Mechanical and physical properties of bottom ash/fly ash geopolymer for pavement brick application

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Abstract. Geopolymers are amorphous to semi-crystalline with excellent physical and mechanical properties. It has been used to become a potential binder to Ordinary Portland Cement (OPC) in certain applications due to its lower emission of carbon dioxide gases and low energy consumption sustainability criteria. Bottom Ash (BA) is one of the main industrial by-products and it is produced at the bottom of the furnace during the coal combustion process in electricity generation. The application of BA as a sustainable construction material in the building industry plays an important role in order to decrease the volume of residual waste and conserving existing natural fine aggregates. The objectives for this study is to study the effect of fly ash to bottom ash ratio and to determine the optimum ratio of fly ash to bottom ash geopolymer for pavement brick application. The chemical composition and morphology of geopolymer reinforcement was analysed by using X-ray Fluorescence and Scanning Electron Microscope. The molarity of the Sodium Hydroxide solution is fixed at 12M. The parameter used in this study are different weight percentage of fly ash geopolymer 0 wt%, 10 wt%, 20 wt%, 30 wt% and 40 wt%. The solid to liquid ratios for this study is 2.0. The curing temperature of this study is 80°C and the curing time is 24 hours. 100% of bottom ash geopolymer is used as a control variable for this study.

1. Introduction
Geopolymer is a new type of cementitious material which has received extensive attention internationally in recent years due to its use of industry waste source material and lower carbon dioxide (CO2) emission during the material production [1]. The basic structure of geopolymer is silicon-oxygen tetrahedron and aluminium-oxygen tetrahedron that are connected by oxygen atom [2] [3]. Geopolymers are formed in high alkaline medium which releases Si and Al species through geopolymerization reaction involving the dissolution of aluminosilicates sources where the formation of small coagulates structures, gelation and lastly hardening to form hard solid. This reaction produced amorphous to semi-crystalline three dimensional polymeric structures containing Si-O-Al. This is suitable for materials that containing alumina and silica barrier phases such as natural rocks or secondary raw materials [4-8].
Bottom ash is a part of non-combustible residue of combustion in a furnace or incinerator. It was produced in coal combustion and it sticks to hot side walls of coal-burning furnace and produces traces of combustible embedded in forming clinkers [9]. It contains ferrous (Fe) and non-ferrous (NFe) metals that can be recovered. It has high potential to be used in construction as lightweight aggregate as it has various properties such as high porosity, low shrinkage value, low density, good fire and chemical resistance and good immobilization of heavy metal [10]. The physical characteristics of bottom ash is determined by some factors which are type and quality of coal source, pulverized fineness and operating conditions of power plant. The particle of bottom ash is porous textures in angular shapes and dark colour [11].

Fly ash is a by-product of pulverized coal combustion in electric power generating plants. It reacts aggressively with calcium hydroxide to form compounds with cementitious value which increases concrete strength [12]. Selecting fly ash in concrete will attain environmental, economic and also structural benefits as increasing strength and fire resistance of concrete structures [13]. Adding of fly ash to concrete could improves mechanical properties of concrete at late ages but obtains lower strength up to 28 days. The reduction in early strength is a function of replacement percent. The mechanical properties and abrasion resistance showed continuous and significant improvement at the ages of 91 and 365 days due to the pozzolanic reaction of fly ash [14].

In this research, the effects on the addition of fly ash to the bottom ash-based geopolymer were studied based on the mechanical and physical properties of geopolymer mixtures. The properties of geopolymer was followed the American Society for Testing and Materials (ASTM) Standard for pavement brick application.

2. Experimental
2.1. Raw Materials
Raw materials used in this research were fly ash, bottom ash and alkaline activator. In this research, the alkaline activator used is combination of sodium hydroxide solution (NaOH) and sodium silicate solution (Na$_2$SiO$_3$). Fly ash and bottom ash are by-product that produced from coal combustion was supplied by Cement Industries of Malaysia Berhad (CIMA). Class C fly ash was used in this study. The sodium hydroxide in pallets form was used and dissolved in distilled water for one day to produce specific molarity concentration of sodium hydroxide solution. Then, the sodium hydroxide solution was added with sodium silicate solution to form an alkaline activator solution.

