Case study

Impact of curing temperatures and alkaline activators on compressive strength and porosity of ternary blended geopolymer mortars

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ABSTRACT

Impact of changing temperatures of curing (27, 60 and 90 °C) and types of alkaline activator solution on the properties of geopolymer mortars (GPMs) prepared by combining agricultural and industrial wastes including granulated-blast-furnace-slag (GBFS), fly-ash (FA), and palm-oil-fuel-ash (POFA) was examined. Sodium Hydroxide (NH), Sodium Silicate (NS) and NHNS alkaline solutions were used as alkali activators. Proposed GPMs were synthesized using NH solution of molarity 8 M, ratios of alkaline solution to binder were 0.30, NS to NH was 3.0 and binder to fine aggregate (sand) was 1.5. The mechanical properties of the studied GPMs and the products of reaction were greatly sensitive to the variation of mix compositions, alkaline activators type, and temperatures of curing. Furthermore, the formation of crystalline calcium silicate hydrate (C-S-H), calcium aluminium silicate hydrate (C-A-S-H) together with additional amorphous gel led to the compressive strength enhancement of the GPMs as the content of FA was increased and activated with NS solution. A linear correlation was established between compressive strength, ultrasonic pulse velocity and porosity of the proposed GPMs.

1. Introduction

Cement industries worldwide are the major emitters of greenhouse gases (GHGs). In fact, more than 1 ton of CO_2 is generated and 2.5 tons of materials including fuel and other raw materials are consumed for every one ton of ordinary Portland cement (OPC) production [1–5]. Ever-increasing need of OPC by the construction sectors demanded its massive production, and thereby elevated the emission of carbon dioxide (CO_2) [6–9]. To mitigate such problems, use of waste materials from manufacturing industries as an alternative to OPC was thought to be advantageous for the creation of concretes. Such waste materials based concretes reveal improved mechanical performance and durability traits [10–13].

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Recycling the wastes for making useful construction materials is also a key issue towards sustainable development. In this regard, geopolymer played a significant role as an unconventional binding agent for the production of concretes [10,14–17]. Several studies [18–21] mentioned many distinct attributes of geopolymers such as OPC-free production, recycles of abundant wastes, low cost and environmental amiability in terms of low GHGs emission. All these properties made geopolymers potential alternative to traditional concrete for the construction purposes.

Geopolymers are inorganic aluminosilicate polymers that are produced via the alkali activation of alumino-silicate (SiO_2-Al_2O_3) through a process called geopolymerisation. Aluminosilicate materials are generally mined from the earth or produced by the industries as wastes including calcined kaolin or metakaolin, POFA, FA, and GBFS [16,22,23]. Geopolymerization reaction produces a complex three dimensional polymer network [24], wherein the end product (geopolymer) depends largely on the types and proportions of source materials, curing regime and alkaline activators. Amorphous nature of granulated blast furnace slag (GBFS) enriched in CaO, SiO_2 and Al_2O_3 is greatly suitable as geopolymer binder [25,26]. According to Rashad [27], alkali activated GBFS reveals pozzolanic characteristics. Deb et al. [28] advertised the enhanced microstructure and mechanical properties GBFS incorporated FA geoplymer, where the improved characteristics were attributed to the generation of excess calcium resulted from GBFS. A geopolymer binder was prepared by combining GBFS with FA at different ratios, where the concentration and type of alkaline activators was varied [29,30]. Mechanical strength of hydration yields from FA combined GBFS was studied [31], which was shown to withstand a compression strength of 50 MPa at the age of 28 days. Shi and Day [46] confirmed strength improvement of FA combined GBFS mixes at early age, where the activation via alkaline NH solution together with the inclusion of hydrated lime in a small quantity was shown to play vital role in such modification. Furthermore, such FA and GBFS blend resulted in an improved resistivity in coping with the dry shrinkage and creep [32]. A high quantity of slag in the mixture was asserted to reduce the rate of geopolymer concrete deterioration.

Low calcium FA is considered as the main precursor in the geopolymer industry due to its high content of SiO_2 and Al_2O_3 [33,34]. FA is a waste which is created from the burning of coal at power-producing factories. The mechanical strength of geoplymer containing low calcium FA was found to improve after being cured in the temperature range of 40 to 85 °C [35,36]. So far, the major hindrance of using FA by geopolymer industries is related to the achieved poor compressive strength (CS) under ambient condition of curing temperature [22]. Several studies were conducted to improve the structural strength of GPMs produced using FA with the inclusion of CaO as additive [37,38]. Incorporation of CaO was shown to affect greatly the GPMs mechanical strength [39], wherein an excess amount of C-S-H was formed during geopolymerization [40–42]. The achieved GPMs' resistance to compression was found to degrade upon surpassing a certain quantity of calcium. Besides, the resistance to compression revealed a degradation after two weeks of curing for calcium peroxide content above 10% [43].

Malaysia is one of the largest palm oil producing nations beside Thailand and Indonesia. Malaysia produces approximately 0.1 million tons of POFA annually and the amount will continue to rise in next few years with increasing number palm oil tree plantation [44,45]. Presently, excessive amount of palm oil by-product that is released to the water bodies [46] poses a threat to the environment. Some research findings revealed that such waste by-products can be used for sustainable constructions and development [46–48]. Implementation of such silica-rich by-products can mitigate the environmental concerns generated by large OPC production [42,49–51]. Recent studies [52,53] showed that the strength of the concrete can be enhanced using POFA as replacement to OPC. The richness of silicate and low content of aluminium often limits the use of POFA as a main binder in geopolymer production [54]. Rakhimova and Rakhimov [55] acknowledged that activators comprising of NH and NS are beneficial for the enhancement of geopolymers' mechanical strength made of GBFS and FA. Alkaline activation of GBFS led to produce C-S-H and/or C-A-S-H gels akin to OPC [56] while N-A-S-H gel was formed due to alkaline activation of FA [57]. Meanwhile, the glassy phase of GBFS allowed stronger alkali activation compared to FA. Moreover, to facilitate the accelerated reaction in the crystalline phase of FA, temperature between 40 to 85 °C was required [27,58]. Incorporation of GBFS into FA based geopolymers was found to improve its overall properties [28]. Despite many dedicated research effort, the CS and porosity of ternary geopolymers made from GBFS, FA and POFA cured at different temperatures and alkaline activator solutions has not been evaluated.

Although different source materials can be used to prepare geopolymer binders, the production of high-performance geopolymer binder by blending FA and POFA with GBFS is an emerging trend. Thus, FA, POFA and GBFS are regarded as very useful source materials for producing geopolymer binder. Despite many dedicated research efforts the effects of curing temperature, alkaline solution types, and the ratio of waste materials in mix design on the performance of ternary GPMs/ concrete has not been clearly understood. In this view, we evaluated the effects of varying curing temperatures and alkali activator solution types on the CS, ultrasonic pulse velocity (UPV) and porosity of GPMs made by blending GBFS, FA and POFA with natural sand as fine aggregates. GBFS was incorporated into low calcium FA and POFA mixes and subsequently activated using alkaline NH, NS and NHNS solutions. The performance of the proposed ternary GPMs was assessed in terms of porosity, CS, UPV, setting time, and workability. The impacts of varying curing temperatures, calcium to silicate ratios, and alkali activator types on the properties of the proposed GPMs were determined.

