

Comparison of using NaOH and KOH Activated Fly Ash-based Geopolymer on the Mechanical Properties

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Abstract. Geopolymer synthesis has two main requirements to fulfil which are the source material that is rich in Silicon (Si) and Aluminum (Al) and alkali activator such as sodium/potassium hydroxide. Sodium hydroxide (NaOH) is widely used for the synthesis of geopolymer compared to potassium hydroxide (KOH) with addition of silicate solution for the purpose of increasing dissolution process. However, the comparison of using different activator in the absence of silicate solution for geopolymer synthesis is not well established. This paper presents an evaluation on compressive strength of fly ash-based geopolymer by using different activator (KOH and NaOH) with respect to different curing conditions (time and temperature) in the absence of sodium silicate. The samples were mixed using mortar mixer and prepared in 50mm x 50mm x 50mm mould for determination of compressive strength. It can be observed that the highest compressive strength up to 65.28 MPa was obtained using NaOH. Meanwhile, synthesis using KOH only recorded 28.73 MPa. The compressive strength was better when cured at elevated temperature (60°C) than room temperature (25°C). Further analysis on the microstructure of the highest compressive strength geopolymer samples for both activators was carried out using Field Emission Scanning Microscopy (FESEM) and Raman spectroscopy.

Introduction

In recent years, there has been an increasing interest in geopolymer or inorganic polymer. Geopolymer is a new group of materials that are produced from chemical reaction between alumina-silicate oxides and alkali metal. Geopolymer has amorphous to semi-crystalline three-dimensional polymeric structure and have been widely studied to replace Ordinary Portland Cement (OPC) and as coating material. The geopolymer has 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 thus making it a potential material for various application [1].

Two main constituents of geopolymer are the source material that is rich in silicon (Si) and Aluminum (Al) and alkali activator. The source material can be natural minerals or by products materials that contains Si and Al. Alkali activator is required for dissolution of solid Si and Al from the source material to the aqueous phase [2]. The type of alkali solution plays an important role for the dissolution process. It was reported that sodium hydroxide (NaOH) is widely used for the synthesis of geopolymer compared to potassium hydroxide (KOH). The difference between KOH and NaOH is due to ionic size where Na⁺ is having smaller ionic size compares to K⁺. The size of Na⁺ is 116 pm while K⁺ is 152 pm. Due to that, Na⁺ will be more active thus will enhance the dissolution process of alumino-silicate minerals [3].

Most studies in the geopolymer have also focused on using silicate solution together with the alkali solution for activation process [4], [5]. The purpose of adding silicate solution is to provide Si ions in aqueous phase and helpful in activating the precursor of geopolymer material therefore can increase the dissolution process and produced good mechanical properties [6]. Besides adding the silicate solution to improve the dissolution process, type of activator also does affect the dissolution process thus the mechanical properties.

The purpose of the current study was to compare the effect of using different alkali activator on the compressive strength for silicateless solution synthesis and fly ash was used as source material. To date there has been little info on the compressive strength of geopolymer with the absence of silicate solution in the geopolymer synthesis[7]. Eliminating the use of silicate solution in the synthesis can produce greener material. In addition, the compressive strength development with respect to curing time and curing temperature was investigated.

Experimental procedure

Materials

Fly ash (FA) of Class F type from local power plant was used as source material (Table 1). The sodium hydroxide (NaOH)/potassium hydroxide (KOH) solution of 8M concentration was used as the activator. The alkali activators was prepared 24 hours prior to use by dissolving NaOH/KOH pellets in distilled water in order to ensure the heat produced from the reaction is dissipated.

Table 1: Chemical Composition of fly ash

| Component | Chemical composition (%) |
|--------------------------------|--------------------------|
| SiO ₂ | 44.52 |
| Al ₂ O ₃ | 22.49 |
| Fe ₂ O ₃ | 11.58 |

Geopolymer synthesis

The geopolymer synthesis was done by mixing alkali activator and fly ash using a mortar mixer manufactured by ELE International. The geopolymer paste was placed in the 50x50x50mm mould for compressive strength and cured in 60°C and room temperature (25°C). Summary of parameters studied is shown in Table 2 and mix proportion to prepare geopolymer is depicted in Table 3.