2.2. Mixing Process
First, bottom ash was mixed with fly ash at a different parameter (10 wt%, 20 wt%, 30 wt% and 40 wt%). Then, the mixture of bottom ash and fly ash were mixed with alkali activator solution for about 5 minutes or until achieved the homogeneity. The molarity of NaOH solution used is 12 M based on the previous study done by Wan Mastura et al., [15]. The mass ratio of Sodium Silicate to Sodium Hydroxide of 2.5, by mass was used and the ratio of solid to liquid ratio of 2.0, by mass was used in this research. The mixture was then poured into a 50 mm x 50 mm x 50 mm cube moulds in accordance with ASTM C109 [16] and covered with plastic film to avoid moisture evaporating during heat curing. The samples were cured in the oven for 24 hours at temperature 80˚ C.

2.3. Testing and Characterization
2.3.1. Chemical Composition Analysis
Chemical composition analysis has been done by using X-Ray Fluorescence Spectrometer (XRF). X-ray fluorescence spectrometer (XRF) is the analysis to get the information or study the element contained in raw materials. It is to determine the oxides content and percentage of the elements.
2.3.2. Microstructure Analysis
Scanning electron microscopy (SEM) model Hitachi TM 3000b is used in this research to analyse the microstructure on the surface of the geopolymer. This testing was undergone after the compressive test to determine the surface structure. The magnification used in this research are 100x, 500x and 1000x.

2.3.3. Compressive Strength Test
The compressive strength test was measured by using a Universal Testing Machine (UTM) in compliance with ASTM C 109 [16] on the (50×50×50) mm specimens. Compressive strength testing is the maximum compressive stress that under gradually applied load a given solid material will sustain without fracture. The samples were tested at 7th days of ageing. The three samples for each different percentage was compressed by using UTM machine and the results were recorded.

2.3.4. Water Absorption Analysis
Water absorption test was performed on 50×50×50 mm cube after 7 days of ageing according to ASTM C 140 [17]. Water absorption test is used to determine the amount of water absorbed under specified conditions. The formula in Equation 1 is used to measure the water absorption.

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

(1)

2.3.5. Density Analysis
Density measurement usually performed to determine the density of the samples. This measurement was performed by dividing the mass of the samples to the volume of the samples. The formula in Equation 2 is used to measure the density.

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

(2)

3. Results and Discussion
3.1. Chemical Composition Analysis of Raw Materials
In this research, X-Ray Flourescence (XRF) analysis was analysed to characterize the chemical composition of the raw material. Each composition of bottom ash and fly ash can be summarized in Table 1 below.

| Compound | Bottom ash (wt. %) | Fly ash (wt. %) |
|----------|-------------------|-----------------|
| SiO2     | 51.27             | 30.80           |
| Al₂O₃    | 24.70             | 13.10           |
| Fe₂O₃    | 4.31              | 22.99           |
| TiO₂     | 1.24              | 0.90            |
| CaO      | 7.46              | 22.30           |
| MgO      | 2.19              | 4.00            |
| P₂O₅     | 0.40              | 1.31            |
| SO₃      | 5.57              | 0.49            |

The main constituents of bottom ash geopolymer and fly ash geopolymer are SiO2 and Al₂O₃ which is about 76% wt for bottom ash and 44% wt for fly ash. The variation in the principle constituents is about silica (25% wt-60% wt), Alumina (10% wt-30% wt), and Iron Oxide (5% wt-
25% wt). This is the expected composition since Silica and Alumina were compulsory for the geopolymerization process. Materials with high amount of Silica and Alumina content are good for binding action [18]. According to ASTM C618 [19], When the sum of silica and alumina of fly ash is less than 50 wt% and the CaO is more than 20 wt%, the fly ash was classified in class C.