2. Materials and methods

2.1. Constituent materials selection

In this work, the three components (GBFS, FA and POFA) were blended to produce the GPMs. Table 1 enlists the detail proportions and physical properties of GBFS, FA and POFA obtained from X-ray Fluorescence (XRF), particle size analyser

Table 1
Physical and chemical properties of GBFS, FA and POFA obtained from XRF analysis and.

Material	GBFS	FA	POFA
SiO_2	30.8	57.20	64.20
Al_2O_3	10.9	28.81	4.25
Fe_2O_3	0.64	3.67	3.13
CaO	51.8	5.16	10.20
MgO	4.57	1.48	5.90
K_2O	0.36	0.94	8.64
Na_2O	0.45	0.08	0.10
SO_3	0.06	0.10	0.09
Physical properties			
Density(kg/m^3)	2900	2200	1900
Specific gravity	2.9	2.2	1.9
Surface area-BET (m^2/g)	13.6	18.1	23.1
Mean diameter (μm)	12.8	10	8.2
Colour	Off-white	grey	Dark-grey

(PSA) and Brunauer, Emmett and Teller (BET) measurements. GBFS was produced from quenched iron slag melt (a spin-off of iron and steel manufacturing in blast furnace) in water. The resultant amorphous granular material was dried and crushed to get fine powders. The geopolymer binder GBFS was acquired from Ipoh (Malaysia).The unique pozzolanic and cementitious characteristics of GBFS were attractive as supplemental cementitious resources. It developed a distinctive hydraulic reaction upon mixing with water and produced a shade of white tone. Presence of alumina, silica and calcium above 90% in GBFS is responsible for its Pozzolonic characteristic. The specific surface area of GBFS was 13.6 m^2/g and medium particle size of 12.8 μm (Fig. 3a). Fly Ash was consisted of fine particulates produced from burned fuel that was ejected from the boilers (fired by coal) alongside emitted gases from chimney. Class F FA was collected from Tanjung Bin Energy Power Plant (Malaysia). The total chemical content FA was consisted of 86% SiO_2 and Al_2O_3 (Table 1). The exact specific gravity of FA was 2.20 with a particle size of 10 μm (median) and Blaine fineness of 18.1 m^2/g.

Palm Oil Fuel Ash (POFA) is a spin-off product of burned fibers, shells and empty fruit bunches as fuel in palm oil mill boilers. Untreated POFA was acquired from Malaysian palm oil mill. Partially combusted shells and fibres were withdrawn through a 300? ?m strainer and the fully combusted ones were rid of moisture through oven-drying over one day at 105 °C. Subsequently, it was ground with Los Angeles machine to reach a granularity of 10? ?m. Upon reaching 12,600 revolutions after 6 h the POFA met the required fitness (above 66%) with a grey tone. Pozzolan that passed a 45? ?m wet sieve was accrued at 86% in accordance to ASTM:C618-12a standard [59]. Table 1 depicts the chemical composition and physical properties of POFA.

Fig. 1 shows the X-ray diffraction pattern (XRD) of the constituent raw materials (GBFS, FA and POFA) of GMPs. The presence of a halo in the angular range of 29 to 40° and absence of any sharp crystalline XRD peaks of GBFS verified its true amorphous nature, where a tiny quantity of magnetite is seen. Present findings are in good agreement with other work reported in the literature [30]. The presence of a broad hump in the range of 18 to 62° and few sharp crystalline diffraction peaks of FA indicated its dominant amorphous phase and crystalline phases of quartz (Si), (Al_4Ca), mullite ($Al_6Si_2O_{13}$), calcium silicate ($CaSiO_2$), and magnesium silicate (Mg_2Si). Likewise, POFA was comprised mostly of amorphous phase
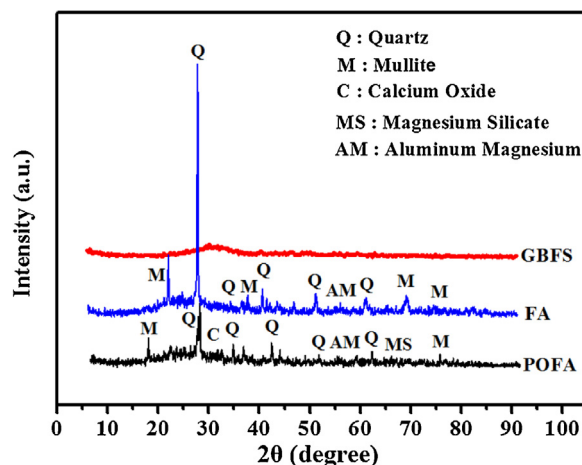

Fig. 1. XRD pattern of GBFS, FA and POFA.

revealing a halo in the range of 26 to 51° together with some crystalline phases assigned to (Ca_2Si), (Mg_2Si), ($Al_{13}Fe_4$), and quartz (Si).

Fig. 2 illustrates the scanning electron microscopy (SEM) images of GBFS, FA and, POFA used as constituents of GPMs to obtain improved strength and enhanced microstructures. The observed uneven and bony particles in GBFS was consistent with the previous report [60]. Moreover, FA and POFA manifested even spherically shaped particles.

Fig. 3b presents the particle size dependent cumulative passing percentage of natural river sand (conforming to ASTM C33 standard) which was used as fine aggregate to prepare the proposed GPMs. The sand was cleaned using water in accordance with ASTM C117 standard to diminish the silt and impurity contents. Subsequently, the cleaned sand was oven dried at 65 °C for 24 h. The modulus of fineness and specific gravity of sand were 2.1 and 2.6, respectively.

The solution of NH, NS and a mixture of NH (purity of 98%) plus NS was used as alkaline activators for alumina and silica present in GBFS, FA, and POFA. The NS mixture was comprised of SiO_2 (29.5 wt%), Na_2O (14.70 wt%), and H_2O (55.80 wt%). These were purchased from Quality Reagent Chemical Asia (QREC, Malaysia). To make NH solution (8 M), the pellets of NaOH were suspended inside water. The second mixture was using NS with NH (8 M) with NS:NH ratio of 3.0. The other solution was NS without containing NH.

2.2. Mix proportion

The effects of different GBFS:FA:POFA levels (weight%), alkaline activator solution types (molarity (M) and solution modulus (Ms)) and curing temperatures (in °C)on the properties total nine GPM mixes were determined as depicted in Table 2. Three levels of ternary blended (GBFS:FA:POFA) 80:10:10, 75:15:10 and 75:10:15 were considered in the mix design. Alkali activators including NH, NS, and a mixture of NH and NS were used. The achieved GPM mixes were cured at different temperatures 27, 60 and 90 °C to study the effect of curing temperature on high calcium geopolymer binder. The ratios of binder to aggregates (B:A), solution to binder (S:B) and molarity (M) of all solutions were maintained constant (Table 2). Impact of alkali solution types, ratios of GBFS to FA to POFA and curing temperatures on the CS, UPV and porosity of GPMs were evaluated.

