

STUDY OF FLY ASH GEOPOLYMER AND FLY ASH/SLAG GEOPOLYMER IN TERM OF PHYSICAL AND MECHANICAL PROPERTIES

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Abstract

This article presents the mixing parameters of fly ash geopolymer based on NaOH concentration (6M - 14M), S/L ratio (1.0 - 3.5), Na₂SiO₃/NaOH ratio (1.0 - 3.0) and ladle furnace slag replacement (10% -40%). Additionally, a comparative study between fly ash geopolymer and fly ash/slag geopolymer with respect to the bulk density, water absorption, apparent porosity and compressive strength were investigated. The general bulk densities of fly ash/slag geopolymer were higher than fly ash geopolymer. The apparent porosity and water absorption of fly ash/slag geopolymers were comparatively lower than fly ash geopolymers. High compressive strength achieved by fly ash/slag geopolymer was contributed by high bulk density and low apparent porosity and water absorption. In other words, fly ash geopolymer obtained lower strength was due to lower bulk density and higher apparent porosity and water absorption.

Keywords: parameters, fly ash geopolymer, fly ash/slag geopolymer

Introduction

Nowadays, industrial by-products are increasing attention due to its disposal problem where they could occupy the landfill space. Fly ash and slag are some types of industrial solid wastes. Fly ash can be sorted by Class F (fine) and Class C (coarse) [1]. Slag can be classified by iron slag (ground-granulated blast furnace slag [2]) and steel slag (basic oxygen furnace slag, electric arc furnace slag, and ladle furnace slag [3]). The current daily production of fly ash and slag in Malaysia itself recorded 1,620 tons [4] and 7.5 tons [5], respectively. Hence, recycling the fly ash and slag in the construction field is a strategic way to solve the issues of disposal at the same time decrease the environmental impacts.

Numerous researchers have been utilized various aluminosilicate materials (such as metakaolin [6], kaolin [7], fly ash [8] and slag [3]) and alkaline solution (such as sodium and potassium-based [9]) to synthesize aluminosilicate geopolymer. Hence, the type of aluminosilicate sources used, the type of alkaline activator used and the mixing parameters are making attention nowadays. For instance, sodium hydroxide (NaOH) and potassium hydroxide (KOH) concentration were a key parameter affecting the mechanical strength [10-12]. Cai et al. [12] concluded that increasing KOH concentration between 4M to 16M based on metakaolin geopolymer increased the compressive strength from 42 MPa to 60 MPa. Narimani Zamanabadi et al. [10] studied the influence of using NaOH concentration of 8M, 12M and 16M to synthesize slag geopolymer and revealed that 12M was optimized with the compressive strength of 47.6

MPa. For fly ash geopolymer, the increase of NaOH concentration (6M, 8M and 10M) improved the compressive strength (39.1 MPa) at 10M [11]. However, further increased the NaOH concentration after the optimum molarity could deteriorate the strength of geopolymers due to the severity of efflorescence increased [13].

Besides, other researchers [14-16] suggested that the solid to liquid (S/L) ratio could affect the mechanical strength. Arafa et al. [14] studied the various S/L ratio (1.7 to 2.7) on fly ash geopolymer and suggested that the increase of compressive strength (87MPa) was attributed to the density gained when increasing S/L ratio up to 2.5. Moreover, Ling et al. [15] focused on S/L ratio in the range of 1.67 to 3.03 and concluded that increasing this ratio tends to positively influence the compressive strength from 40MPa to 75MPa on fly ash geopolymer. Some of the researchers [16] also concentrated on the S/L ratio based on geopolymer mortar and concrete. They stated that increasing S/L ratio up to 2.22 contributed to the compressive strength of geopolymer mortar with 25.83MPa whereas the optimum strength (11.5MPa) of geopolymer concrete obtained at 3.03 of S/L ratio. However, further increase the S/L ratio tend to increase setting time [17] and viscosity [14] which degrade the strength of geopolymer.