Besides that, the percentage of Fe₂O₃ also higher for fly ash compared to bottom ash. Dakhane et al., [20] stated that Fe₂O₃ may effect on the strength development of the sample. Due to the both bottom ash and fly ash are rich in silica and alumina content, these materials are suitable as the source material to produce geopolymer for pavement brick application.

3.2. Microstructure Analysis

The Scanning Electron Microscopy (SEM) was used to analyse the microstructure of bottom ash, fly ash, and geopolymer to characterize the shape and surface texture of the mixture particles, and to acknowledge the behaviour of the materials. Figure 1 (a – e) present the microstructure of five different proportions of bottom ash/fly ash which were 0 wt%, 10 wt%, 20 wt%, 30 wt% and 40 wt%.

![Microstructure images of five different proportions of bottom ash/fly ash geopolymer](figure1.jpg)

**Figure 1.** Microstructure images of five different proportions of bottom ash/fly ash geopolymer: a) 0 wt% b) 10 wt% c) 20 wt% d) 30 wt% e) 40 wt% under the magnification of x500.
Figure 1 (a) shows the 0 wt% of fly ash sample, indicating that it was 100 wt% of bottom ash, was used as the controlled sample. It showed that there were vast amount of cracks and pores existed. This is because, bottom ash had irregular shape, was rough and had gritty textural surface [10]. Figure 1 (b) shows the 10 wt% of fly ash geopolymer sample, it showed that the alkaline activator did not react completely with the mixture of ashes. It also showed the existence of voids and cracks but lesser than Figure 1 (a). Figure 1 (c) shows the 20 wt% of fly ash geopolymer sample, it showed that the number of porosity and crack still existed but in decreasing trend. Figure 1 (d) shows the 30 wt% of fly ash geopolymer sample. It showed that the number of cracks and pores decreased but still there were few small cracks can be seen. Figure 1 (e) shows the 40 wt% of fly ash geopolymer sample. It showed that there was less porosity found on the surface. The alkaline activator also reacted with the particle and there were still a few fine round particles which were not reacted or partially reacted that still can be seen.

The best microstructure that can be seen was at Figure 1 (e) which represented by proportion of fly ash geopolymer of 40% wt, shows denser and compact due to good reaction between the alkaline activator solution and the source material occurred. This was proved by the excellent results in compressive test.

3.3. Compressive Strength
The mechanical properties of bottom ash was evaluated through compression test. Several tests have been carried out to study the strength of the bottom ash at different weight percentage of fly ash added. In general, it has been observed that the compressive strength of the samples have been increased linearly with decrease of the percentage of fly ash added as shown in Figure 2.

![Compressive strength graph](image)

**Figure 2.** Compressive strength of five different proportions of bottom ash/fly ash geopolymer.

From the graph, the 0 wt% of fly ash geopolymer sample which act as the controlled sample presented lowest strength which is 3.136 MPa. This is due to the surface of the bottom ash was coarse and there were a lot of porosity, voids and cracks were found as shown in Figure 1 (a). Furthermore, the low strength produced also due to the non-completion of hydration between the alkaline activator and the bottom ash during the curing process. However, when the percentage of fly ash added with 10 wt%, 20 wt%, and 30 wt%, the strength of the sample increased to 15.250 MPa, 19.146 MPa and 24.531 MPa, respectively. The highest compressive strength obtained was 35.883 MPa when the weight percentage of fly ash added was 40 wt%. This can be proved by the microstructure of the geopolymer sample where the alkaline activator reacted completely with the mixture of bottom ash and fly ash. The cracks, voids and porosity has been decreased obviously as can be seen in the microstructure (Figure
The addition of fly ash favors in increasing the strength during geopolymerization reaction process. It can be observed that the increase of fly ash increased the strength of all mixtures. This is because the reaction of fly ash with the bottom ash during hydration. The increase of fly ash content in the mixture resulted in chemical reaction and hence increased the strength [21].