The studied GPMs were prepared by assembling GBFS, FA and POFA with fine aggregates homogeneously in a mortar mixer for 3 min before the alkali solution was added. However, the alkaline solution was prepared before 24 h and left to be cool before adding to geopolymer matrix including ternary binder and fine aggregate. After added the alkaline solution, the materials were mixed for another 4 min before casting in moulds. All nine cubic specimens of dimension 50 mm × 50 mm × 50 mm were casted with GPMs in double layers wherein every layer was shacked in vibration table for 15 s. Next, these moulds were kept at room temperature (27 °C) and the upper surface was exposed to air to maintain 75% of relative
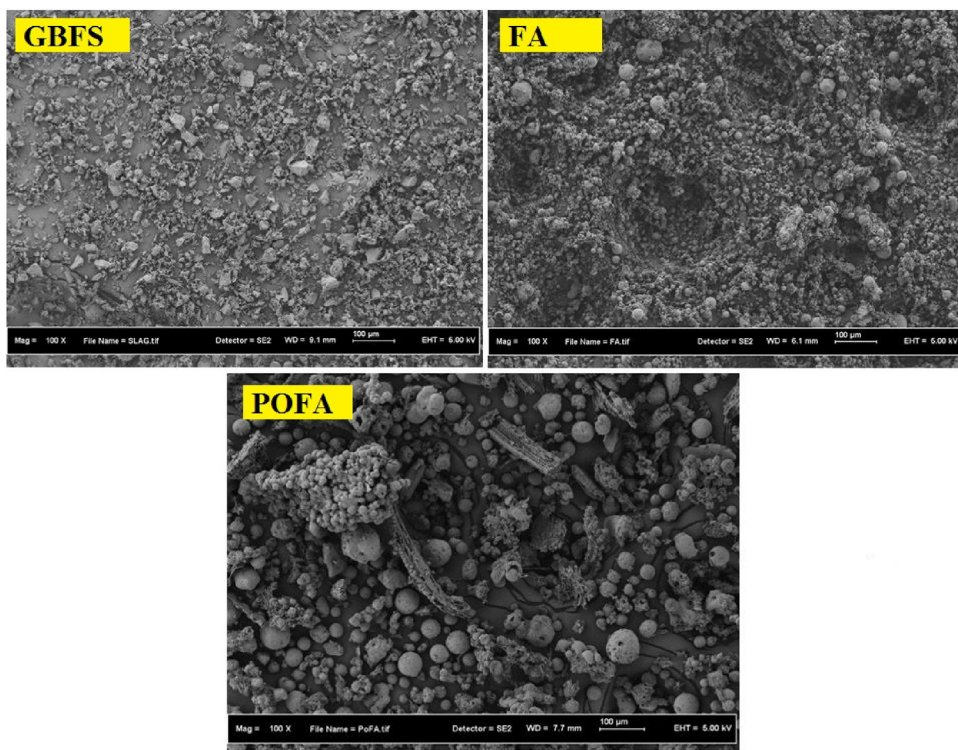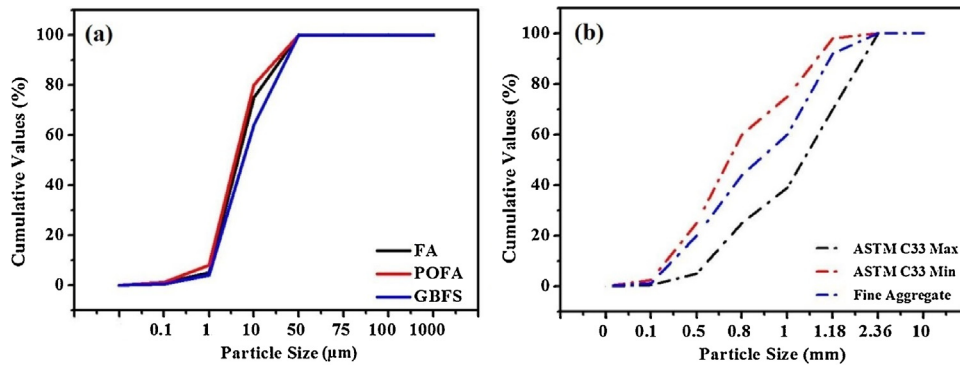

Fig. 2. SEM images of GBFS, FA and POFA.

Fig. 3. Particle size analysis of (a) Binders included FA, POFA and GBFS (b) Sand as fine aggregate.

Table 2
Proposed GPMs prepared under varying conditions.

Mix	Binder (weight %)			B:A	S:B	Alkali Activator			Curing Temperature (°C)
	GBFS	FA	POFA			Ms	M	Types	
GP$_1$	80	10	10	1.5	0.3	1.6	8	NHNS	27, 60, 90
GP$_2$	80	10	10	1.5	0.3	0	8	NH	27, 60, 90
GP$_3$	80	10	10	1.5	0.3	2.01	8	NS	27, 60, 90
GP$_4$	75	15	10	1.5	0.3	1.6	8	NHNS	27, 60, 90
GP$_5$	75	15	10	1.5	0.3	0	8	NH	27, 60, 90
GP$_6$	75	15	10	1.5	0.3	2.01	8	NS	27, 60, 90
GP$_7$	75	10	15	1.5	0.3	1.6	8	NHNS	27, 60, 90
GP$_8$	75	10	15	1.5	0.3	0	8	NH	27, 60, 90
GP$_9$	75	10	15	1.5	0.3	2.0	8	NS	27, 60, 90

humidity. Every specimen was de-moulded after 6 h before being cured at 27 °C. Other 18 samples (nine samples in each batch) were oven cured for 24 h at 60 °C and 90 °C to determine the impact of curing temperature on the properties of GPMs. Table 3 summarizes the detail composition of binder for each mix together with their ratios depended on chemical composition of raw materials (Table 1) and mix design (Table 2).

Fig. 4 demonstrates effect of GBFS:FA:POFA levels dependent variation in the mass ratios of CaO, SiO$_2$ and Al$_2$O$_3$. The amount of CaO was diminished with increasing of FA and POFA level as substitution of GBFS. Furthermore, the content of SiO$_2$ was raised with the increasing POFA level. Excess quantity of Al$_2$O$_3$ (13%) was achieved for the mixture consisted of 15% of FA (Table 3 and Fig. 4).

2.3. Test program and procedure

The United States of Environmental Protection Agency (US EPA) instituted the toxicity characteristics leaching procedure (TCLP) that has broadly been used to classify harmful solid wastes and evaluate the highest leachate contents during land filling. About 50 g of GBFS, FA and POFA specimen (oven dried) was weighed and put in High Density Polyethylene (HDPE) bottles of volume 2 L. The extract was removed from the bottle after 18 h in the liquid to solid proportion of 20:1. The contents of weighty metals such as Arsenic (As), Beryllium (Be), Cadmium (Cd), Chromium (Cr), Copper (Cu), Nickel (Ni), Lead (Pb), Titanium (Ti) and Zinc (Zn) in the raw materials (GBFS, FA and POFA) are tested to guarantee the materials' safety.

The fresh properties of the synthesized mixes such as flow ability and setting time were measured in accordance with relevant standards. The ASTM C1437 standard was followed for the flow measurement of GPMs. The initial and final setting times of the GPMs were recorded on a manual VICAT apparatus following the ASTM C187 procedures. The CS test was conducted according to ASTM C109 standard. The results for each mixture were averaged on three curing ages (1, 7 and 28

Table 3
Composition of binders and corresponding proportions.

