



Research Article

The influence of curing conditions on physicomechanical properties of alkali-activated slag/fly ash based mortars

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ABSTRACT

Geopolymer mixes is a promising solution for achieving environmentally friendly and sustainable concrete in the future. Due to its superior performance compared to traditional Portland cement mixes, it exerts a significant influence on the construction industry. Hence, this study has sought to understand the influence of various curing conditions on the physical and mechanical properties of alkali-activated slag/fly ash-based mortar mixtures activated with sodium hydroxide and sodium silicate. In this study, four distinct curing regimes were employed to investigate the impact of curing types on the properties of geopolymer mortars. These curing regimes included ambient curing at 22 ± 2 °C, moist curing at 95% RH, steam curing, and heat curing at 80°C for 24 hours. The experimental research explored the fundamental physico-mechanical properties of alkali activated mortar samples, including compressive and flexural strength, ultrasonic pulse velocity, volume of permeable pores, and water absorption. Based on the test results, the oven and steam cured samples exhibited superior mechanical and physical properties compared to ambient and moist cured samples. On the other hand, the compressive and flexural strength values of the ambient cured specimens were comparable with the oven and steam cured samples at later ages. The moist-cured samples performed inferior mechanical and physical properties contrary to conventional cement-based materials due to the discharge of the alkaline solution on the sample surface with elapsed curing age which restricts their use in moist environments. Statistically significant relationship was established between compressive and flexural strength.

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INTRODUCTION

Portland cement (PC) is one of the most predominant man-made building materials globally. By 2050, it is predicted that the world population will increase to 9.8 billion, constituting 68% of the population living in cities. It is considered that this situation will bring along the demand for housing and industrial facilities and will lead to a further increase in cement production, which is 4.2 million tons

in 2020 [1]. Concrete is mainly composed of water, PC, aggregates (fine and coarse), and admixtures (chemical or mineral) and as odd as this may seem, it is the second most utilized material worldwide, after water [2]. Annually, approximately 14 million cubic meters of concrete are manufactured, and with the sustained economic expansion and new infrastructure investment pledges of emerging countries, this figure is anticipated to increase[1].

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PC is one of the most expensive components of concrete, constituting about 10% of the conventional concrete mass. Besides, during its production, the consumption of natural resources and raw materials has a crucial environmental and economic impact. As such, many severe ecological problems arise due to raw material extraction which damages the landscape, and the availability of these raw materials reduce all over the world [3]. PC production process mainly includes collecting, smashing, and milling of the clay and calcareous materials, heating the raw materials in a rotary kiln, cooling the resulting output (i.e., clinker) obtained, adding gypsum to the clinker, and bundling the resulting cement. Hence, the manufacturing of PC involves a significant amount of natural resources, which are subjected to an energy-intensive process and emit a substantial amount of carbon dioxide. The production of PC is also detrimental to the environment, as the production of one ton of cement releases approximately one ton of carbon dioxide into the atmosphere [4–6]. Greenhouse gas emissions such as CO₂ reduce not only the quality of the air but also have negative impacts on human health, leading to global warming, ozone depletion, acid rain, biodiversity loss, and reduced crop productivity [7].

Currently, many studies are being carried out to find more sustainable alternatives that can be used in replacement of PC, one of which is using supplementary cementitious materials (SCM) [8] derived from several industrial and agricultural processes. Alkali-activated materials (AAMs) might be regarded as a new kind of environmentally friendly materials produced through the reaction of an alkali source as the activator and alumina-silicate powders as the binder. AAMs have gained much interest not only because of their superior mechanical and durability performance, such as resistance to chemical attacks, fire, and corrosion but also due to their significant contribution to sustainable construction [9]. AAMs evidently have less CO₂ emission and consume less energy compared to PC based materials. Therefore, AAMs manufactured using industrial byproducts such as ground granulated blast furnace slag (GGBS) and fly ash (FA) are considered more eco-friendly binding materials [10]. Alkali activators such as alkali hydroxides and alkali silicates have been preferred predominantly in previous researches to activate these binding materials.

The utilization of AAMs in precast applications comes into prominence due to factors such as the alkali activator solution preparation process, and especially the low-strength development of binders such as FA under ambient conditions [11]. On the other hand, it has been shown that the use of precursors rich in calcium content, such as GGBS, enables strength development under ambient conditions as well [12,13].

