The Effects of Solid to Liquid Ratio on Fly Ash Based Lightweight Geopolymer

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Abstract. Geopolymer material was used as the raw material because it promotes the green technology. In this study, lightweight geopolymer was produced using fly ash as raw material with the addition of alkali activation which is mixture of sodium silicate and sodium hydroxide, foaming agent that gives lightweight properties and finally, underwent curing process. The molarity of sodium hydroxide (NaOH) used was fixed at 12 M while the ratio of fly ash to alkali activator (solid to liquid) used were varied in the range of 2.0, 2.5, 3.0 and 3.5, by mass. Besides that, foaming agent (Polyoxyethylene alkyl ether Sulfate) was added to the geopolymer sample to give the lightweight properties. The samples were cured at 80 °C for 24 hours in the oven for curing process and left at room temperature prior for testing for 14 days. The testing of sample was conducted in this study which includes density test, compression strength test, water absorption test and scanning electron microstructure (SEM) test. The results obtained for optimum solid to liquid ratio is 2.5, by mass with the optimum value of compressive strength density value. The mechanical and physical properties of lightweight geopolymer were based on the ASTM International Standard.

1. Introduction
Geopolymer is relatively a new class of construction materials. Its materials are usually made of an aluminosilicate that acts as precursors. It will be incorporated with an activating liquid which consist of sodium hydroxide and sodium silicate solution [1]. Thereupon, reducing the emission of carbon dioxide (CO$_2$) which can be represented as an environmentally friendly innovative for cement. Moreover, this technology is getting interests in various application fields like fire resistance materials, low cost ceramics, refractory filters and lightweight panels for thermal and acoustic isolation [2].

Fly ash is commonly used and competent waste material in geopolymerization. This is because huge amount of fly ash produced worldwide. It is approximated to be around 780 million tons
annually [3]. Currently, lightweight concrete is widely well-known as it caters additional advantages to the concrete production industry. Lightweight concrete can be exemplified as a type of concrete which involves an expanding agent. This agent surge the capacity of the mixture. Hence, this gives extra properties such as workability and lower dead weight [4].

The dead load for normal concrete is higher which is approximately 2400 kg/m³. The density of conventional lightweight concrete is higher which is 1440 kg/m³ to 1840 kg/m³ [5]. However, the density of lightweight concrete varies between 300 kg/m³ to 1800 kg/m³ depending on the materials used [6]. Lightweight concrete has high flow ability, less uses of aggregates, optimum low strength, low weight and excellent thermal-insulation properties. Moreover, it also speed up the building rates in construction and lower the haulage and handling costs [7].

This research proposes to study the strength properties by the effects of fly ash to alkaline activator ratio on lightweight geopolymer. The foaming agent which is Polyoxyethylene Alkylether Sulfate is incorporated into geopolymer paste to produce a lightweight geopolymer. It is a synthetic foaming agent. This type of inorganic geopolymer has highly potential to be used as thermal insulator material and lightweight concrete in construction field.

2. Experimental
2.1. Material
The raw materials that are involved in this research are fly ash, alkaline activator and foaming agent. Sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) solution are combined together to be act as alkali activator solution. Fly ash is a by-product of coal combustion.

The type of fly ash used in this research is Class F Fly Ash. The sodium hydroxide in pallet form was diluted in distilled water to produce 12 M concentration. Sodium silicate are in the form of aqueous solution. The foaming agent used in this research was Polyoxyethylene Alkylether Sulfate. It is a synthetic foaming agent. It has high pH as it is alkali substance and has the appearance of a brown liquid and manufactured from coconut oil. This foaming agent is cheap, foams quickly and can be cleansed effectively.

2.2. Mixing Process with Different Solid to Liquid Ratio
The effect of solid to liquid ratio is investigated by fixing the ratio of sodium hydroxide (NaOH) to sodium silicate (Na₂SiO₃) at 2.5, by mass [8].

The solid to liquid ratio was fixed at 2.0, 2.5, 3.0 and 3.5, by mass. However, the ratios of 1.0 and 1.5, by mass were unable to use because of high workability of the geopolymer paste. The ratio of foaming agent to water and foam to geopolymer paste were fixed at 1:10, by volume and 1:1, by volume. The details of mix design are stated in Table 1.