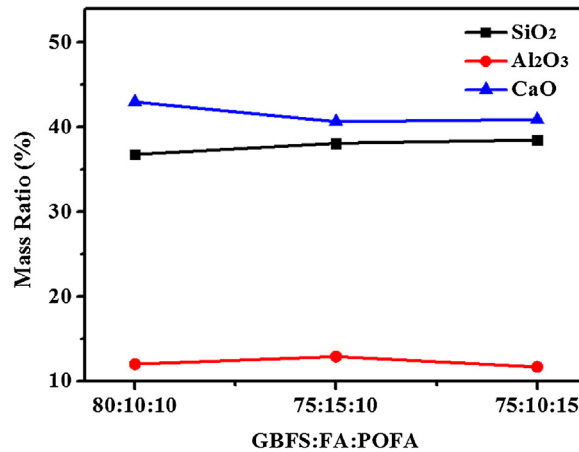| Mix	SiO$_2$	Al$_2$O$_3$	CaO	CaO:SiO$_2$	SiO$_2$:Al$_2$O$_3$
GP$_{1-3}$	36.78	12.03	42.98	1.16	3.06
GP$_{4-6}$	38.10	12.93	40.65	1.06	2.94
GP$_{7-9}$	38.50	11.70	40.90	1.06	3.29

Fig. 4. Effect of GBFS:FA:POFA levels on the mass ratios of SiO_2, CaO and Al_2O_3.

days) and curing temperature. The specimen was subjected to a constant load rate (2.5 kN/s) to inspect its failure. The UPV was measured following ASTM C597 standard. The test was carried out on cubic sample cured for 28 days. A total of three samples were analysed at every curing temperature.

Porosity of the proposed GPM was determined using vacuum saturation method. In this method, standard cubic sample each of dimensions (50 mm $\times$ 50 mm $\times$ 50 mm) and weight W_A (in gm) was oven dried at 105 °C for 24 h before being cool down at ambient temperature. The weight (W_D in gm) of the cooled specimens was recorded before they were subjected to vacuum condition for 3 h. Then the specimens were totally immersed in de-aired distilled water for about 24 h and weighted (W_W in gm).

3. Results and discussion

3.1. Toxicity characteristic leaching procedure (TCLP)

The toxicity of GBFS, FA and POFA were tested via TCLP. Method recommended by US EPA was used in TCLP. Table 4 summarizes the TCLP outcomes compared to conventional procedures. Results revealed that all elements had weaker leaching compared to standard convention as documented in US EPA SW-846. Furthermore, the leaching of heavy metals from GBFS, FA and POFA were unimportant and thus can be disregarded. Actually, wasted materials such as GBFS, FA and POFA might be exploited in construction sectors to produce sustainable green mortars and concretes.

3.2. Workability

Fig. 5 presents the measured flow diameter of the mortar as a function of changing GBFS:FA:POFA levels and alkali solution. Irrespective of the types of alkali solution, an increase in the content of FA and POFA from 10 to 15% led to reduce the workability of the mortar (flow diameter). Besides, an increase of FA level from 10 to 15% in place of GBFS at fixed POFA content led to reducing the flow diameter with changing NHNS, NH and NS alkali solution types. Yet again, a reduction in the GBFS content and increase in POFA at fixed FA content (10%) led to reduce the workability of GPMs regardless of the types of alkali solution (NHNS, NH and NS). However, the spherical nature of FA affected on workability of the geopolymer mixture prepared with 15% FA and presented higher flow diameter comparing to 15% POFA. Some research displayed that materials having large specific surface area could have negative impact on the mortar's flowability [61]. Hassan et al. [48,62] showed that POFA microstructures possessed large BET surface area as much as 23.75 m²/g because of the presence of high porosity and uneven (nonuniform) particles. It was disclosed that to increase the binder's volume fraction and surface area high doses

Table 4
TCLP of GBFS, FA and POFA materials.

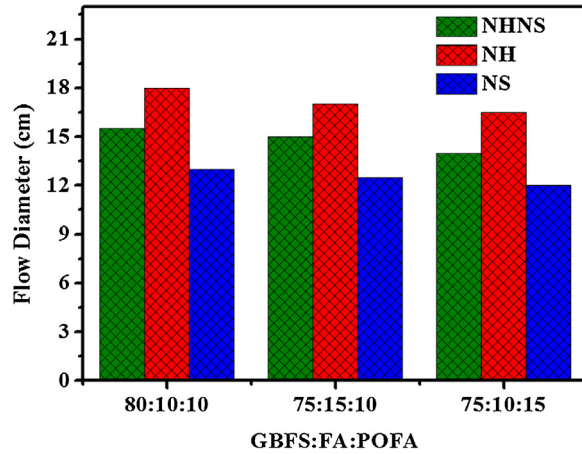| Materials	Trace Elements (Concentration (mg/L))								
	As	Be	Cd	Cr	Cu	Ni	Pb	Ti	Zn
GBFS	0.034	0.004	N.D	N.D	0.006	0.182	N.D	0.012	0.45
FA	0.093	0.019	0.011	0.294	0.037	0.082	0.024	0.008	0.047
POFA	0.107	0.008	0.002	0.167	0.031	0.056	0.011	N.D	0.326
TCLP limit	5	0.5	1	5	1	2	5	2	5

Fig. 5. Effect of GBFS:FA:POFA levels and alkali solution on the workability of GPMs.

of water could be necessary at high POFA percentage for attaining the saturated flowability. All GPMs mixtures prepared with NH solution showed higher workability compared to those made with NHNS and NS solution. Furthermore, the flow diameter was shrunk from 18 to 13 cm as the sodium silicate (NS) content was increased. By using NS without NH the mortar's flowability could be lowered because of highly viscous nature of the former one.

Fig. 6 shows the impact of GBFS:FA:POFA levels and alkali solution on the setting time of ternary blended GPMs. Three levels of GBFS:FA:POFA were used. An increase in the FA content from 10 up to 15% and subsequent reduction of GBFS content was found to increase the initial (8 to 10 min) and final setting time (14 to 16.5 min) of the mortar. In NHNS alkali solution (Fig. 6a), the replacement of GBFS by increasing POFA content was shown to elongate the initial and final setting time. The batch mixture containing 10% of FA, 75% of GBFS and 15% of POFA displayed the highest initial (13 min) and final (18 min) setting time. The slow rates of both setting times with the increase in FA and POFA levels could reduce the content of CaO (Table 3 and Fig. 4). Increasing SiO_2 content and decreasing CaO content could restrict the C-S-H and C-A-S-H gels formation
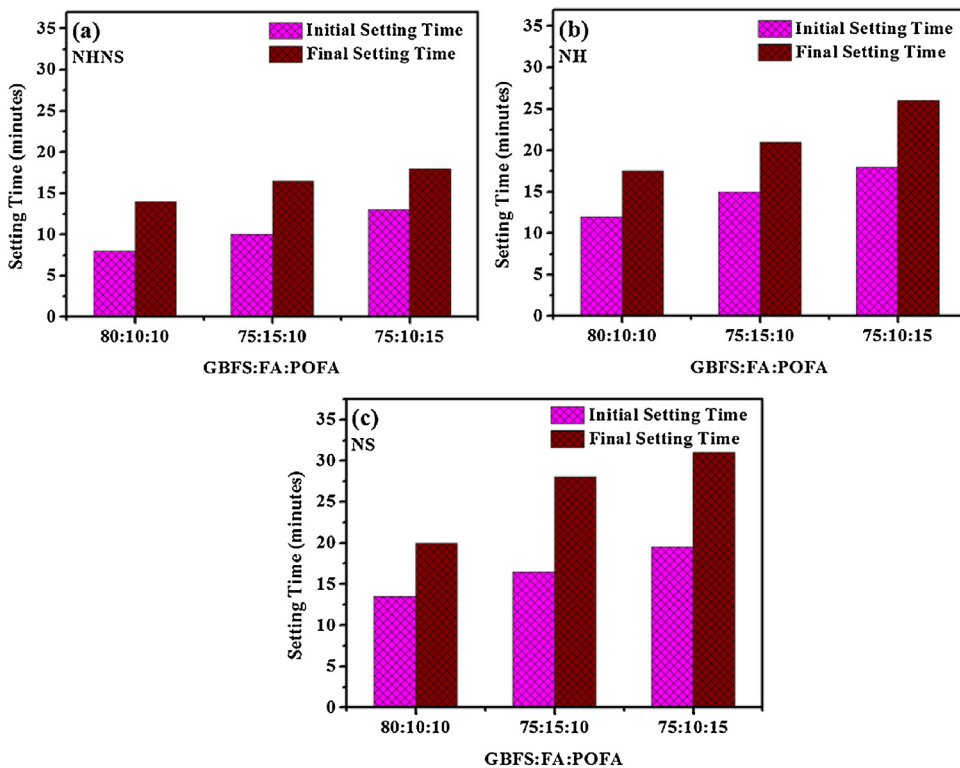