The curing conditions such as temperature and relative humidity (RH) are extremely significant factors for synthesizing alkali-activated binders, which affect the ultimate strength and durability of AAMs [8,14]. Oven curing plays a

significant role in the activation of Class F FA which promotes strength development; therefore, some researchers have investigated the characteristics of alkali-activated binders under oven curing. Previous studies [15,16] have shown that FA-based geopolymers need to be cured at a relatively high temperature to exhibit desirable mechanical properties due to the low reactivity of FA at ambient temperature resulting from insufficient calcium content in its chemical composition. The increase in curing temperature of alkali-activated binders can also promote the hydration reactions, which enhances the microstructure, reduce micro-cracks [17], and consequently increase the mechanical strength [18]. Bakhariev [19], reported that the early-age compressive strength of sodium silicate activated Class F FA-based geopolymers maintained at 95 °C was greater compared to that of geopolymer mortars kept at 75 °C. However, it was reported that the ultimate compressive strength of both mixes had similar results when they are cured at room temperature after heat treatment. In addition, steam curing is also an alternative curing method preferred especially in precast applications to enhance the early age strength of cement-based materials and AAMs which have low calcium content [20]. Hou et al. [21], stated that the compressive strength of alkali-activated FA geopolymer mix exposed to steam curing at 80 °C exceeded the mechanical strength of those heat-cured at 50 °C and 65 °C. Nevertheless, it is also reported that once the temperature of curing process surpasses 120 °C, the strength increase ratio of AAMs containing FA is immensely low compared to that of others. Besides, the duration of heat or steam curing is one of the remarkable parameters that affects the physical, mechanical, and durability properties of AAMs. Jamdade [22], studied the effect of heat curing duration on AAMs' properties and revealed that the compressive strength of FA-based AAM samples is the greatest once curing at 90 °C for a duration of 18 to 24 h. It has also been shown that curing for two days at a temperature around 50 °C yields comparable strength with curing for 28 days at room temperature [23]. Other studies indicated that the ideal temperature and duration of curing of AAMs containing FA and GGBS is 80 °C and between 12 and 24 h, respectively [24,25].

The available literature review showed that generally the influence of oven curing temperature and duration on the properties of FA- and GGBS-based AAMs were previously investigated. However, a comparative study considering the influence of various curing conditions on the behaviour of AAMs containing FA and GGBS is limited [26–28] and requires further research. The main objective of this study was therefore to investigate the effect of various curing conditions on the mechanical and physical properties of FA and GGBS based AAMs. Four different curing conditions were employed: oven curing (OC), moist curing (MC), steam curing (SC), and ambient curing (AC), and their effect on the physical and mechanical properties of the mortar samples were researched. Each sample was left to cure at a specific condition and was

subjected to different temperatures and relative humidity. The physical and mechanical behavior of the mixes were examined by determining ultrasonic pulse velocity, compressive strength, and flexural strength. Furthermore, other properties such as water absorption, oven-dry density, and voids content were also investigated. The ideal curing condition for FA and GG-BS-based alkali-activated mortars were determined based on the physical and mechanical test results, and the required analyses of the results are given in this paper.

MATERIALS AND METHODOLOGY

Materials

The locally supplied precursor materials used to manufacture AAMs were FA and GGBS. The specific gravity of FA and GGBS were determined as 2.21 and 2.91, respectively. Figure 1 shows the physical appearances of the raw binding materials used in the research. The oxide compositions of GGBS and FA were obtained by X-ray fluorescence (XRF) analysis and listed in Table 1. The major chemical components of GGBS are CaO, SiO₂, and Al₂O₃, whereas SiO₂ and Al₂O₃ were mainly detected in FA. Tap water, NaOH, and sodium silicate (SS) were used to prepare the alkaline solution. The physical and chemical properties of the alkaline solutions are tabulated in Table 2. Natural siliceous sand with a specific gravity of 2.67 and a particle size distribution that ranges between 0.08 and 2.00 mm was used as fine aggregate in all mixes for the preparation of mortar samples.