The geopolymer sample was poured in (50 x 50 x 50) mm cubic moulds for all ratios. Then, the samples were cured in the oven for 24 hours at 80°C. After 24 hours, the geopolymer samples were left at room temperature for 1 day before demoulded and cured in room temperature for another 14 days before testing process.

| Ratio of fly ash to alkaline activator | Ratio of sodium silicate to sodium hydroxide | Ratio of foam to paste | Fly ash (g) | Sodium silicate (g) | Sodium hydroxide (g) |
|----------------------------------------|---------------------------------------------|------------------------|------------|--------------------|---------------------|
| 2.0                                    | 2.5                                         | 1.0                    | 468.7      | 143.49             | 67.0                |
| 2.5                                    | 2.5                                         | 1.0                    | 502.21     | 143.49             | 57.4                |
| 3.0                                    | 2.5                                         | 1.0                    | 527.30     | 140.6              | 35.2                |
| 3.5                                    | 2.5                                         | 1.0                    | 546.86     | 111.6              | 44.6                |
2.3. Curing Process
During curing process, the geopolymerization process starts to build up. Due to the increasing of temperature, polymerization become quicker and the strength can increase 70% within 3 hours to 4 hours of curing. The samples were dried by using an oven at temperature about 80 °C for 24 hours.

2.4. Mechanical and Physical Analysis
There are several physical and mechanical tests that will be done in this research which are compressive strength, density and water absorption test to determine the properties of lightweight geopolymer. These test were done after curing process.
2.4.1. Compression Strength Test. The compressive strength test for lightweight geopolymer was carried out to measure the maximum resistance of a sample specimen to the axial loading. The compressive strength was measured by using a Universal Testing Machine (UTM) in compliance with ASTM C 109-16a on the (50×50×50) mm specimens. The dry density of lightweight geopolymer can be calculated by the formula as shown in equation (1) below.

\[ \rho = \frac{M}{V} \]  

Where:
\( \rho \) = Dry density;
\( M \) = Mass of sample;
\( V \) = Volume of sample.

2.4.2. Water Absorption Test. The water absorption test was conducted accordance to ASTM C140. After the curing process, the samples are weighed and will be immersed in water for 24 hours. Then, samples were removed from the water, pattered dry, blotted with cloth to remove excess water before it will be weighed again. After weighing the saturated sample, the sample is cured in the oven at 105 °C for 24 hours.

\[ \text{Water absorption} = \left( \frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}} \right) \times 100 \]  

The water absorption was calculated based on dried weight by using equation (2) below.

2.4.3. Density Analysis. The density of lightweight geopolymer samples were calculated based on the value of mass of sample and the volume of sample. The densities were measured after fulfilling the aging period of 14 days. Equation (3) is used to measure the density.

\[ \text{Density} = \frac{\text{mass of the sample (kg)}}{\text{volume of the sample (m}^3)} \]  

Average of three samples were tested for each ratios.

3. Results and Discussions
3.1. Compressive Strength of Lightweight Geopolymer
The result of compressive strength for fly ash based lightweight geopolymer with different solid to liquid ratio was illustrated in Figure 1. The highest strength is detected at solid to liquid ratio 2.5, by mass with value of 19.3 MPa while lowest strength is observed at solid to liquid ratio 3.5, by mass with 12.1 MPa. Highest compressive strength is found to be at solid to liquid ratio 2.5 because it has the optimum amount of fly ash and alkaline activator that gives a good workability and activate the fly ash at the highest rate of the geopolymerisation process before it mixes with foam. Hence, this provided sufficient Si species and raises the ratio of SiO₂/Al₂O₃. Besides, this can be related to the less of water available as the solid to liquid ratio increased and consequence in faster hardening of gel.
phase and rapidly bond together the geopolymer framework [9]. During geopolymerization process, the SiO$_2$ and Al$_2$O$_3$ presented in fly ash need sufficient alkalis to allow the glassy structure to dissolve in alkaline medium to form the geopolymer gel structure [10].

The change in the alkaline activator content affects the (Si) species content, the sodium (Na$^+$) ions content and base water content. All these factors may affect during geopolymerization process and on the strength of the resulting geopolymer.

![Figure 1. The compressive strength of lightweight geopolymer with different solid to liquid ratio.](image)

A decrease in the solid to liquid ratio increases the Si species content, because the alkaline activator containing sodium silicate which contain high amount of Si species, increased the SiO$_2$/Al$_2$O$_3$ ratio and also increased the strength of geopolymer samples because of more Si-O-Si bonds produced which are stronger compared with Si-O-Al [11-13].

3.2. Water Absorption of Lightweight Geopolymer

The results of water absorption of lightweight geopolymer were shown in Figure 2. Water absorption is an important factor for determining the durability of concrete.