Fig. 6. Effect of GBFS:FA:POFA levels and alkali solution on the mortars setting times.

and subsequently elongate the time of setting. Mortars prepared with NHNS solution activation was shown to possess shorter setting time than those prepared with NH (Fig. 6b) and NS (Fig. 6c) one. This observation agreed well with the earlier observation wherein the inclusion of NH together with NS was discerned to accelerate the process of geopolymerization and thereby led to mortar's hardening.

3.3. Compressive strength

The influence of different GBFS:FA:POFA levels, alkali solution and curing temperatures on the CS of ternary blend GPMs are displayed in Fig. 7. The strength values were determined at the curing age of 1, 7 and 28 days. The gain in CS was monotonically increased with increasing age. Fig. 7(a–c) shows the effect of FA and POFA replaced GBFS on the strength of GPMs at 1 day of age. Mortars designed using the solution of NHNS and cured at 27 °C revealed a reduction in the early CS from 35.7 to 33.9 MPa after 24 h as the content of FA and POFA were increase from 10 to 15%, respectively (Fig. 7a). The CS of all the GPMs prepared using solution of NHNS and cured in the oven at 60 °C and 90 °C were reduced as the curing temperatures was raised. Conversely, the C-S-H formation influenced by increase the curing temperatures, leading to coarser microstructure of GPM with increased porosity and cracks. Mixture containing 80% of GBFS, 10% of FA and 10% of POFA displayed a reduction in the strength from 35.7 to 19.6 MPa when the curing temperature was raised from 27 to 90 °C. Similar trend was observed for the GPM containing 15% of FA or POFA replaced GBFS where the strength was dropped with the increase of curing temperature. All the mixtures prepared with NH solution presented a very low strength (in the range of 1.9 to 5.3 MPa) irrespective of curing temperatures (Fig. 7b). The effect of NS mixture on the early strength of various GPMs was studied. The CS was increase from 38.1 to 38.6 MPa with rising FA content up to 15% in place of GBFS. However, the strength was dropped to 37.4 MPa when the GBFS was replaced by POFA. GPMs designed with NS mixture presented more CS compared to those activated with NHNS and NH alkali solution (Fig. 7c). Usage of NS only allowed to the production of additional silicate and thus accelerated the process of geopolymerization as well as CS improvement.

The CS development was continued after 7 and 28 days and showed an increase percentage higher than 160% for all alkali solution types and curing temperatures (Fig. 7d–i). The recorded strength values were 57.1, 58.2 and 56.4 MPa for GPMs made
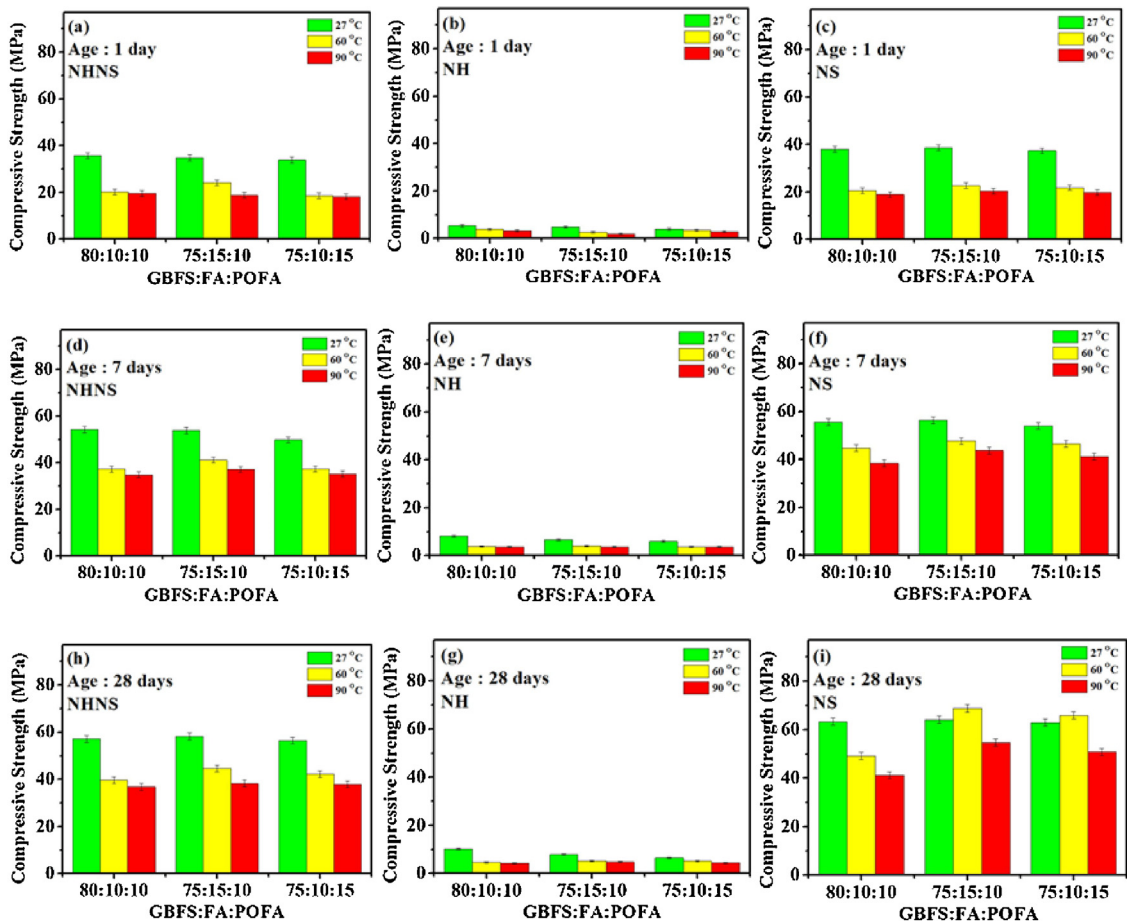

Fig. 7. Effect of GBFS:FA:POFA levels, alkali activators and curing temperatures on the strength of GPMs at various ages.

using NHNS mixture and cured at 27 °C. From the results, it is observed the replaced GBFS by FA with 5% led to enhance the CS after 28 days (Fig. 7h). The keep FA content at 10% and replaced GBFS by POFA with 5% led to reduce the strength to 56.4 MPa compared to 57.1 MPa. The CS of all specimens activated using NHNS mixture showed lower CS when cured at elevated temperatures (oven curing) compared to the one cured at 27 °C. After 28 days, it was found all the GPMs mixtures activated using NS mixture presented the greater CS compared to NHNS alkali solution as shown in Fig. 10. The value of CS was 63.3, 64.1 and 62.9 MPa at ambient curing and 49.2, 68.8 and 65.9 MPa at 60 °C. The replaced GBFS by FA and POFA have effected to enhance the strength at oven curing condition. The increased curing temperature from 60 to 90 °C led to reduce the strength to 41.2, 54.7 and 50.8 MPa compared to results of specimens cured at 60 °C. However, GPMs activated NS mixture displayed higher CS at 60 °C (68.8 and 65.9 MPa) at 28 days curing age compared to the specimen cured at 27 °C and 90 °C for mixtures replaced GBFS by FA and POFA respectively.