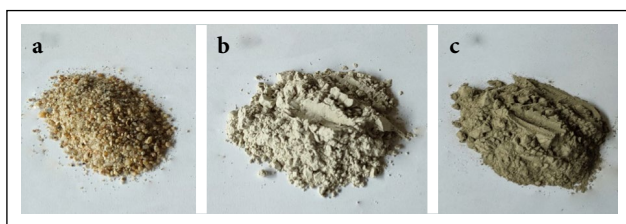


Figure 1. The appearance of: (a) sand, (b) GGBS, and (c) FA.

Table 1. The oxide components of FA and GGBS

Oxide composition (%) by mass	FA	GGBS
SiO ₂	52.6	40.6
Fe ₂ O ₃	5.8	1.2
Al ₂ O ₃	25.0	12.6
CaO	3.3	35.1
MgO	2.1	5.8
SO ₃	1.0	0.1
Na ₂ O	0.3	0.8
K ₂ O	4.1	0.7
Density (g/cm ³)	2.21	2.91

Mix Proportions, Sample Preparation, and Curing

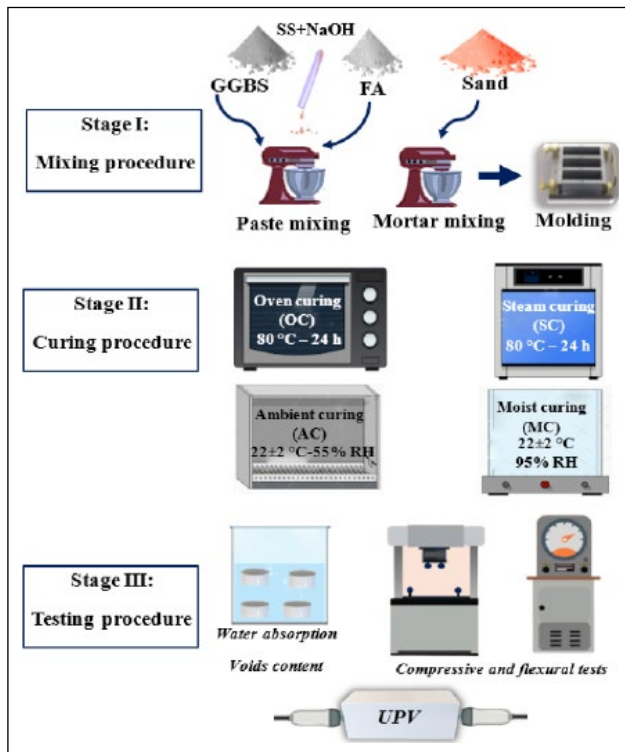
The mixture proportions of alkali-activated mortar specimens are illustrated in Table 3. The design parameters of the alkali solution such as SS/NaOH ratio and NaOH molarity were taken as 1.5 and 8.0 M, respectively. Moreover, the water-to-binder ratio and FA to GGBS ratio were determined according to preliminary mixes so as to ensure sufficient workability properties and determined as 0.5 and 1.5, respectively for all mixes. The alkali activator used to manufacture mortar specimens was a combination of SS and NaOH. The alkaline solution was obtained after dissolving NaOH pellets in the liquid SS solution for about 5 min and left to cool down to room temperature of 22±2 °C, before use. The mixing was performed using a laboratory-type mixer. The prepared alkaline solution and blended FA and GGBS were initially added to the mixer bowl. Subsequently, natural sand was introduced progressively after 30 s of blending, and the stirring process was resumed for about 5 min until a uniform fresh sample was obtained. The fresh samples were then poured into 40×40×160 mm³ steel molds and vibrated for about 5-10 s to release entrapped air voids. The molds were then covered with the sealing sheets to avoid from the evaporation of water and kept for 24 h in the laboratory conditions. The hardened alkali-activated mortar prisms were demolded after 1 day of casting and exposed to four different types of curing conditions: (i) AC, (ii) OC, (iii) SC, and (iv) MC. For AC, the mortar samples were kept in a chamber at a constant RH of 55±2% and temperature of 22±2 °C and for MC, the samples were stored at a constant RH of 95±2% and temperature of 22±2 °C, until the time of testing. The rest of the specimens were sealed with a plastic film and then placed in an oven or steam chamber at 80 °C for 24 h to provide OC and SC conditions, and then kept at ambient conditions until testing age. The flow chart of the experiments is depicted in Figure 2.