For the alkaline activator, the combination of alkali hydroxide and soluble silicate positively influenced the organization of the geopolymeric structure and contributed to the strength gain as compared to the solely alkaline hydroxide used [18, 19]. Alkaline hydroxide is used to dissolve the aluminosilicate sources while soluble silicate played the role of binder [20]. The most commonly used alkaline solutions are mixing of hydroxide (sodium or potassium) and silicate (sodium or potassium) such as NaOH mixed with Na_2SiO_3 [9]. Zamanabadi et al. [10] used 1.0, 2.5, and 4.0 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio to activate the slag geopolymer and suggested that 2.5 provided the optimum compressive strength with 47.6MPa as soluble Si from Na_2SiO_3 altered the reaction of kinetics and indirectly improved the condensation process [21]. Saloma et al. [22] studied the ranges from 2.50 to 3.25 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio based on fly ash geopolymer and showed an increment compressive strength from 50MPa to 70MPa up to 2.75 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio. However, high viscosity [23] and long setting time [24] caused strength dropped when higher $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio was used.

Meanwhile, plenty of researches have been utilized two or more precursors to synthesize aluminosilicate geopolymer such as fly ash/slag-based [25], fly ash/metakaolin-based [26], fly ash/kaolin-based [27] and slag/metakaolin-based [28]. The performance of these geopolymers was usually better than the sole precursor used. For instance, some researchers [29] investigated that incorporating ground-granulated blast furnace slag into fly ash geopolymer could enhance the mechanical performance and microstructure of geopolymer. Niklić et al. [25] utilized electric arc furnace slag to replace with fly ash geopolymer and it was beneficial to the sample as strength improved.

However, the use of ladle furnace slag to substitute fly ash geopolymer is still lack. Wang et al. [30] utilized ladle furnace slag to synthesize geopolymer and successfully obtained a high strength. Hence, ladle furnace slag is believed to have potential use to incorporate into fly ash geopolymer. In this paper, the objective is to investigate the mixing parameters (NaOH concentration, S/L ratio, $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio and ladle furnace slag replacement) towards fly ash geopolymer by physical and mechanical testing included bulk density, apparent porosity, water absorption and compressive strength.

Materials and Methods

The aluminosilicate materials used were fly ash and ladle furnace slag. They were supplied in the form of grey powder. The chemical composition of aluminosilicate materials was acquired from X-ray fluorescence (XRF) analysis. The main compounds of fly ash were SiO_2 and Al_2O_3 . The CaO content of fly ash was 3.89% which is less than 20%, and summation of the SiO_2 , Al_2O_3 and Fe_2O_3 content were more than 70% which is 91.16%, and hence, these are evident to classify

that fly ash as Class F according to the description of ASTM C618 [31]. Besides, the main compound of slag was CaO which indicated the grey colour of slag. Slag further contained 21.3% of SiO₂ and 2.3% of Al₂O₃.

Table 1. Chemical composition of fly ash and slag, as determined using XRF analysis

Compound	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	TiO ₂	K ₂ O	MgO	Other
Fly ash (wt.%)	56.3	28.00	3.89	6.86	2.17	1.49	-	1.29
Slag (wt.%)	21.3	2.30	63.59	8.08	0.5	-	2.6	1.63

The alkaline solution used was the combination of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃). NaOH was in the form of white caustic soda pellets with 99% purity and supplied by Sigma-Alrich, Germany with the brand name of HmbG®. The colourless of Na₂SiO₃ solution was collected from South Pacific Chemicals Industries Sdn. Bhd. (SPCI), Malaysia with the chemical compositions of SiO₂ (30.1%), Na₂O (9.4%) and H₂O (60.5%). The specific gravity and viscosity of Na₂SiO₃ at 20°C were 1.4g/cm³ and 0.4 Pa.s, respectively.

Geopolymers were sorted by two types of geopolymers which are fly ash geopolymer and fly ash/slag geopolymers. Fly ash geopolymer was prepared by mixing of full fly ash with alkali activator whereby fly ash/slag geopolymer was mixed by fly ash and slag with the proportion of 90:10, 80:20, 70:30 and 60:40 with alkali activator. NaOH pellets were mixed with distilled water and put into a volumetric flask under room temperature in order to cool down. After combined the NaOH and Na₂SiO₃, the samples were kept for 24 hours to achieve equilibrium under room temperature. This solution further mixed with fly ash and slag powder and stirred well for 15 minutes by using a mechanical mixer. Fly ash geopolymer was formulated with NaOH concentration (6M, 8M, 10M, 12M and 14M), S/L ratio (1.0, 1.5, 2.0, 2.5, 3.0 and 3.5), Na₂SiO₃/NaOH ratio (1.0, 1.5, 2.0, 2.5 and 3.0) and slag replacement (10%, 20%, 30% and 40%).

