Concentration of NaOH and the Effect on the Properties of Fly Ash Based Geopolymer

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Abstract

Synthesis of geopolymer matrixes involved utilization of aluminosilicate materials and alkali activators as the main starting materials where the former could be fly ash, slag or kaolin and the latter commonly used are sodium hydroxide (NaOH) and sodium silicates (Na$_2$SiO$_3$) respectively. In this work, fly ash was chosen as one of the raw material in geopolymer preparation. This is due to the fact that millions tons of fly ash generated yearly by coal-fired power plants and the management of this substance is part of the matter of concern. Hence, one of the approaches to address this issue is by turning the industrial byproduct (fly ash) into a valuable resource material in geopolymer production. This paper will further describe the effect of NaOH concentration towards the properties of fly ash based geopolymer product. Geopolymer samples were prepared under fixed curing temperature (60°C) and curing time (1 day). The influence of NaOH concentration in the range of 4 M to 18 M was systematically studied using Fourier Transform Infrared Spectroscopy (FTIR) for structural elucidation, Scanning Electron Microscope (SEM) to observe the morphology and determination of the mechanical properties (flexural strength) was carried out by Universal Testing Machine (UTM). Based on the results obtained, the optimum NaOH concentration (12 M) at which geopolymer exhibits the best mechanical properties was attained.

Keywords: Geopolymer; Concentration; Sodium Hydroxide; Fly ash

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1. Introduction

Geopolymers was first introduced by Davidovits back in 1972 where based on the research studies, it was feasible as a new material in cement production for the future [1]. Over the last decades, research on this inorganic polymer widens where it shows promising use in various application namely toxic metal immobilization, waste management, fire resistance, construction repair and coatings [2]. Geopolymer material has captured the interest of many researchers from different industries because it demonstrates excellent properties such as resistance towards fire, acid, thermal and etc [3]. Moreover, it is green due to the fact that it can be prepared from industrial waste and natural resource. Further advantages of geopolymer is the production of is very economical and cost effective as the waste is available at low cost and the process is hassle free [4].

Any aluminosilicate source (such as metakaolin, kaolin, slag and fly ash) that can dissolve in alkaline activator solution (such as NaOH or KOH) will act as geopolymer precursor and geopolymerise [5]. Researchers opted to utilize industrial by product for instances, slag and fly ash as the source materials for geopolymer because they have high silica and alumina contents which also abundantly available in landfill site [6]. Whereas, the alkaline solutions play role in geopolymerization at the early stage as it dissolve the active aluminosilicate species in the reaction [7].

Geopolymerization occur in few stages where the first stage involves in the releasing of silicate (SiO$_4$) and aluminate (AlO$_4$) as starting materials which activated by alkali and results in geopolymer gel as the final yield [8]. Hydrolysis took place in the second stage where the water molecule presence helps to further break the bond and allow these SiO$_4$ and AlO$_4$ tetrahedral units to link with each other yielding polymeric precursors. Polycondensation occurs in the final stage where the geopolymer gel solidified and formed three-dimensional aluminosilicate network [9].

Synthesis of geopolymers was affected by different types of raw materials, concentration and alkaline activator involved, curing time and temperature plus any other factors. These will affect the formation of aluminosilicate geopolymer network and consequently the properties of the end product [10]. Geopolymer is well known of its unique characteristic where it can be prepared exclusively to fulfill the demand required in specific application. Hence, the main objective of this study is to prepare thin geopolymer sample from different NaOH concentration and the influence towards its flexural strength.

2. Experimental Method

2.1. Materials

An industrial byproduct namely fly ash as the source of aluminosilicate was obtained from one of the local power station. Composition and concentration percentage of elements present in fly ash were determined by X-ray fluorescence (XRF) analysis. Alkali activator solution utilized in this study was prepared by dissolving specified amount of sodium hydroxide pellets (MERCK) with distilled water to achieve concentration in the range of 4M to 18M. The dissolution process involved exothermic reaction hence the mixture were cooled down to room temperature for 24 hours before it can be used.