3.4. Density Test

The physical properties of bottom ash also assessed through density test. Based on the observation, the density of the geopolymer sample shown the small increment from 0 wt% to 10 wt% which is 1446.56 kg/m³ to 1526.56 kg/m³. Then, the density increased slightly to 1830 kg/m³ at 20 wt% of fly ash. However, the density of the bottom ash decreased drastically to 1553.6 kg/m³ at 30 wt% of fly ash before it speeded to 1884.8 kg/m³ at 40 wt% of fly ash added. The results of the density test have been plotted in Figure 3.

![Figure 3. The relationship between the weight percentage of fly ash and the density test.](image)

From the graph, it can be observed that the lowest density recorded is when the weight percentage of fly ash is at 0 wt% which is 1446.56 kg/m³. The formation of large pores occurred at 0 wt% contributed to the lowest density. This results related to the lowest compression strength of the sample at 0 wt%. Besides, lowest density also due to the poor geopolymerization coalescence in pore formation results in larger pores [22]. The highest density has been recorded at the weight percentage of fly ash is 40 wt% which is 1884.8 kg/m³. This is due to the uniform pore formation due to the effective geopolymerisation reaction process. The porosity of the sample is decreased, indicating to the geopolymer formed in hydration reaction gradually filled the pores and it became denser [23]. This can be proved from the compression strength where the strength of the sample was at highest at 40 wt%.

3.5. Water Absorption Test

The physical properties of bottom ash were evaluated through water absorption test. Based on the observation, the pattern of the water absorption had increased slightly at weight percentage of fly ash 0 wt% from 7.343% to 8.518% at weight percentage of fly ash 10 wt%. Then it declined drastically from 8.518% to 3.634% at 20 wt%. However, the trend increased drastically back to 8.228% at 30 wt% before it decreased slightly when the weight percentage of fly ash at 40 wt% which is 6.048%. The trend of the water absorption test have been summarized in Figure 4.
Figure 4. The relationship between the weight percentage of fly ash and the water absorption.

Based on the graph above, it can be concluded that the water absorption was at highest when the weight percentage of fly ash at 10 wt%. The water absorption rate is highest due to the increase in porosity. The water absorption rate received the maximum value when the porosities were found inside the sample. This can be proven from the morphology analysis where a vast amount of porous, voids and cracks can be found there. When the water absorption rate is increase, it will affect the density of the sample thus it will also affect the strength of the sample. This is why the compression test of the sample weight percentage of 0 wt% - 10 wt% were at lowest. However, during the water absorption rate at 30 wt% it had showed that the trend was drastically declined. This is due to the formation of pores became smaller and the water absorption rate became slower. Water absorption is a key factor affecting the durability of pavement brick [24]. The geopolymerisation process took place effectively during the reaction thus making the strength of the sample increased. The increase in density will attribute the particle size distributions and affected the compounds particles became denser and more reasonable in decreasing the porosity and the pore sizes inside the geopolymer [25].

4. Conclusion
The comparison between the microstructure, chemical composition, compressive test, density test and water absorption test have been successfully evaluated throughout this research. The influence of adding the fly ash into the bottom ash geopolymer can refine the microstructure and improve the strength of the geopolymer as long as the mixtures were homogenously mixed according to the parameter. The effects of fly ash to the bottom ash ratio can be seen in many aspects. Fly ash helped to increase the mechanical and physical properties of bottom ash geopolymer in term of strengths, microstructures, surface, density and the water absorption. The highest compressive strength that have been recorded in the research was 38.883 MPa compared to the controlled with 100 wt% of bottom ash geopolymer which was 3.136 MPa. Overall, it can be concluded that 40 wt% of weight percentage of fly ash geopolymer gives better properties and suitable to be used as pavement brick application.

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