After mixing, the geopolymer pastes were then rapidly poured into cube (50 × 50 × 50mm) plastic moulds and hence, compacted and tamped the paste based on the description of ASTM C109 [32]. Afterward, the samples were pre-curing at room temperature (25°C) for one day and further went through the curing process at 60°C for 24 hours in an oven which was manufactured by Young Chenn Instruments model types COH-545. The exposed portion of the samples was covered by plastic in order to prevent the moisture escape during the curing stage. After the curing process, all samples were removed from oven and further kept at room temperature for 28 days before undergoing testing.

To evaluate the bulk densities, measured the mass and dimensions of the samples based on BS EN 12390-7 [33] as shown in equation 1. To clarify the water absorption, measured the mass (wet and dry) of the specimens whereby further measured the suspended mass for apparent porosity as shown in Equation 2 and Equation 3 according to ASTM C642 [34]. The test of compressive strength was to determine the highest resistance of a sample who can resist the axial load by using a Mechanical Tester with a constant rate of 5mm/min. Three specimens were tested for each ratio.

$$\text{Bulk density (kg/m}^3\text{)} = \frac{\text{Mass}}{\text{Volume}} \quad (1)$$

$$\text{Water absorption (\%)} = \frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}} \cdot 100 \quad (2)$$

$$\text{Apparent porosity (\%)} = \frac{\text{Wet weight} - \text{Dry weight}}{\text{Wet weight} - \text{Suspended weight}} \cdot 100 \quad (3)$$

Results and Discussion

Bulk Density

Fig. 1 showed the bulk density of fly ash geopolymers with various parameters. In general, the bulk densities of fly ash geopolymer and fly ash/slag geopolymer optimized at a certain point (8M of NaOH concentration, 3.0 of S/L ratio, 1.5 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio and 20% of slag replacement) and then reduced. The bulk densities of fly ash geopolymer (Fig. 1a-c) were in the range between 1568.33kg/m^3 and 2050kg/m^3 . This observation was consistent with the density result of around 2000kg/m^3 which was reviewed by Muthu Kumar & Ramamurthy [35]. However, the overall bulk densities of fly ash/slag geopolymer (2055kg/m^3 to 2100kg/m^3) were comparatively higher than those fly ash geopolymer alone as shown in Fig. 1d. This is because the slag source material consisted of 1238kg/m^3 density which was heavier than fly ash source materials (1168kg/m^3). It was in alignment with Pilehvar et al. [36].