Table 2. Technical properties of sodium hydroxide and sodium silicate

Properties	Sodium silicate	Sodium hydroxide
Molecular formula	Na ₂ SiO ₃	NaOH
Molecular weight (g/mol)	122.06	40.00
Physical appearance	Liquid	Pellet (Granular)
pH	11.0~11.5	>14.00
Density (g/cm ³)	1.35	2.13
H ₂ O (%)	63.8	-
Na ₂ O (%)	8.4	-
SiO ₂ (%)	27.8	-

Table 3. The mixture proportion of mortar mixtures

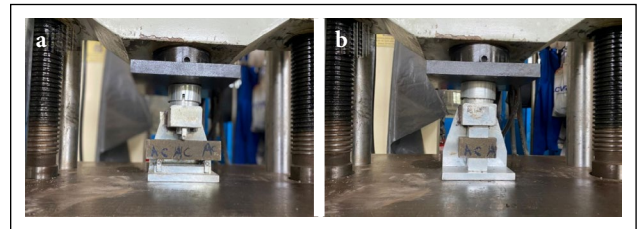
FA (wt% of binder)	GGBS (wt% of binder)	NaOH molarity	SS/NaOH ratio	Sand to binder ratio vol, %
60	40	8.00	1.50	50

**Figure 2.** The schematic illustration of the present study.

TESTING PROCEDURE

Physical Properties

The physical investigation of the alkali activated mortars such as water absorption and volume of permeable pores were conducted at 28-days on two replicate specimens with sizes of $4 \times 4 \times 16 \text{ cm}^3$ by following the practices depicted in ASTM C642. The mortar samples were initially dried in an oven at $105 \text{ }^\circ\text{C}$, and the mass of each sample was recorded (W_a). The specimens were then immersed in water for 48 h and the saturated and surface dried (SSD) masses (W_b) were determined after the removal of samples from water. In order to determine the volume of permeable pores, the mortars were stored in the water at $100 \text{ }^\circ\text{C}$ for about 5 h and permitted to cool down in water for about 14-15 h. Subsequently, the SSD mass of the samples was determined in air (W_c) and in water (W_d). Water absorption (W_{abs}) and volume of permeable pores (V_{pp}) were calculated using Eq. (1) and Eq. (2), respectively. The ultrasonic pulse velocity (UPV) of the samples was determined on three $40 \times 40 \times 160 \text{ mm}^3$ prismatic samples at 28 days in accordance with ASTM C597. The UPV was calculated by dividing the distance between the transducers (160 mm) by the transit time.

**Figure 3.** Mechanical properties of mortars: (a) flexural strength; (b) compressive strength

$$W_{abs} = \frac{W_b - W_a}{W_a} \quad (1)$$

$$V_{pp} = \frac{W_c - W_a}{W_c - W_d} \quad (2)$$

Mechanical Properties

Figure 3 shows the mechanical tests performed on the samples. The flexural strength of the AAM was determined using $40 \times 40 \times 160 \text{ mm}^3$ prismatic specimens following ASTM C348 standard. The samples were tested at a loading rate of $40 \pm 5 \text{ N/s}$ until failure and the flexural strength was determined using Eq. (3). The broken specimens obtained from the flexural strength test was used to determine compressive strength of the mortar samples in accordance with ASTM C349 by using Eq. (4). The samples were tested at a loading rate of $1200 \pm 50 \text{ N/s}$ until failure. The mechanical tests were performed at 3, 7, and 28 days on three replicates and the average values were reported.

$$S_f = 0.0028P \quad (3)$$

where, S_f and P represent the flexural strength (MPa) and failure load (N), respectively.

$$S_c = 0.00062F \quad (4)$$

where, S_c and F represent the compressive strength (MPa) and failure load (N), respectively.

