_3 of 84.30% and the CaO content is 3.89% which is less than 20% [13]. Figure 1 displayed the SEM micrograph of fly ash particle which was spherical in shape with smooth surfaces. The technical grade sodium silicate solution (Na_2SiO_3) has chemical composition of 30.1% SiO_2 , 9.4% Na_2O and 60.5% H_2O with modulus $\text{SiO}_2/\text{Na}_2\text{O}$ of 3.2, specific gravity at 20°C = 1.4 and viscosity at 20°C = 0.4 Pa.s. The sodium hydroxide (NaOH) pellets used was classified as caustic soda micropearls with 99% purity.

Table 1. Chemical composition of fly ash.

Compound	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	K_2O	Others
Mass (%)	56.30	28.00	6.86	3.89	2.17	1.49	1.29

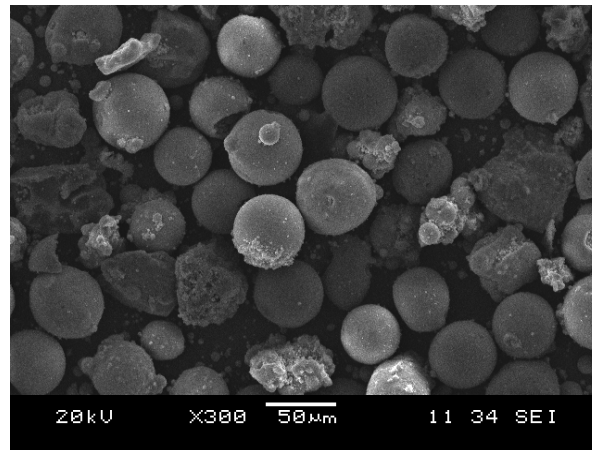


Figure 1. SEM micrograph of fly ash particles.

2.2. Geopolymer Formation

The NaOH solution with range of 6M-14M was prepared and then allowed to cool down to room temperature. The NaOH solution was mixed with liquid Na_2SiO_3 ratio of 1:2.5 to prepare the alkali activator. Then, the fly ash was mixed with the alkali activator at ratio of 1:2.5 until homogenous slurry was obtained. The fresh paste was then rapidly moulded and compacted into a 160 40 10 mm moulds. The moulded geopolymer samples were cured at 60°C in oven for 6 hours. The cured samples were kept at room temperature for 28 days before testing.

2.3. Testing and analysis

Flexural strength tests were performed according to ASTM C1505-15 by using the Instron machine series 5569 Mechanical Tester with a span length of 80 mm at a crosshead speed of 5 mm/min. JSM-6460LA model Scanning Electron Microscope (JEOL) was performed to reveal the microstructure of the fracture surface of fly ash geopolymer.

3. Results and Discussions

3.1. Flexural Strength

Based on the data obtained in [14], the flexural strength of fly ash geopolymer increased continuously when the molarity of NaOH increased from 6M to 12M but beyond of that the flexural strength reduced. The flexural strength and bulk density obtained is tabulated in Table 2. This flexural strength result complied with the bulk density of the fly ash geopolymer obtained where a more compact density indicates a higher flexural strength. This is further supported by the SEM micrographs shown in Figure 3. In this context, the geopolymerisation process becomes undesirable after reaching an optimum NaOH molarity [15]. The high viscosity of high molarity NaOH solution would hinder the solubility and deionisation of Si and Al ions from aluminosilicate sources. This statement is supported by He *et al.* [16] as they observed the occurrence of premature precipitation in geopolymer matrix and lead to deterioration in mechanical properties of the final product due to presence of remnant precursor material.

Table 2. Flexural strength of fly ash geopolymer with varying molarity of NaOH solution.

NaOH Molarity	6M	8M	10M	12M	14M
Flexural Strength (MPa)	2.35	1.95	3.31	4.80	3.53
Bulk Density (kg/m ³)	1753	1702	1852	1900	1856

3.2. Microstructural Analysis

Figure 2 displays the SEM micrograph of alkali activated fly ash particles. As compared to the fly ash micrograph in Figure 1, it could be observed that the surface of spherical fly ash particles opened up when the fly ash was mixed with the alkali activator. Fernandez-Jimenez *et al.* [17] reported that the dissolution process of Al and Si occurs when fly ash is added with alkali activator and followed by the condensation of higher molecules into a gel via nucleation and polymerisation. Then, the alkali attack opened up the spheres and exposed the internal small spheres. The small spheres inside would dissolve when in contact with alkali and the reaction products were formed inside and outside the sphere.