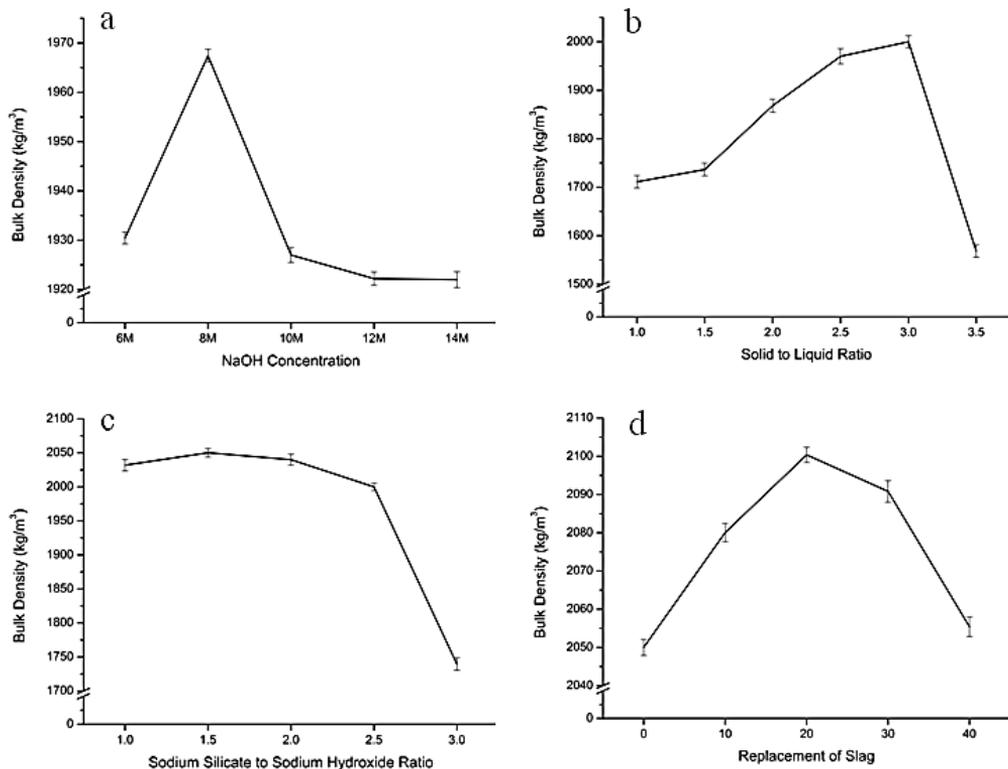


Fig. 1. Bulk density of fly ash geopolymer with a) NaOH concentration, b) S/L ratios, c) $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios and d) slag replacement.

As a comparison, similar trend was conducted by Patankar et al. [37] when increasing NaOH concentration in fly ash geopolymer mortar. The densities were in the range between 2157.65kg/m^3 to 2215.67kg/m^3 and these denser ranges were due to the addition of filler sand. Next, Rahim et al. [17] investigated the S/L ratio from 3 to 5 on fly ash geopolymer and cured for one day. They obtained the bulk densities ranges from 2900kg/m^3 to 3600kg/m^3 whereby similar trends observed. Various densities range achieved can be explained by the data collection at varying aging days. Besides, Kamseu et al. [18] concluded that increase Na_2SiO_3 reduced the bulk density of rice husk ash/metakaolin geopolymer composite from 1700kg/m^3 to 1420kg/m^3 . Various trends obtained might be due to different aluminosilicate source used. Furthermore, Guo

& Yang [38] studied the fly ash/slag geopolymer composite with the addition of fiber. They revealed that by increasing slag content from 7% to 28% tended to increase the apparent density (1650kg/m^3 to 1850kg/m^3) continuously. The trend was different due to the presence of fiber in their study.

Apparent Porosity and Water Absorption

Fig. 2 displayed the apparent porosity and water absorption with various parameters on fly ash geopolymer. Overall, the results of water absorption follow the trend of apparent porosity results due to the higher porosity content tended to absorbed more water in the samples. The overall apparent porosity and water absorption of fly ash geopolymer were between 19% to 40% and 9% to 22%, respectively.

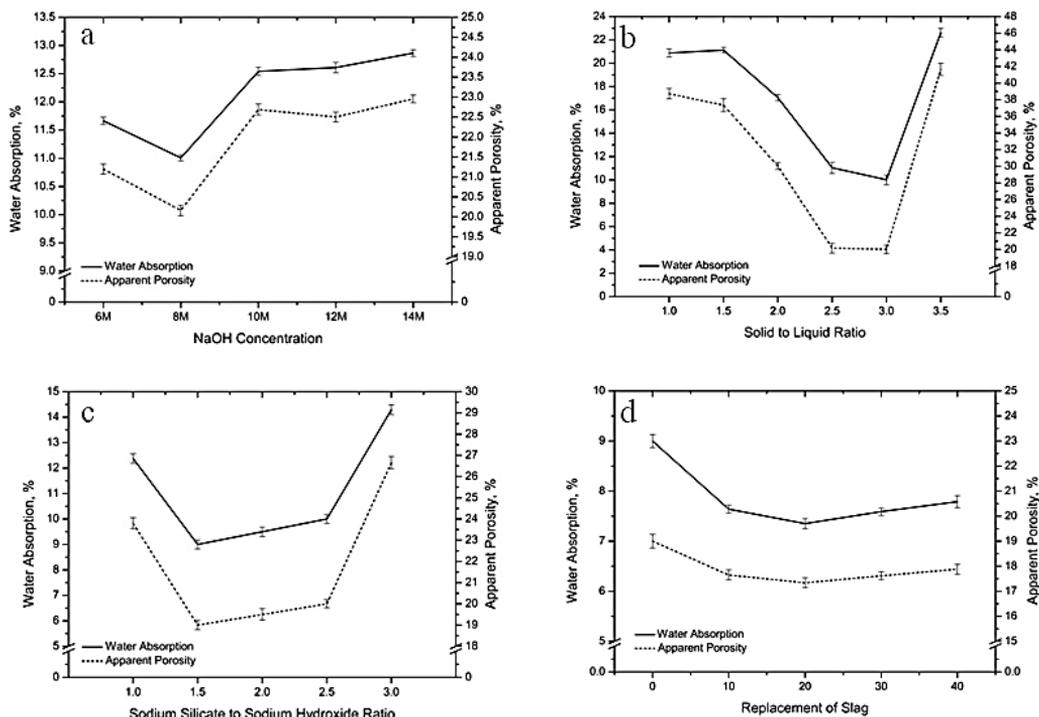


Fig. 2. Apparent porosity and water absorption of fly ash geopolymer with a) NaOH concentration, b) S/L ratios, c) $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios and d) slag replacement.