RESULTS AND DISCUSSION

Physical Tests

UPV is a non-destructive test which can be employed to evaluate the stiffness of mortars or concretes, which is primarily influenced by the presence of percolated solids, open voids, and defects in the matrices of materials [29]. The UPV test results of the mortar samples are presented in Figure 4. The results showed that the UPV of the samples varied between 2957 and 3235 m/s. The highest UPV value was obtained in AC specimens, followed by OC, SC, and MC. Furthermore, it has been observed that

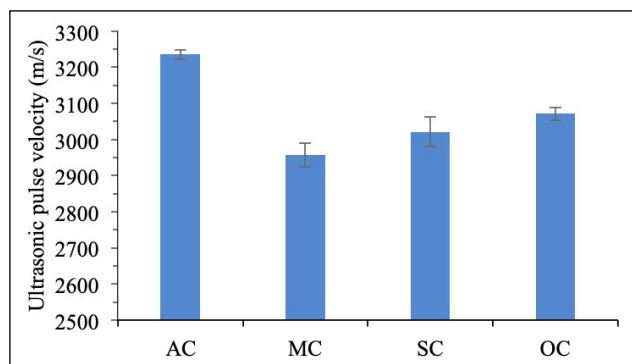


Figure 4. Ultrasonic pulse velocity test results.

under the effect of moist environment (i.e. MC and SC) the UPV had the lowest values, indicating that high humid environment modify the pore structure and possibly increase the porosity. This has been especially observed on the MC samples with the appearance of discharged crystals on the samples' surface which increased with time as shown in Figure 5. In order to find the type of discharged crystals, various alkaline solutions such as NaOH solution, SS solution, and NaOH+SS solution were prepared as shown in Figure 6. The prepared alkaline solutions were stored in moist conditions (95% RH, 22±2 °C) to observe which chemical compound was responsible for the crystal formation. After 3 days, the NaOH solution exhibited the formation of crystal-like structures under the influence of moisture, whereas no such formation was detected in the SS solution. Furthermore, it was noted that the aforemen-

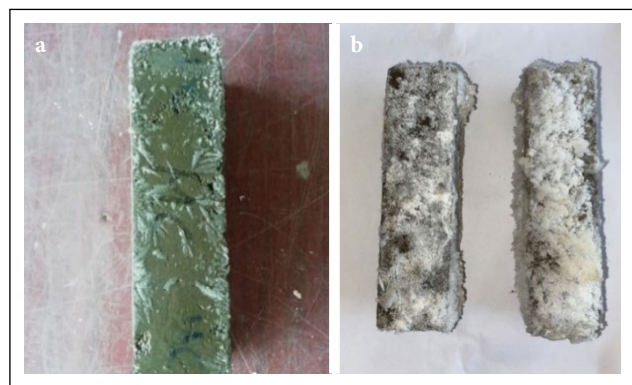


Figure 5. The formation of crystals on MC samples: (a) at 7-days and (b) at 28-days

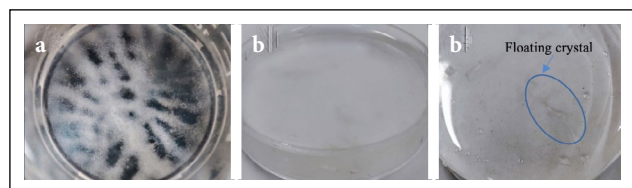


Figure 6. The alkaline solutions exposed to moist curing conditions: (a) NaOH solution, (b) SS solution, and (c) NaOH+SS solution.

tioned crystals were generated in the SS+NaOH solution, albeit to a lesser extent when compared to the solution containing solely NaOH. Therefore, it is suitable to assume that the crystals formed under the influence of moisture are NaOH crystals.

The water absorption and the permeable pore ratio of the mortar samples cured at different conditions are presented in Figure 7. The water absorption of the mortar mixes varied between a narrow range of 6.06% and 6.69%, and the OC samples achieved the lowest value. On the other hand, the permeable pore volume of the AC samples was notably higher than that of the others and was determined as 16.0%. The rest of the samples had similar permeable pore volume ranging between 13.1 and 13.7%. This can be attributed to the lower strength development of fly ash abundant geopolymer mixes under ambient conditions [30,31]. The lower strength development leads to the formation of connected pore structure and increases the volume of permeable pores in the sample. On the other hand, the lower volume of permeable pore values observed in the samples kept in MC conditions despite the low strength development and ambient temperature curing may be attributed to the formation of crystals that block the continuous voids, thereby influencing the outcomes.