Table 1. Percentage of elements presence on fly ash

<table>
<thead>
<tr>
<th>Formula</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>43.73%</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>20.18%</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>12.37%</td>
</tr>
<tr>
<td>CaO</td>
<td>11.14%</td>
</tr>
<tr>
<td>MgO</td>
<td>3.75%</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>1.96%</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>1.45%</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>0.93%</td>
</tr>
</tbody>
</table>
2.1 Synthesis of geopolymer

Preparation of geopolymer paste is easy where fly ash was activated by the alkaline activator (NaOH) during mixing process. Overhead mixer was used to perform such procedure for few minutes in order to yield a homogenous mixture of geopolymer paste. After completion of previous mixing stage, the geopolymer paste were moulded in an acrylic mould for testing and analysis purpose. The end product were then exposed to 60°C and cured for 1 day for flexural strength.

2.2 Test and analysis method

Infrared spectra were recorded from 4000cm\(^{-1}\) to 400 cm\(^{-1}\) using Perkin Elmer and the specimen were prepared using KBr pellet technique. Field Emission Scanning Electron Microscope (FESEM) model Veiss SUPRA was utilized to study the morphology of the raw material (fly ash) and geopolymer paste. Flexural strength measurements were conducted using three point bending fixture using Universal Testing Machine (UTM) with Al-7000S model with a span length of 48 mm at a crosshead speed of 1.28 mm/min. Load displacement curves recorded.

3. Results and Discussions

Characterization of geopolymers was carried out by FTIR as one of the tools to indicate the formation of geopolymers from the raw materials (fly ash). The FTIR spectra in Figure 1 (a) of the end product (geopolymers) display some significant differences as comparison to the spectrum of the starting material (fly ash). During the reaction, the band at 792.28 cm\(^{-1}\) due to AlO\(_4\) disappears whereas the band at 1095 cm\(^{-1}\) due to asymmetric stretching Si-O-Si and Al-O-Si in fly ash shifts to lower frequencies (992 cm\(^{-1}\)). These bands are particularly sensitive to geopolymer gel structure indicating that formation of geopolymers took place in the reaction. In geopolymers materials, band appeared in the region of 1600 and 3450 cm\(^{-1}\) which were assigned to bending vibrations (H-O-H) and stretching vibration (\(-\)OH), respectively. These bands indicate the presence of bound water molecules [11]. In addition one new band appeared at the region 1420 cm\(^{-1}\) due to formation of sodium carbonate. However, there were no significant shifting can be observed in geopolymers prepared from various concentration of NaOH which depicted in Figure 1 (b).

![FTIR spectra](image)

**Fig.1.** FTIR spectra of (a) Fly ash and OFA -12 and (b) geopolymer prepared with different NaOH concentration

The morphology of fly ash displayed in Fig.2 (a) where the FESEM micrograph shows round and sphere shape of fly ash particles with different sizes (large, medium and small). Additional to that, geopolymer formation can also be determined by FESEM where the morphology of geopolymer gel portrayed in the image of Fig.2 (b). In geopolymer gel, there are partially unreacted fly ash particle, due to the incomplete chemical reaction during
geopolymerization which might be contributed by the smooth, glassy and inert surface of the large size of fly ash.

Fig. 2. Fesem Micgroprahs of (a) Fly ash and (b) Fly ash based geopolymer (12M NaOH Concentration)

Mechanical properties of geopolymers samples were identified by performing flexural test using Universal testing machine (UTM). The results of flexural strength displayed in Fig.3 shows increasing pattern as the NaOH concentration utilized in geopolymer synthesis increases from 4M to 12M. However, higher concentration of 14M, 16M and 18M shows decreasing flexural strength. Geopolymers prepared from the 4M NaOH exhibited lowest flexural strength (3.193 Mpa) whereas NaOH concentration of 12M gave high flexural strength of 10.119 Mpa. Dissolution of silica and alumina from fly ash were greatly influenced by NaOH concentration where it has been shown that increasing molarity will increase the dissociation of the active species of raw material and yielding formation of more geopolymer gel network [12]. However, too high NaOH concentration may disrupt the geopolymerization process due to the excessive quantity OH ions which lead to inefficient reaction.

Fig. 3. Flexural strength of geopolymer prepared with NaOH concentration ranging from 4 to 18 M.

4. Conclusion

Analysis based on FTIR and FESEM had qualitatively shown that geopolymerization took place and the geopolymers matrix had been formed. This is due to the presence of significant band appeared and from the images captured respectively. Mechanical properties of geopolymers demonstrated by flexural strength shows that, NaOH concentration gave a significant impact in enhancing the properties where the optimum concentration of NaOH of 12 M gave better strength properties of geopolymer.
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