Based on Fig. 2a, the lowest apparent porosity (20.16%) and water absorption (11.00%) were located at 8M of NaOH concentration. As a comparison, Aliabdo et al. [39] investigated the influence of NaOH concentration between 12M to 18M based on fly ash geopolymer concrete. They obtained the lowest porosity content with 11.15% and water absorption consisted of 5.30% at 16M of NaOH. Their optimized NaOH concentration was different from this study (8M) was due to the addition of aggregates and sand.

From Fig. 2b, 3.0 of S/L ratio obtained the lowest values of apparent porosity and water absorption. As compare with Krishnendu et al. [40], they fixed the L/S ratio at 0.32 (S/L ratio around 3) on fly ash geopolymer with alkaline activator (KOH and Na_2SiO_3). They revealed that the apparent porosity consisted of 10.5% whereas water absorption contained 7.06% which is lower than the values obtained in this work. The reason behind suggested that the type of alkali hydroxide was different in used with this study as they were used potassium hydroxide while this

research was using sodium hydroxide. It is well known that different alkali solution used could contribute to different properties such as porosity of geopolymers.

Furthermore, Fig. 2c observed that the apparent porosity and water absorption of fly ash geopolymer reduced from $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 1.0 to 1.5, but increased linearly until 3.0. Higher amount of silicate could provide a higher amount of aluminosilicate gel formed which indirectly constituted the better bonding within inter-particle [41]. Thus, it could be suggested that the silicate occupied the cavities between the fly ash particles, as a result, acquired lower water absorption at $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 1.5. However, water absorption kept increasing when further increased the silicate content (3.0 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio) in fly ash geopolymer indicated that higher void content observed. As a comparison, different observations on fly ash geopolymer concrete where water absorption (5.40% to 4.85%) and porosity (11.30% to 10.02%) continuously reduced from 2.0 to 3.33 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio [39].

Generally, the apparent porosity (~17%) and water absorption (~7%) of fly ash/slag geopolymers (Fig. 2d) were comparatively lower than fly ash geopolymers. The apparent porosity and water absorption reduced from 10% of slag content to 20%, but beyond that increased to 40%. This observation was similar to Niklić et al. [25] where they stated that increasing the amount of electric arc furnace slag tended to decrease the porosity until a limit but increased again.

Compressive Strength

Fig. 3 plotted the compressive strength of fly ash geopolymers with various parameters. The optimal compressive strength of 38.89MPa was achieved at 8M of NaOH concentration, 3.0 of S/L ratio and 1.5 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio for fly ash geopolymer whereas fly ash/slag geopolymer contained the optimum strength (40.46MPa) at 20% of slag replacement. Generally, compressive strength followed the trend of bulk density where higher bulk density contributed to the strength gained. Bulk density was closely correlated to compressive strength [42]. Additionally, low strength observed was related to the high apparent porosity and water absorption (Fig. 2).

Accordingly, lower strength observed at each of the initial mixing parameters (6M of NaOH concentration, 1.0 of S/L ratio and 1.0 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio). There was insufficient of Na^+ ions for the dissolution of fly ash when low NaOH concentration was used. It was in accordance with previous study [43]. When utilized low S/L ratio indicated that the liquid content was higher than solid content which could cause slow dissolution process. This statement was in alignment with Aliabdo et al. [39]. Low $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio indicated that Na_2SiO_3 is lesser compared to the NaOH content. It meant that the greater amount of NaOH content caused the greater amount of water escaped from the mixture as the unbound water from the geopolymers samples were easily expelled out from NaOH rather than from Na_2SiO_3 . Similar observation was found by Ahuja & Dong [44].

However, optimum strength obtained at 8M of NaOH concentration was due to its better reaction between solid particles and aqueous phase in the final geopolymeric structure. This was consistent with previous research [39]. The highest strength fell at 3.0 of S/L ratio can be explained by the high amount of solid improved the interaction between alkaline solution and the reaction products, as a result, lower porosity content observed (Fig. 2b). S/L ratio could affect the volume of porosity in the paste and indirectly affected the mechanical performance of geopolymer [39]. High $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio leading the reacting materials with more soluble Si and greater mechanical strength was obtained at 1.5 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio. Similar conclusion was confirmed by Glid et al. [21].