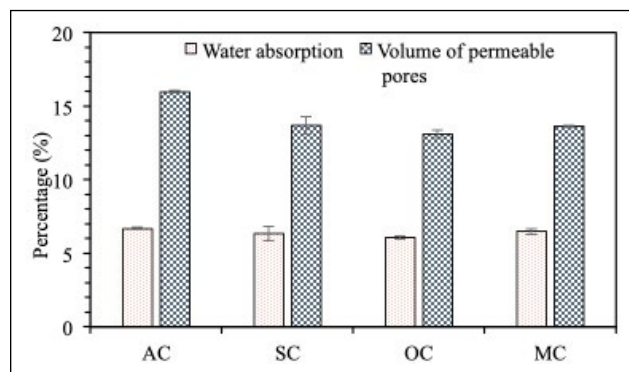


Figure 7. The water absorption and voids content of the mixes.

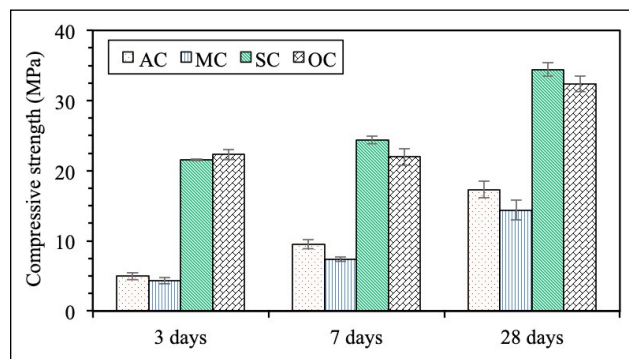


Figure 8. Compressive strength development of the mortar mixes.

Compressive and Flexural Strength

The compressive and flexural strength development of mortar specimens subjected to four different curing conditions are depicted in Figures 8 and 9. The test results indicated that both compressive strength and flexural strength increased with curing age. Figure 8 shows that the compressive strength of OC and SC samples were comparable and notably higher than that of AC and MC samples at all tested ages. At the age of 3 days, the compressive strength of OC and SC specimens were about 5 times greater than that of AC and MC samples. However, at later ages the strength values were found to be closer and the strength of OC and SC specimens were about 2.5 and 2.0 times greater than AC and MC samples, respectively. The greatest compressive strength was attained in SC samples as 34.4 MPa, followed by OC, AC, and MC as 32.4 MPa, 17.3 MPa, and 14.4 MPa, respectively, at 28 days. Moreover, the compressive strength of OC and SC samples increased by about 45% and 60% from 3 days to 28 days, respectively, whereas the strength increase rate of MC and AC specimens was 230% and 250% from 3 to 28 days. The improved early age strength during high temperature curing (80 °C) can be ascribed to an increased rate of SiO_2 and Al_2O_3 dissolution from precursor particles, which accelerated the synthesis of reaction products such as calcium aluminate silicate hydrate (C-A-S-H) and sodium aluminate silicate hydrate (N-A-S-H), which alleviate the strength of alkali-activated materials [32]. Once the AAM specimens were subsequently cured under ambient conditions, inadequate SiO_2 and Al_2O_3 were released due to the earlier consumption of the alkaline solutions, leading to slower strength development.

Figure 9 shows the flexural strength development of the mixes. The OC samples achieved the greatest flexural strength at all ages followed by SC, AC, and MC samples. The highest flexural strength was achieved by OC samples as 9.6 MPa at 28 days, whereas MC had the lowest flexural strength of 4.1 MPa.

The higher mechanical strength achieved in OC and SC mixes might be due to the higher curing temperatures resulting in the synthesis of higher reaction products; hence, with the appropriate curing temperature, more metal ions

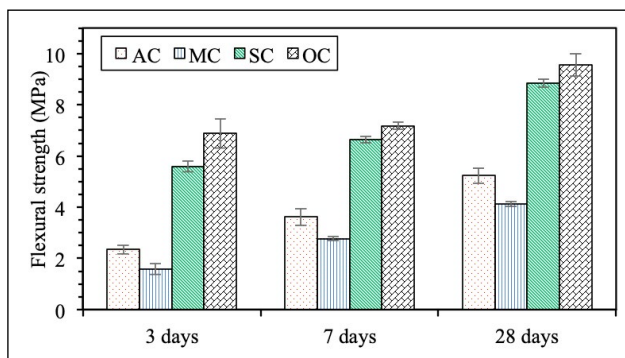


Figure 9. Flexural strength development of the mortar mixes.