![Figure 2. The water absorption of lightweight geopolymer with different solid to liquid ratio.](image)
The less water infiltrates into concrete, the more durability of the concrete and resistance to the natural environment are anticipated. The lowest water absorption is found at solid to liquid ratio 2.5, by mass with 6.08% while highest water absorption is found to be at solid to liquid ratio 3.5, by mass with 11.61%. An increment in water absorption from 7.14% to 11.61% was observed with the increase of solid to liquid ratio from 2.5 to 3.5, by mass.

The results found that the water absorption increases with increasing of solid to liquid ratio. The previously reported by Zhao et al. [10], with the increasing volumes of foam, more and more interconnected pores which are advantage for flow of water are performed. Nevertheless, the increase of water absorption could decrease compressive strength. Based on the results, the porosity also affects the water absorption. When the porosity decreases, the pore size also decreases [14].

### 3.3. Density of Lightweight Geopolymer

The densities of lightweight geopolymer with different solid to liquid ratio are shown in Figure 3. The densities vary in the range of 1307 kg/m³ to 1800 kg/m³. The highest density is found at solid to liquid ratio 2.5, by mass with 1375 kg/m³. The lowest density is observed at solid to liquid ratio 3.5, by mass with 1267 kg/m³. The density of lightweight geopolymer has slightly decreases from solid to liquid ratio 2.5 to 3.5, by mass ranging from 1373 kg/m³ to 1267 kg/m³.

![Figure 3. The density of lightweight geopolymer with different solid to liquid ratio.](image.png)

Density at solid to liquid ratio 3.5, by mass is the lowest because more volume of foams has been used in the paste. Mass of fly ash is more higher compared to alkaline activator which means less liquid is inadequate in the paste. Therefore, to improve the workability, more volume of foams is added in the paste. The increases of voids in the geopolymer samples due to the foaming agent will reduce the density of the sample. Thus, the compressive strength will also decrease with the increment of those voids [15].

### 3.4. Microstructure of Lightweight Geopolymer

Figure 4 shows the microstructure of lightweight geopolymer with different solid to liquid ratio. The microstructure of lightweight geopolymer showed some unreacted or partially reacted fly ash particles and formed of a continuous alumina-silicate gel. For solid to liquid ratio 2.0, by mass unreacted fly ash particles are found on the microstructure. Pores also formed. This is because there is too much water from alkaline activator and foams. Therefore, the concentration of sodium hydroxide decreases resulting in lower compressive strength and pore formation on the microstructure. Cracks also
observed on the surface can lead to the reduced mechanical strength observed at these activation conditions [7].

![Microstructure of lightweight geopolymer with different solid to liquid ratio](image)

Figure 4. Microstructure of lightweight geopolymer with different solid to liquid ratio; (a) 2.0, (b) 2.5, (c) 3.0, (d) 3.5 and (e) Unfoamed.

Therefore, the concentration of sodium hydroxide decreases resulting in lower compressive strength and pore formation on the microstructure. Cracks also observed on the surface can lead to the reduced mechanical strength observed at these activation conditions [10]. For solid to liquid ratio 3.5, more unreacted particles are found on the microstructure compared to the other ratios. There were small and large pores due to incomplete reaction with fly ash, alkaline activator and foam. Microcracks were also observed which contributed to their lower strength by increasing their water absorption and porosity. The lower values of compressive strengths of lightweight geopolymer attributed to the presence of voids happen when cured in the oven. This might have resulted due to poor surface structure of the lightweight geopolymer as shown in Figure 4(d) [16-17]. For unfoamed
geopolymer, there were minimal numbers of unreacted fly ash particles found on the microstructure. This is because there were adequate amount of fly ash and alkaline activator to react with each other.

In conclusion, solid to liquid ratio 2.5, by mass has the better microstructure compared to other ratios. It has less unreacted fly ash, more completely reacted fly ash, no microcracks and tiny pores as shown in figure 4. Hence, contributing to higher strength and lower water absorption.

4. Conclusions
An investigation of the effect of different solid to liquid ratio on fly ash-based lightweight geopolymer was conducted. The effects of solid to liquid ratio were studied. From the research, the solid to liquid ratio that gives the best strength is 2.5, by mass. This ratio caters the highest compressive strength of 19.3 MPa, lowest percentage of water absorption at 7.14% and density at 1373 kg/m³ of lightweight geopolymer. The microstructure of the concrete is was smoother and denser with less unreacted fly ash particles and pores. It was shown that the content of alkaline activator solution is the most crucial parameter affecting the strength of lightweight geopolymers.

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