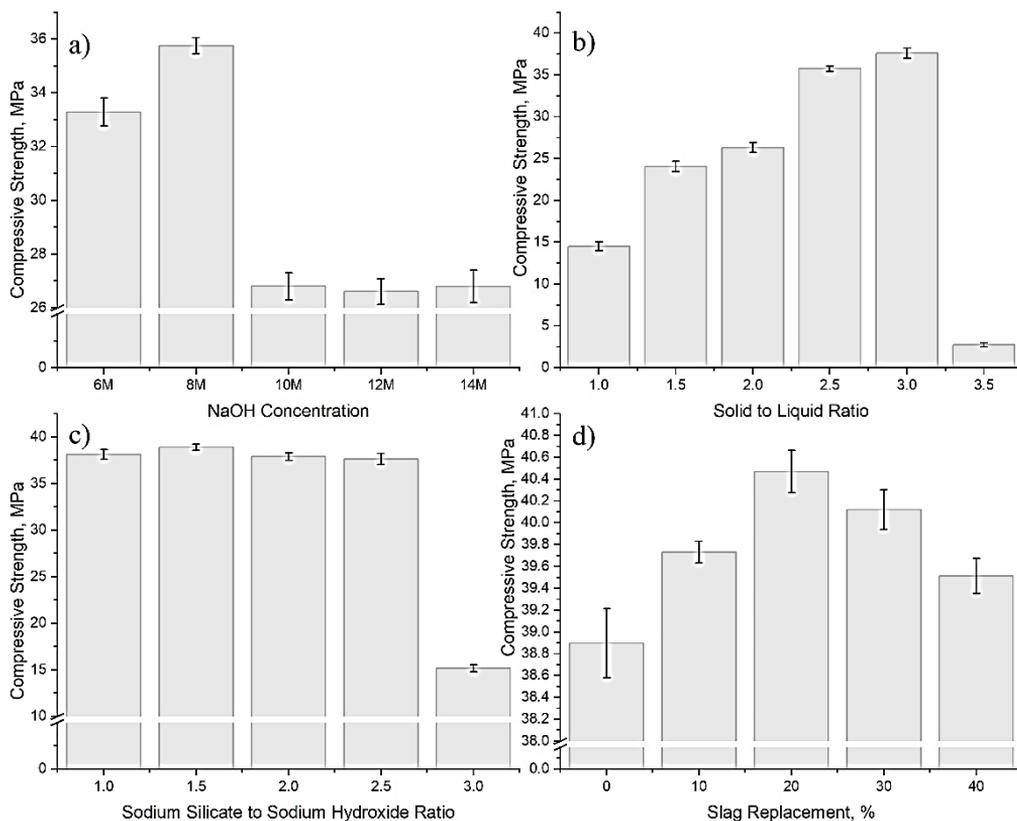


Fig. 3. Compressive strength of fly ash geopolymer with a) NaOH concentration, b) S/L ratios, c) $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios and d) slag replacement.

Besides, reduction in strength observed when beyond each of the optimal ratios. The excess of Na^+ ions concentration deteriorated the structure of geopolymer when high NaOH concentration was used which was correlated with previous researchers [39]. The ions could further react with atmospheric CO_2 to create sodium carbonate (Na_2CO_3) [45] and hence, this was the reason behind of strength dropped. In the case of high S/L ratio, the limited amount of solution was not enough to interact with the excess fly ash during geopolymerization process and thus, caused extremely high viscous of the mixtures. This high viscous caused the mixture difficulty in compaction during the moulding process, as a result, low extent of binder formation caused low strength [46]. When increasing $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio indicated that Na_2SiO_3 content was higher than NaOH content. The excess Na_2SiO_3 hindered the water evaporation meanwhile the viscous nature of Na_2SiO_3 caused a highly sticky mixture formed which prevented the further formation of the geopolymer matrix. Similar statement was agreed by Škvára et al. [47].