can be integrated into the matrix, reducing the metal ion concentration [3]. Noushini et al. [33], examined alkali metal leaching by monitoring the pH of hardened geopolymer pastes and found that the high temperature cured geopolymers had a lower pH value than geopolymers cured at ambient temperature. Excessive alkalinity in ambient temperature curing indicates that the samples have a lower degree of polymerization and, therefore, less reaction product formation and lower mechanical strength.

The compressive strength and UPV of the samples did not follow a consistent trend. Even though outperformed mechanical properties of SC and OC compared to AC samples their UPV values were lower than AC samples. This can be attributed to the formation of larger pores or micro-cracks in SC and OC samples due to the higher initial curing temperature [34]. Similarly, Balcikanli et al. [35], found that the correlation between compressive strength and UPV values of alkali activated concrete is not as statistically significant as in cement-based concretes, and they stated that UPV evaluation is not an appropriate assessment for the geopolymer mixtures.

Relationship Between Compressive Strength and Flexural Strength

The relationship between flexural and compressive strength of the mortar samples are shown in Figure 10a. It was observed that there is a robust relationship between compressive strength and flexural strength, irrespective of the curing conditions. Mechanical test results were all used to establish an equation to predict the flexural strength of the alkali-activated mortars by using compressive strength values as the concrete design guidelines generally offer equations to forecast the flexural strength of samples from their compressive strength. Using the equations prescribed by the AS 3600 and ACI 318 standards, flexural strengths of geopolymer mortar samples were predicted and compared with the test results obtained in this study (Figure 10a).

Eq. (5), as outlined by AS 3600, can be used to calculate the characteristic flexural strength at 28-days. The value obtained from Eq. (5) is multiplied by 1.4 and 1.8 to provide the mean and maximum characteristic values, respectively. Eq. (6) describes the correlation between compressive and flexural strength proposed by ACI 318 as shown in Table 4.

Furthermore, Nath and Sarker [36] proposed a model which predicts the flexural strength of FA-based alkali-activated concrete cured at room temperature using the compressive strength results and stated that the flexural strength of alkali-activated concretes exhibited disparities compared to cementitious concrete guidelines. For the same compressive strength values, flexural strength of geopolymer mixes was found to be higher compared to traditional concrete. In this study, it was observed that the test results of AC and MC mixes were somehow compatible with the model proposed by Nath and Sarker [36]. However, when heat curing is applied on the samples (i.e., OC or SC), flexural strength

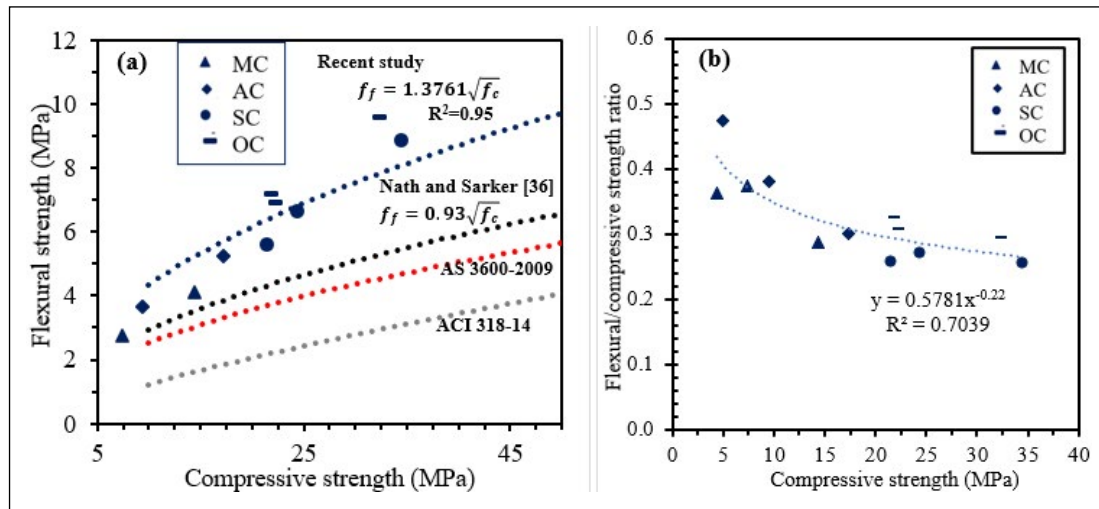


Figure 10. The relation between: (a) flexural strength and compressive strength (b) f/c ratio and compressive strength.

