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Determination of Excess Sodium Hydroxide in Geopolymer by Volumetric Analysis

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Abstract

Efflorescence in geopolymers can be a significant issue as the binder contains much higher soluble alkali metal concentrations than conventional cements. This paper reports the quantification method used to evaluate efflorescence potential on fly ash based geopolymer from the leaching behaviour of alkali metal in the solution followed by volumetric analysis. In this study, NaOH was used as the only alkaline activator. Geopolymer synthesis at higher molarity of NaOH gives higher percentage of excess sodium hydroxide content in geopolymer, indicating the higher potential of efflorescence. Meanwhile, the effect of hydrothermal curing condition at the same alkaline molarity results in the lowest percentage of 4.84% excess sodium hydroxide content in geopolymers compared to others. The systems were subjected to 65% relative humidity at 80°C for 5 days. An optimum Na₂O content along with hydrothermal curing condition can result in optimization of geopolymerization reaction.

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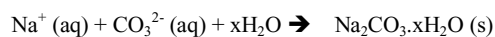
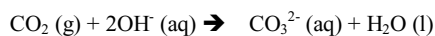
1. Introduction

Sodium hydroxide (NaOH) is the most commonly used alkaline activators in geopolymer synthesis. The extensive use of NaOH as an activator in geopolymer synthesis, from both fly ash and metakaolin precursors is due to its low cost, abundance and low viscosity. Furthermore, leaching of Al³⁺ and Si⁴⁺ ions are generally high with NaOH solution compared to potassium hydroxide (KOH) solution [1]. As an activator, NaOH accelerates the reaction more rapidly. Sodium hydroxide provides both hydroxide anion (OH⁻), which act as a catalyst for dissolution of the aluminosilicates in the first stage and sodium cation (Na⁺) which acts as structure-forming element, balancing the negative framework charge carried by tetrahedral aluminium in the last stage [2]. NaOH was found to significantly affect both the compressive strength and structure of the geopolymers [3].

However, sodium aluminosilicate geopolymers, can suffer from unsightly efflorescence caused by excess sodium hydroxide unreacted in the material, particularly those synthesized at high Na₂O/Al₂O₃ ratio [5]. The reason for efflorescence in geopolymer is due to the open microstructure of some materials which have a lower extent of reaction, resulting in excess or unreacted alkaline solution. It is partly due to the weak binding or exchangeability of sodium in the geopolymer structure as well [6]. Temperature and humidity influence the content of water (H₂O) in efflorescence product (hydrated alkali carbonates). The mechanism of efflorescence formation is outlined below [4]:

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Under natural carbonation condition, dissolved CO_2 acts as an acid and consumes hydroxides and therefore a partial neutralization process. The main factor for efflorescence in the geopolymer synthesis is due to the availability of mobile Na^+ and OH^- in geopolymers binders [4].

Thus, a research had been done to evaluate the excess of sodium hydroxide in geopolymer formation. This work quantified the excess NaOH in fly ash geopolymer by direct leaching analysis and indirectly by volumetric analysis. In this work, fly ash and sodium hydroxide were used as raw materials to produce geopolymer. Besides, this present work examined the effect of curing condition to the excess sodium hydroxide content in geopolymer synthesis at constant $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ ratio.

2. Methodology

2.1. Materials

Fly ash (FA) of class F was obtained from a nearby coal power. Research-grade NaOH (99%) was purchased from Merck and made into 6,8,10,12,14 and 16M solutions using distilled water. The solutions were kept for 24 hours prior to use. In volumetric analysis acid-base titration, 1M of hydrochloric acid (HCl) solution were used as a titrant.

2.2. Geopolymer Synthesis

All geopolymer prepared were synthesized by maintaining the solid to liquid ratio of 3:1 by weight. 20g of sodium hydroxide solution as alkaline activator was added into the 60g fly ash. The samples were mixed using mechanical mixer for 5 minutes. The fresh geopolymer paste was quickly casted in 178 x 175 x 3 mm flexural mold and was cured at 60°C for 24 hours in a drying oven. The temperature was selected based on previous work to obtain the optimum compressive strength of geopolymer.

2.3. Leaching of Alkali and Acid-base titration

The produced geopolymer was crushed into powder. The powdered sample then was mixed with 100 mL of distilled water. The pH of the solution was monitored twice a day using a MeterLab 450 pH analyser until the reading is stabilized, implying the equilibrium leaching of NaOH into the solution. The final solutions then were filtered using Filters FIORONI, Grade 601 filter paper. A 20 mL of the filtrate solution was titrated using 1M of hydrochloric acid (HCl). The moles of sodium hydroxide in titrate solution is equal to the excess of NaOH in the geopolymer formed. This is expressed in percentage (%) and was calculated using the Equation 1:

$$\% \text{ excess NaOH} = \frac{\text{weight of NaOH in titrate}}{\text{weight of NaOH in geopolymer}} \times 100$$

Equation 1

2.4. Hydrothermal Curing

The effect of curing condition at the similar molarity of alkaline activator to the leaching of excess sodium hydroxide in geopolymers also been studied. Based on previous study, the using of 10M NaOH as alkaline activator produces optimum compressive strength. Therefore, 10M NaOH is used to synthesis the geopolymer cured at 60°C. After 24 hours, the samples were taken out and were put in humidifier for 1, 2, 3 and 5 days. The systems were subjected to 65% relative humidity at 80°C. Finally, all the samples will undergo the same procedure of alkali leaching and acid-base titration.

3. Results and Discussion

Volumetric analysis is used to determine the concentration of analyte. It is a quantitative analytical method that involves the measurement of volume of a solution of known concentration. Since the sodium hydroxide is a strong base, the concentration of excess sodium hydroxides can be measured by direct acid-base titration. The acid and base react quantitatively with each other where the product are water and salts and a neutralization reaction. So, it is possible to determine the concentration of excess sodium hydroxide in aqueous solution with high accuracy.

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