Figure 2. SEM micrograph of alkali activated fly ash particles.

The microstructures of the fracture surface of fly ash geopolymer with varying NaOH molarities are shown in Figure 3. It could be observed that there are large portion of unreacted or partially reacted fly ash particles present in the matrix of 6M and 8M NaOH-activated fly ash geopolymer (Figure 3a, 3b) which possessed a lower flexural strength. Similar observation was observed in Abdullah *et al.* [18] where the undissolved fly ash particles were loosely bonded to the geopolymer structure and interfacial boundaries could be observed in the structure. Besides, the presence of pore could be observed as well in the geopolymer matrix and these pores caused the imperfection in microstructure which result in poor mechanical properties [19].

On the other hand, fly ash geopolymer with a higher flexural strength (10M, 12M and 14M) have a smoother and more densified microstructure (Figure 3c, 3d and 3e). These densified and compacted structure once again complied with the flexural properties in Table 2 [14]. Hamidi *et al.* [20] reported that the geopolymeric gel formed from a greater extent of geopolymerisation was able to hold the constituent and fly ash particles well together to form a more compact structure. In this context, the increase in molarity of alkali solution could promote the formation of geopolymeric gel and hence results in a better mechanical properties [18].

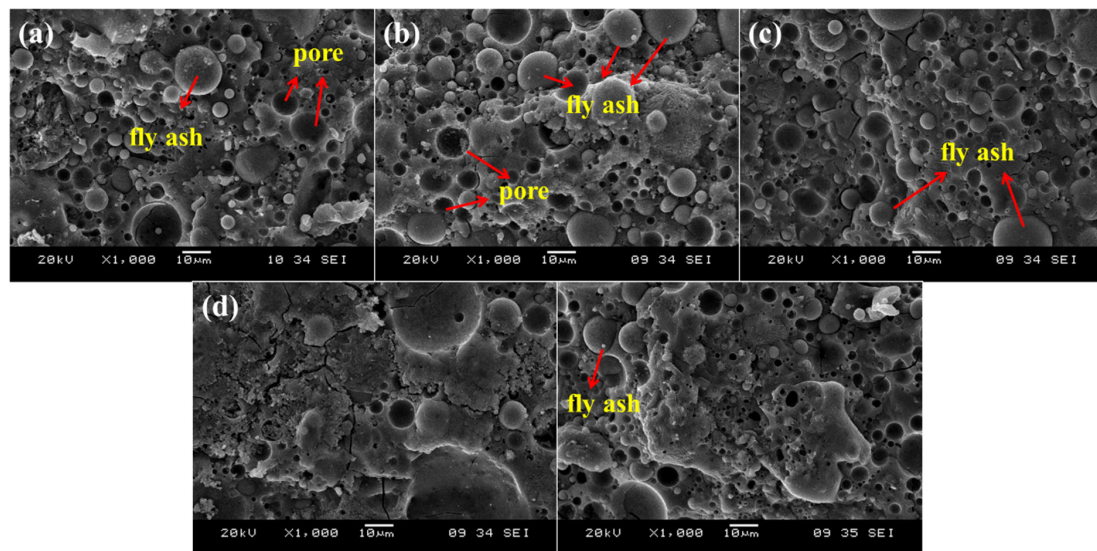


Figure 3. SEM micrographs of the fracture surface of fly ash geopolymer with NaOH molarity of (a) 6M; (b) 8M; (c) 10M; (d) 12M and (e) 14M

4. Conclusions

This paper studied the relationship between the molarity of sodium hydroxide with the flexural properties and microstructure and fly ash geopolymer. The optimum molarity of NaOH is 12M which gave better strength properties of fly ash geopolymer. A more compacted and densified microstructure is observed in which correlated with the flexural properties of 12M NaOH-activated fly ash geopolymer. However, further work should be implemented to boost the flexural strength of fly ash geopolymer.

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