Rashad [27] reported a decrease in the CSs of GPMs (cured at ambient temperature) with the increase of FA and POFA content, where several factors were attributed to the observed reduction. First, the difference in chemical composition of the GBFS, FA and POFA which significantly affected the process of alkali activation of the binders. Second, the lower reaction rate of FA and POFA than GBFS allowed the partial dissolution of the constituents [63]. Third factor was associated with the lowering of the compactness and density of the geopolymer matrix with increasing FA and POFA content. The fourth factor was linked to the low molarity of NH (8 M) where the CS mainly depended on the calcium oxide content that replaced the low amount of sodium oxide. The reason being that the use high GBFS content (80%) led to form more C-S-H and C-A-S-H gels beside the N-A-S-H gel which could enhance the strength of the GPMs. By increasing the FA and POFA level up to 15% a reduction in calcium oxide content from 43 to 40.6% (Fig. 4) was achieved which affected the hydration process and reduced the C-S-H gel product.

The influence of POFA substitution to GBFS on the CS of the GPMs was evaluated (Fig. 7). It was shown that an increase of POFA level to 15% in high volume GBFS matrix could reduce the CS by 1.2%. The mixture comprised of 15% of POFA, 10% of FA and 75% of GBFS displayed a strength of 56.4 and 62.9 MPa after 28 curing at ambient temperature with NHNS and NS alkali solution activation, respectively. These values were lower compared to the mixture that was cured at 60 °C and activated with NS alkali solution, which exhibited a CS of 65.9 MPa after 28 days. The early CS was more influenced by the increasing content of GBFS than the one obtained after 28 days. Briefly, GPMs containing higher level of FA and POFA activated with NS alkali solution and cured at 60 °C revealed higher strength.

3.4. Effect of CaO:SiO$_2$ and SiO$_2$:Al$_2$O$_3$

Fig. 8(a) displays the influence of varying temperatures on the CS of GPMs at 28 days of age for varying CaO:SiO$_2$ and SiO$_2$:Al$_2$O$_3$ levels prepared with NHNS alkali activator. A higher strength (58.2 MPa) was achieved at lower ratio of Si to Al (2.94) for GPMs cured at ambient condition and activated with NHNS solution. The value of CS was reduced from 44.6 to 38.2 MPa with the increase of curing temperature from 60 and 90 °C respectively. Nonetheless, the strength was influenced remarkably with rising POFA level and dropped to 56.4 MPa (Fig. 11). This lowering in the CS values was ascribed to the high level of SiO$_2$:Al$_2$O$_3$ (3.29) and low ratio of CaO to SiO$_2$ (1.06) (Table 3 and Fig. 4). An increasing amount of POFA in the GPM led to a reduction in the Al$_2$O$_3$ and CaO content and augmentation of SiO$_2$ content. It was acknowledged that [64] the Al component could easily dissolved than Si element. This enabled a higher rate of condensation between silicate and aluminate species than the condensation among silicate species only.

Fig. 8(b) shows the effects of different curing temperatures on the CS of GPMs at 28 days of age for varying Ca:Si and Si:Al levels prepared with NS alkali activator. The CS of GPMs at 28 days of age revealed highest value of 64.1 MPa for Si to Al ratio of 2.98 (cured at 60 °C) which is consisted with earlier reports [42,49,50]. The GPMs containing lower level of Ca:Si (1.06) showed a slightly decrease in the strength from 68.8 and 65.9 MPa at 60 °C than the one revealed at 27 °C. However, at 90 °C the slow rate of geopolymerization process led to further reduction in the CS for all GPMs. Meanwhile, the binder containing highest amount of Ca:Si (1.16) activated with NS exhibited lowest strength at 90 °C compared to other temperatures.
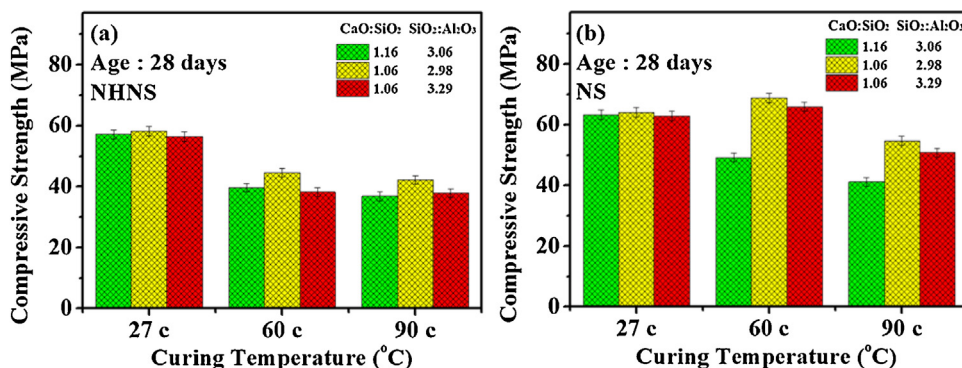

Fig. 8. Effect of curing temperatures, Ca to Si and Si to Al ratios the CS of GPMs at 28 days prepared with (a) NHNS (b) NS solution.
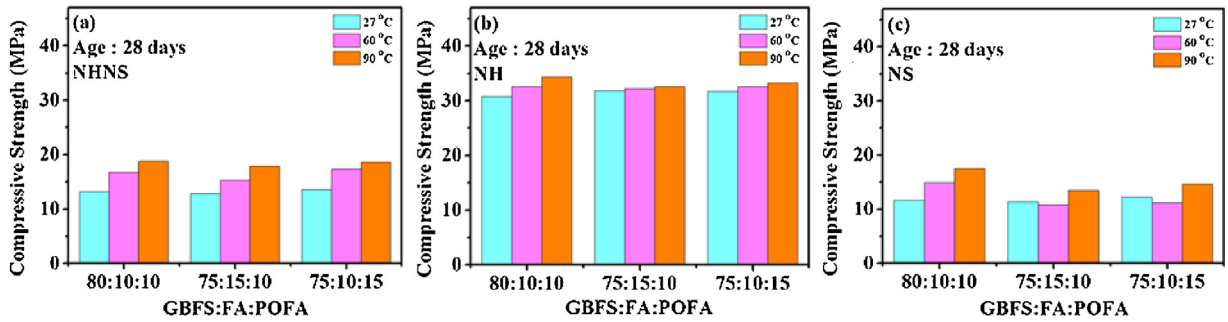
Fig. 9. Effect of varying GBFS:FA:POFA levels and curing temperatures on the UPV of GPMs prepared with (a) NHNS (b) NH (c) NS alkali activator solution at 28 days of age.

However, the reduction in Al_2O_3 content with 15% of POFA replacement affected to reduce the strength comparing to mixture prepared with 15% FA. It's well knows that the Al is more readily dissolved than Si and will result in a higher rate of condensation. It was affirmed that increasing temperature for curing could affect the formation of C-S-H gel, leading to the generation of coarser GPMs microstructures with high porosity and cracks [49,65].