As a comparison, Somna et al. [48] achieved an optimum compressive strength of 25.5MPa at 14M of NaOH molarity which was higher than the optimized NaOH molarity (8M) in this work. The various observation was due to the used of fly ash with different chemical composition. They carried out the weight percent of SiO_2 (31.2wt%) and Al_2O_3 (18.9wt%) which were comparatively lower than this work based on XRF analysis (Table 1). The mechanical and physical properties of geopolymers are very depended on the amorphous Si/Al ratios. On the other hand, the compressive strength results revealed that the optimal S/L ratio and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio to synthesis fly ash geopolymer was 3.0 and 1.5, respectively. It was employed in accordance with previous study where Cai et al. [49] used S/L ratio of 3.0 to produce

fly ash geopolymer whereby Pavithra et al. [50] optimized the 1.5 of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio in fly ash geopolymer.

In the case of metakaolin geopolymers, Kong et al. [51] investigated that the S/L ratio of 0.8 achieved an optimum compressive strength (38.5MPa) whereas $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 0.24 was used in the study of Wang et al. [52]. As a comparison, fly ash geopolymers obtained optimum S/L ratio (3.0) and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio (1.5) in this study were comparatively higher than metakaolin geopolymers. The chosen of S/L ratio and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio were strongly dependent on the workability of the mixtures. This was due to the metakaolin source materials were layered structure which required high water demand (low S/L ratio) in order to provide higher strength [53] and hence, they required lower $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio to compensate the workability problem. In other words, higher S/L ratio and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio were applied in fly ash geopolymer synthesis was due to the spherical-shaped fly ash particles could enhance the workability in the mixture [46].

On the other side, the overall strength of fly ash geopolymer (2.73MPa to 38.89MPa) was slightly lower than fly ash/slag geopolymer (39.51MPa to 40.46MPa) as shown in Fig. 3d. It was in accordance with apparent porosity and water absorption values (Fig. 2) where higher porosity content on fly ash geopolymer tended to deteriorate the strength. Puligilla & Mondal [54] stated that the slower setting of the fly ash geopolymer mixture was caused by the low reactivity of fly ash which directly led to weaker compressive strength than fly ash/slag geopolymer. It is believed that the source of CaO content that came from slag in the initial solid mixture has been enhanced the strength of geopolymers. CaO introduced soluble Ca^{2+} ions in a geopolymer mixture that caused the formation of calcium silicate hydrate (C-S-H) gel along with sodium aluminosilicate hydrate (N-A-S-H) gel which contributed to the strength gained [55-57]. Consequently, it is believed that the optimum strength obtained at 20% content of slag on fly ash geopolymer was due to the formation of C-S-H gel in addition to N-A-S-H gel.

However, the specimen with slag content exceeded 20% exhibited strength dropped was due to the rapid geopolymerization reaction caused by an increase of slag content. It was also proven by the fact that increasing the amount of slag in the paste caused the rate of setting quicker [58]. Therefore, the fly ash/slag geopolymer with a rapid rate of setting caused not enough time for geopolymeric structure to well interact. It is reasonable to suggest that the rapid rate of geopolymerization caused poor compressive strength at fly ash geopolymer with 30% and 40% of slag content.

In this work, the optimum slag replacement to fly ash ratio was 20:80 which was in agreement with previous authors [25, 57, 59] reported that by using the slag content between 20% to 30% incorporate into fly ash geopolymer was beneficial to the final product. Niklić et al. [25] utilized electric arc furnace slag to replace fly ash geopolymer, 30% content of slag were used in further investigation where this ratio provided optimum strength (32MPa) and exhibited strength decreased when slag exceeded 30%. Similarly, Lee et al. [59] concluded that ground-granulated blast furnace slag with 20% content substituted to fly ash geopolymer obtained optimum strength (30.6MPa) but above 20% the strength dropped gradually. Therefore, the combination of slag and fly ash acted as an alkali-activated binder could enhance the strength of geopolymer where the slag could control the formation of main binding gels and reaction mechanism.

Conclusion

The overall bulk densities of fly ash geopolymer (1967.4kg/m³ to 2050kg/m³) were lower than fly ash/slag geopolymer (2055kg/m³ to 2100kg/m³). The apparent porosity (19% to 40%) and water absorption (9% to 22%) of fly ash geopolymers were comparatively higher than fly ash/slag geopolymers with apparent porosity (~17%) and water absorption (~7%). Hence, it can be concluded that the high bulk density and low apparent porosity and water absorption

contributed to the compressive strength of fly ash/slag geopolymer where fly ash/slag geopolymer obtained higher strength than fly ash geopolymer.

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