Table 2: Summary of synthesis parameters

| Raw material | Fly ash |
|-------------------------------------------|-----------------------------------------------------|
| Ratio of Solid/Liquid (NaOH/KOH solution) | 3 |
| Alkali Activator | 8M of NaOH concentration 8M of KOH concentration |
| Curing time (days) | 1,7 , 14 and 28 days |
| Curing temperature | Room temperature and 60°C |

Table 3: Mix proportion of geopolymer

| Composition | Alkali Concentration (M) | Fly Ash (g) | KOH solution (g) | NaOH solution (g) | Si/Al ratio |
|-------------|--------------------------|-------------|------------------|-------------------|-------------|
| 100% FA | 8 | 1000 | 500 | 0 | 1.86 |
| 100% FA | 8 | 1000 | 0 | 500 | 1.86 |

Testing and Analysis Methods

The compressive strength test was determined using compression strength machine manufactured by ELE International with loading rate of 0.9kN/s according to ASTM C-109. The mean strength of three measurements was recorded. The geopolymer samples from the compressive strength were further analysed using Field Emission Scanning Microscopy (FESEM) and Raman spectroscopy.

Results and Discussion

Effect of different alkali activator

Compressive strength is an indicator by researchers on the success of geopolymer. Figure 1 shows that geopolymer samples using sodium hydroxide (NaOH) exhibit the higher compressive strength compared to KOH regardless of curing condition (temperature and time). The highest compressive strength obtained for NaOH is 65.28 MPa while KOH recorded 28.73 MPa. The result obtained is supported with the literature where as smaller ionic size of Na^+ makes it more active compared to K^+ and favor better dissolution process of raw material [3]. Due to higher extent of dissolution of alumina-silicate minerals in NaOH solution, more oligomers will be formed and developed high compressive strength as shown in Fig. 1. This reveals that different alkali activator gives significant impact on compressive strength even without addition of silicate solution.

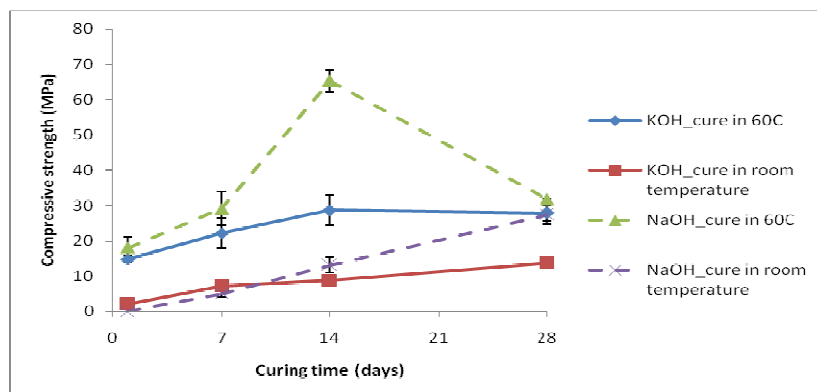


Fig. 1: Compressive strength vs. curing days using different activator and curing temperature

Besides that, the samples cured in room temperature show that the compressive strength is gradually increasing as curing time is increased for both alkali activators. Without the addition of silicate solution, the compressive strength seemed to improve when cured at longer time. Longer curing time seems to increase the geopolymerisation process and develop better compressive strength. It can be concluded that silicate solution is not necessary to be added as the improvement of mechanical properties can be observed by prolonging the curing time for both activators. Similar trend was reported in previous literature with the addition of silicate solution [4], [8], [9]

However, the samples that were cured in 60°C shows that the compressive strength is increased up to 14 days but started to decrease later as shown in Figure 1. The supply of heat seems to accelerate geopolymerisation process until it reaches optimum strength at 14 days. However, prolong curing time after 14 days in elevated temperature result in destruction of geopolymer bonding and produced lower compressive strength [10].

The samples that recorded the highest compressive strength for both activators were further analysed using FESEM. It was observed that microstructure using NaOH in Fig.2 (a) is denser and compact compared to KOH (Fig. 2 (b)) thus leading to high compressive strength. Meanwhile, Fig. 2 (b) shows the microstructure is more porous and result in lower compressive strength. This is conforming to the result obtained in Fig. 1, which using NaOH produced the highest compressive

strength. Besides that, it was proven the usage of NaOH produced more crystalline structure compared to KOH. The crystalline structure was arranged in a well-structured form therefore can contribute to high compressive strength. Concisely, the different activator does influence the microstructure of geopolymer.