3.5. Ultrasonic pulse velocity

Fig. 9(a–c) depicts the impacts of different curing temperatures, GBFS:FA:POFA levels and alkali activators type on the UPV of the GPMs at 28 days of age. An increase in the FA content from 10 to 15% could enhance the microstructure of specimens, and thereby enhanced the UPV from 3526 to 3537 m/s with NHNS activation and from 3671 to 3724 m/s with NS activation when cured at ambient temperature (27 °C). Similar trend was maintained for high FA content GPMs cured at 60 °C prepared with NHNS or NS alkali solution. Conversely, an increasing content of POFA could reduce the UPV of GPMs cured at 27 °C. GPM comprised of GBFS:FA:POFA levels of 75:10:15 showed the higher UPV (3148 and 3778 m/s) than the specimen prepared with GBFS:FA:POFA levels of 75:10:10 activated with NHNS and NS alkali solution and cured at 60 °C. In brief, most of the specimens activated with NS solution displayed very high UPV (Fig. 9c) than the one activated with NHNS and NH solutions.

Fig. 10 illustrates the correlation of compressive (fc) strength and ultrasonic pulse velocity (UPV) of GPMs after 28 days of curing. The linear regression method was applied to correlate the experimental data. The achieved value of R^2 being 0.99 for all samples could signify a good confidence for the correlation. Following linear regression was used to correlate fc with UPV:

$$UPV = 35.603fc + 1498 \ (R^2 = 0.9912) \tag{1}$$

3.6. Scanning electronic microscopy image (SEM)

The EDS spectra corresponding to the scanned area from FESEM images of different GPMs illustrated in Fig. 11, for samples with CaO to SiO_2 ratio of 1.08 with NHNS solution (Fig. 11a), CaO to SiO_2 ratio of 1.41 with NHNS solution (Fig. 11b) and CaO to SiO_2 ratio of 1.08 with NS solution (Fig. 11c). It was found that these precipitates/intermetallic compounds with the SiO_2 addition are related to two phases: $Ca_2SiO_4.H_2O$ and $Al_2SiO_4.(OH)_2$. Increase in Ca/Si ratio was also noticed and indicated that
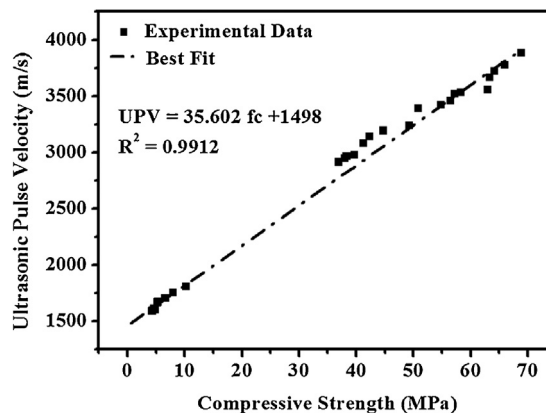

Fig. 10. CS and UPV correlation for the proposed GPMs after 28 days of curing prepared with various alkali activators solution.
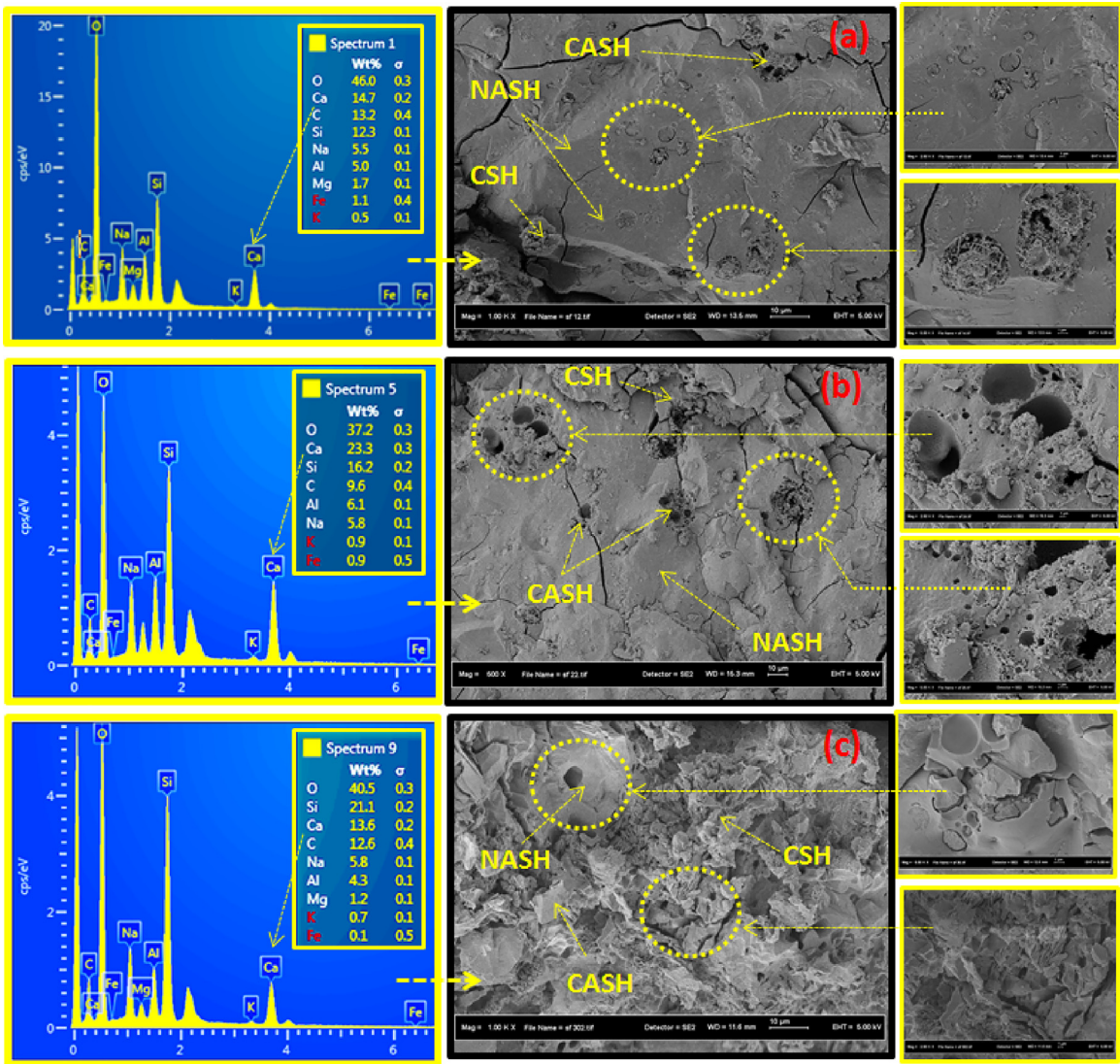
Fig. 11. SEM images and EDS spectra of samples having CaO to SiO$_2$ ratio of (a) 1.06 with NHNS solution (b) 1.16 with NHNS solution (c) 1.06 with NS solution.

this phase has a stronger influence on the developed strength. It is seen from Fig. 11b that the percentage of calcium is more than 23% in comparison to the results shown in Fig. 11a (less than 15%), which lead to development of compressive strength of GP1 after 7 days in comparison to GP4. Also the percentage of Na and Al in GP1 from scanning showed better results, contributing for the increase in strength of geopolymer. Effect of activator solution type on the strength of geopolymer and development of geopolymerization process was clearly evidenced. The silicate ratio of mortar activated with NS showed higher percentage than silicate mortar prepared with NHNS solution.

