The effect of KOH concentration on setting time and compressive strength of fly ash-based geopolymer

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Abstract. Geopolymer is produced from the alkali activation of materials rich in Si and Al such as fly ash. Sodium hydroxide (NaOH) with high concentration is normally used in geopolymerization. Limited research has been done with low concentration of alkali activator. This study confirms that KOH with low concentration affect the setting time and compressive strength of geopolymer in order to have good mechanical properties. Optimum result was observed at 4.5 M KOH. This result can be further developed to produce geopolymer with low alkaline activator for coating applications.

Introduction

Inorganic polymers, more commonly referred to as "geopolymers" are aluminosilicate materials which exhibit excellent physical and chemical properties with a diverse range of potential applications, including concrete, immobilization of toxic, and coating application [1]. In general, geopolymers can be synthesized by alkali activation of material rich in SiO₂ and Al₂O₃. The mechanism involves the dissolution of Al and Si in the alkali medium, transportation (orientations) of dissolved species, followed by a polycondensation, forming of 3D network of silico-aluminate structures [2]. The setting (hardening) of geopolymer is believed to be due to the polycondensation of hydrolysed aluminate and silicate species. The formation of this phase appears to be very sensitive to the alumunium, silicon, and activator concentration in the mixture and is directly influenced by raw materials used.

Activator such as NaOH is used to influence dissolution process. In recent papers reported, NaOH with high concentration and sodium silicate solution as additive are used to increase dissolution in order to increase compressive strength. Limited work has been done using KOH as activator. The concentration of K^+ and OH⁻ ions in a medium modifies the pH level and is a primary parameter for controlling the silica dissolution and silicon concentration. The typical geopolymer composition is generally expressed as $nM_2O.Al_2O_3.xSiO_2.yH_2O$, where M is an alkali metal [2].

Geopolymer for coating application has been studied for metal substrate and marine concrete. Generally, few properties for coating such as acceptable setting time, high strength, and low permeability are required [3]. They examined that geopolymer can be a promosing material for protective coating for different surface due to good mechanical, chemical and thermal resistance properties. Geopolymer as marine concrete coating had shown excellent anti corrosion property with efficient bonding with cement phase and mortar. The acceptable setting time was less than 6 hours [3].

This paper presents the effect of KOH concentration on setting time and compressive strength of fly ash-based geopolymer for coating application.

Experimental Procedure

A. Material

Class F fly ash from industrial waste was used as raw material. Laboratory grade potassium hydroxide (KOH) and distilled water was used to prepare alkaline solution. For alkaline solution preparation used various KOH molarity was used.

B. Methods

Mixing was done in an air conditioned room at approximately 25° C. The mixing procedure started with mixing of KOH solution and fly ash for 5 min. Well-mixed geopolymer was used for the measurement of setting time using the Vicat apparatus according to ASTM C191. Setting time is the time required by the fresh paste for hardening, which reflected the water content in the mixture. Geopolymer cubes were also prepared by casting the geopolymer mixes in the 50 mm x 50 mm mould and vibrated for 5-10 minutes to reduce entrained air for compressive strength tests according to ASTM C109. Table 1 shows the synthesis parameter used in this work.

Raw material	Fly ash
Alkali activator	1.5; 2.5; 3.5; 4.5; 5.5 M of KOH concentration
Ratio of solid/ liquid	3
Curing time (days)	7 days
Curing temperature	60°C and room temperature

Table 1: Synthesis parameter

C.Analysis

Characterization was done using FESEM to investigate the microstructure of geopolymer.

Result and Discussion

A. Effect of KOH concentration on setting time

Setting time is one of the material properties that can be used in the prediction of the adhesion strength of the material in coating application. Decrease in setting time is expected to develop good adhesion strength between substrate and coating material, which is desirable [4]. However, if the geopolymer set or hardened too fast it would result in bad coating quality during the coating process. Based on Figure 1, the setting time decrease as the KOH concentration increase. Alkali solution with higher concentration provided better dissolution of fly ash a particles and generating more reactive bonds for monomer, which in turn increased geopolymerization of pastes. The lowest setting time occur at 5.5 M KOH concentration. But if the setting time is too short, it can lead to crack when applied to substrate. So, from the result in Figure 1, KOH concentrations of 3.5M, 4.5M, and 5.5M show acceptable setting time of less than 6 hours which can used for coating application [3]. At 3.5 M KOH concentration, setting time of 174 minutes at room temperature and 50 minutes at 60 °C were achieved and could be applied for coating application.

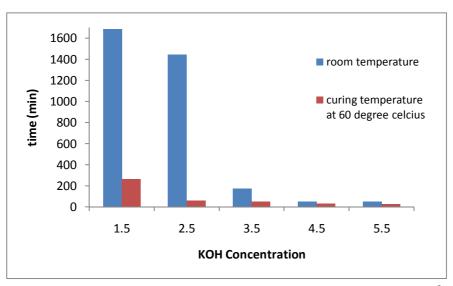


Figure 1. Setting time of geopolymer at room temperature and 60°C

B. Effect of KOH Concentration on Compressive Strength

Figure 2 shows the result of compressive strength of geopolymer samples to KOH molarity. The strength increased with increase of KOH concentration. In other words, the high K_2O/Al_2O_3 and K_2O/SiO_2 ratio promoted the strength enhancement. The highest compressive strength obtained was 17.54 MPa by using 4.5 M KOH at 60°C which is good for mechanical property for the storage purposes and to avoid the coating material from been easily ruptured [5]. From references, high concentration of NaOH and sodium silicate as additive reached compressive strength around 15.26-24.4 MPa in the same condition above [6].

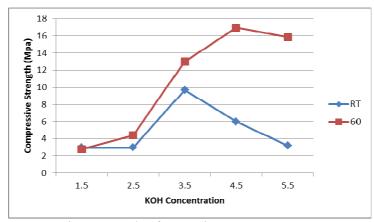


Figure 2. Compressive strength of geopolymer at room temperature and 60°C

C. Microstructure Analysis

Figure 3 shows the FESEM result of geopolymer with various KOH concentration. Reaction between fly ash and KOH increase with increasing KOH concentration. The starting materials for the geopolymeric synthesis were $K_2O:Al_2O_3$ in distilled water, which follow hydrothermal process for generating the Al_2O_3 -2SiO₂-K₂O geopolymer. Upon further inspection, our analysis revealed that geopolymers contained unreacted starting materials. Setting time was too fast, causing raw materials do not have enough time to react with the activator.

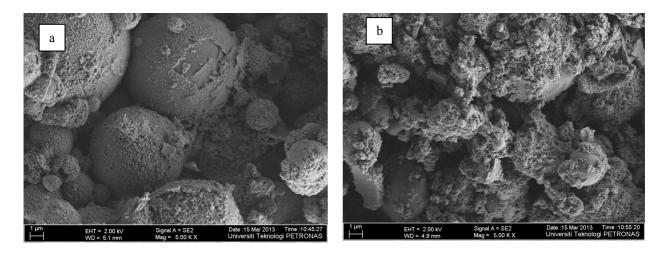


Figure 3. FESEM Result of geopolymer (a) 1.5 M KOH and (b) 4.5 M KOH

Conclusion

Based on the experimental and characterization result, KOH concentration influenced the setting time and compressive strength of geopolymer in order to have good mechanical property and good adhesion for coating purpose. The optimum setting time and compressive strength were obtained at 4.5 M KOH. For future work, this result can be developed to produce geopolymer with low alkaline activator for coating material application.

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