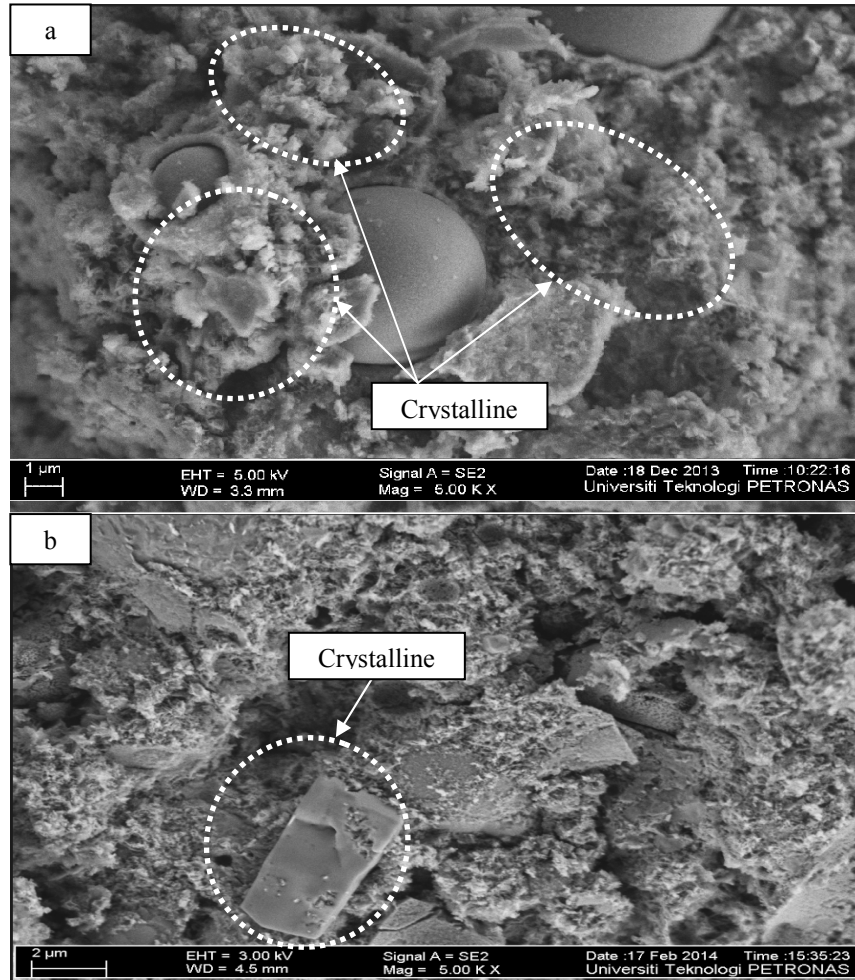


Fig.2: Microstructure of geopolymer samples cured at 14 days in 60°C (a) NaOH (b) KOH

Raman Spectroscopy

Fig. 3 shows the Raman pattern for geopolymer products that are produced high compressive strength for both using KOH and NaOH. The band between 1130- 1140 cm^{-1} as depicted in Fig. 4 was due to the stretching mode of T-O-T (where T=Si or Al) bond. This analysis support that NaOH has higher solubility of Al^{3+} and Si^{4+} and better dissolution process due to the earlier formation of T-O-T (where T=Si or Al) at 1134 cm^{-1} compared to KOH [5]. The earlier formation of structure develops better compressive strength as shown in Fig.1.

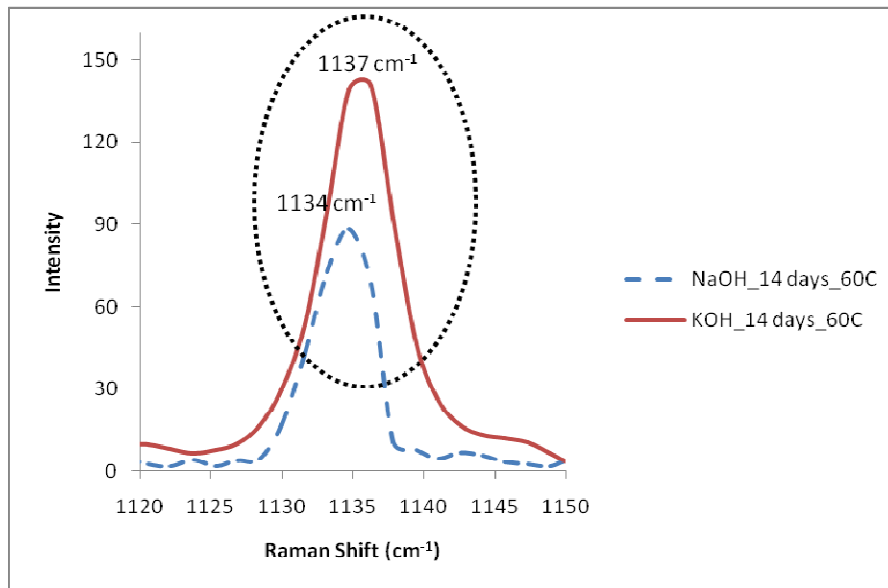


Fig. 3: Raman Analysis for highest compressive strength produced using KOH and NaOH

Conclusion

The results of this study show that high compressive strength can be obtained with the absence of silicate solution. The mechanical property of geopolymer was affected by the activator used as NaOH exhibit better compressive strength compared to KOH. The highest compressive strength of 65.28 MPa was achieved by using NaOH when cured at 60°C for 14 days meanwhile KOH only recorded 28.73 MPa. It was shown that curing at 60°C results in better compressive strength compared to curing at room temperature.

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