3.7. Porosity

Fig. 12(a–c) shows the effect of GBFS:FA:POFA levels, alkali solution type and curing temperatures on the porosity of GPMs. The porosity was increased with the increase of curing temperatures for all GPMs mixtures prepared with NHNS and NH alkali solutions. An increase in curing temperatures led to an increase in the porosity of the GPMs from 13.2% to 18.8% with increased curing temperature from 27 to 90 °C. Similar trend was observed for GPM with 15% of POFA replacement to GBFS and activated with NHNS solution. When the POFA level as alternative to GBFS was raised from 10 to 15% in the presence of 10% FA in GPM it revealed to drop in the porosity from 13.2 to 13.6%. This observation was supported by other reports [22,66] wherein it was shown that a reduction in GBFS content in the geopolymer matrix and activated with NHNS solution could produce poor structure with high porosity. For specimens prepared with NS solution presented lower porosity comparing to specimens activated with NHNS and NH solutions (Fig. 12c). However,
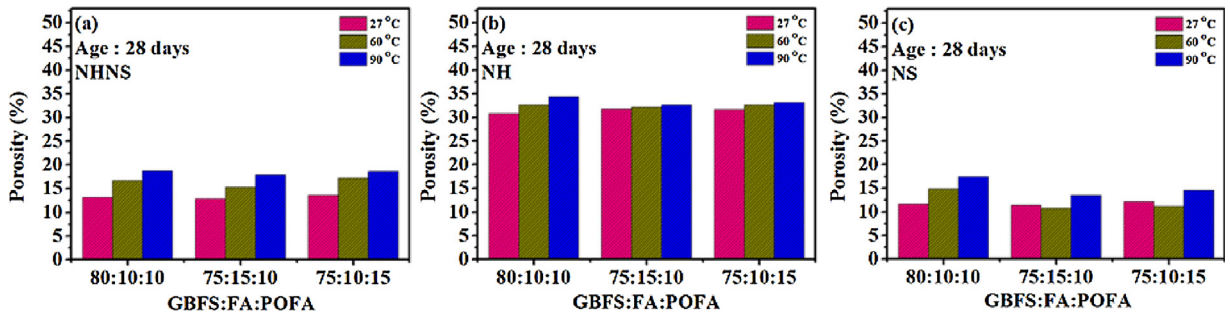
Fig. 12. Effect of changing GBFS:FA:POFA levels, temperatures of curing and types of alkali solution on the porosity of GPMs at 28 days.

the lowest value of porosity (10.8%) was achieved for sample containing 15% of FA, NS solution activation and curing temperature of 60 °C.

Fig. 13 displays the relationship between the porosity and CS of achieved GPMs for varied temperatures of curing. The observed linear correlation between the porosity values (P) and the CS (fc) was in good agreement with other reports [19,67]. The linear regression method (with R^2 values 0.97–0.99) was applied to correlate the experimental data which signified good confidence. Following relations was used for samples cured at temperatures 27, 60 and 90 °C:

$$P = -0.3620 fc + 34.351\,(R^2 = 0.9982) \tag{2}$$

$$P = -0.3608 fc + 33.434\,(R^2 = 0.9701) \tag{3}$$

$$P = -0.4150 fc + 34.924\,(R^2 = 0.9889) \tag{4}$$

The porosity values were found to be correlated with UPV (Fig. 14). The linear regression method was applied to correlate the experimental data. The achieved value of $R^2 \approx 0.99$ for all GPMs indicated a good confidence for the correlation. Following linear regression was used:

$$P = -0.0104 UPV + 49.61\,(R^2 = 0.9933) \tag{5}$$

4. Conclusions

This paper determined the influence of varying curing temperatures (27, 60 and 90 °C) and alkali activator types (NH, NS and NHNS) on the CS, UPV and porosity of a series of ternary GPMs. These ternary mortars were synthesized by blending various waste materials such as GBFS, FA, and POFA, where natural sand was used as fine aggregates. Proposed GPMs were systematically characterized using various tests. The main idea was to mitigate the environmental concern raised by the overproduction of OPC and dumping of the plentiful industrial wastes as land fillers. The feasibility of using such wastes as
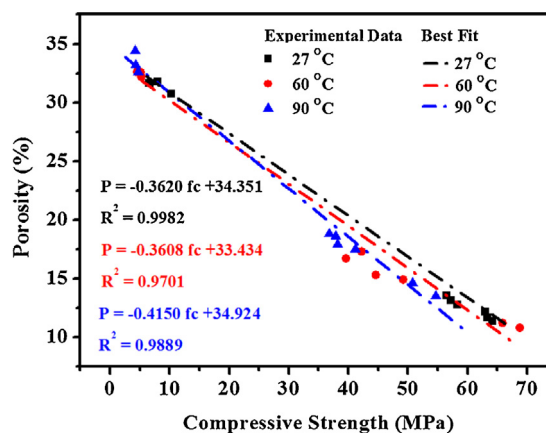

Fig. 13. Relationship between CS and porosity of GPMs at different curing temperatures.
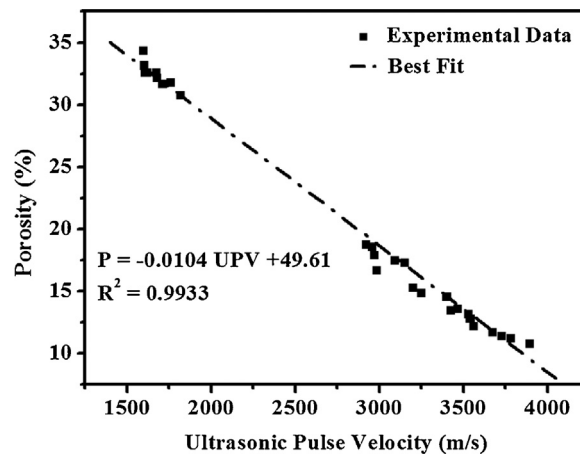
Fig. 14. Relationship between UPV strength and porosity of GPMs.

alternative materials to OPC for achieving mortar with improved attributes in an easy and cost-effective way was explored. Depending on diverse analyses outcomes underlined conclusions are drawn:

i) i) Irrespective of the temperatures of curing and types of alkali solution activation the workability of GPMs was decreased with rising FA and POFA content as alternative to GBFS due to enlarged specific surface area of both materials compared to GBFS. However, the setting time of GPMs was extended with rising FA and POFA content.

ii) Based on TCLP test of GBFS, FA and POFA materials it was concluded they had minimal toxicity due to leaching, indicating the environmental friendliness of these wastes for making geopolymer binders with improved efficiency and reduced production costs.

iii) For curing at ambient temperature, activation of binders through NHNS solution was found to be more suitable. The early strength of GPMs was affected more strongly via the activation of NHNS solution than NS solution. However, activation through NS solution revealed stronger influence on the strength of GPMs specimens tested after 28 days and cured at ambient temperature than other solution.

iv) At oven curing condition, mortars activated using NS mixture revealed higher CS at 60 °C compared to NHNS and NH solution activation.

v) Increase in the FA and POFA contents in the geopolymer network was discerned to reduce the early CS and enhance the strength at 28 days of age.

vi) In short, the achieved GPMs with improved strength and porosity were demonstrated to be prospective for sustainable development.

Acknowledgements

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