Table 4. Standard equations to predict flexural strength from the compressive strength

Equation	Standard	Equation ID
$f_f = 0.6 \times \sqrt{f_c}$	AS 3600	Eq. (5)
$f_f = 0.62 \times \sqrt{f_c}$	ACI 318	Eq. (6)
$f_c = f_{cm} - 7.0$ for $f_c < 7$ MPa	ACI 318	Eq. (7)
$f_c = f_{cm} - 8.3$ for $21 < f_c \leq 35$ MPa	ACI 318	Eq. (8)
$f_c = \frac{f_{cm} - 5.0}{1.1}$ for $f_c > 35$ MPa	ACI 318	Eq. (9)

For Eq. (5) and (6), f_f and f_c represent the characteristic flexural and compressive strength of mixes, respectively. f_{cm} demonstrates the average compressive strength of mixes. f_c is taken as 90% of average compressive strength for Eq. (5), whereas f_c of the Eq. (6) was calculated using the Eqs. (7)-(9).

of mortars exhibited higher results compared to the other models. Hence, a new equation was developed considering the effects of steam and oven curing on geopolymer samples using Eq. (10) and the results are plotted in Figure 10a. The equation was developed using the average values of compressive strength, and flexural strength results with the total number of data points are 12. In addition, the coefficient of determination (R^2) value of the developed equation was obtained as 0.95.

$$f_f = 1.3761\sqrt{f_m} \quad (10)$$

where, f_m indicates the mean compressive strength of geopolymer mortars.

It is noteworthy to mention that the concrete guidelines are prepared for cementitious concretes and may not be appropriate to use for predicting the flexural strength of geopolymer samples. This may also be observed from Figure 10 which shows that AS 3600 and ACI 318 models fail to estimate the flexural strength considering the results of the present study. Similar finding was reported by Wardhono et al.[37], who studied the mechanical properties of heat cured FA-based geopolymer concrete.

Figure 10b shows the correlation between flexural to compressive strength ratio (f/c) and compressive strength. It is inferred that the increase in the compressive strength decreases the f/c ratio. It has been reported that an increase in the mortar porosity decreased the compressive strength and increased the f/c ratio [38].

CONCLUSION

In the present paper, the effects of various curing regimes on the physico-mechanical properties of FA- and GGBS-based alkali activated mortar specimens were investigated. Based on the experimental studies, the following key bullets can be noted:

- UPV values of AAM samples varied between 2957 and 3235 m/s. Although the superior mechanical properties of OC and SC samples compared to that of AC and MC, the UPV values did not remarkably change in this scale, probably due to the formation of larger pores and micro-cracks resulting from the higher initial curing temperatures at the early ages.
- The flexural and compressive strength of OC and SC samples were superior at all ages compared to AC and MC samples. On the other hand, both compressive and flexural strength development rate of AC and MC was significantly higher compared to that of others which indicate that higher strength values may be achieved in the long term.
- Statistically significant relationship was observed between compressive and flexural strength, irrespective of the curing conditions, and an equation was developed to predict the flexural strength of alkali-activated mortar specimens.
- The curing conditions did not significantly affect the physical properties of alkali-activated specimens. Nevertheless, the maximum water absorption and voids

content values were obtained for AC samples, whereas the lowest values were determined in OC samples.

- The overall test results showed that OC and SC can be preferred in precast applications due to the rapid strength development and superior physical and mechanical properties compared to other mixes. On the contrary, MC can be suggested as the most unsuitable curing method for AAM samples containing GGBS and FA due to the discharge of alkaline solution under moist environment leading to undesired efflorescence and insufficient physical and mechanical properties.

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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