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Strength and microstructure of alkali-activated binary blended binder containing palm oil fuel ash and ground blast-furnace slag



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HIGHLIGHTS

• Good synergy was observed between ultrafine POFA and GBFS pastes at 80/20 proportion.

• C-S-H and C-A-S-H were identified to be present in the reaction product but not hydrotalcites.

• GBFS contributes to the soluble Ca, amorphousity and formation of CA(A)-SAH products.

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ABSTRACT

In this study, combinations of ultrafine palm oil fuel ash (UPOFA) and ground blast furnace slag (GBFS) have been used to study the compressive strength and microstructure of the alkali-activated GBFS–UPOFA (AAGU) binary blended binder paste. GBFS (G) was added in varying percentages to alkaline activated UPOFA (AAU) such that the ratio of G/U + G varied from 0 to 0.3 at interval of 0.05. Sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH) with a final silicate modulus (Ms = SiO₂/Na₂O) of 1.58 were used as activators. Pozzolanic materials (G + U), activators, and free water were combined by weight ratios as: 1/0.35/0.02. A 28-day compressive strength of 44 MPa was achieved for G/U + G of 0.2 cured for 24 h at 60 °C. Finally, characterization was done using X-ray diffraction, SEM and FTIR and the analyses revealed that GBFS contributes significantly to the mechanical strength by pore filling effects, and the formation of additional calcium (aluminate) silicate hydrate (C–(A)–S–H).

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1. Introduction

Cement plants are reported to emit up to 1.5 billion tons of CO_2 into the atmosphere annually [1] and this will continue to rise at 6% annually from 1988 to 2015 [2]. In fact, the cement industry is responsible for 3.4% of the global anthropogenic CO_2 emission [3]. For every ton of cement manufactured, nearly one ton of CO_2 is produced [2]. The concerns of excessive utilization of energy, proliferation of CO_2 and durability challenges posed by Portland cement concrete have called for the development of an alternative technology. The promising exploratory alternative is the geopolymer or alkaline activation of aluminosilicate materials, such as agricultural wastes (POFA and rice husk ash (RHA)), industrial wastes (fly ash (FA), or pulverized fuel ash (PFA) and silica fume (SF)) and natural pozzolans or calcined clay minerals (metakaolin). Many researchers have studied alkaline activation of different raw materials, such as metakaolin (MK) [4–6], slag [7–10], FA [11–13] or a combination of two or more aluminosilicate materials to study the performance of the resulting alkaline activated binders [8,14–17].

Geopolymer is described as amorphous structure that resembles zeolites of three dimensional frameworks of SiO₄ and tetravalent AlO₄ interlinked by oxygen atom [18]. The Al atom becomes tetravalent due to acquisition of additional negative electrostatic charge from alkaline or alkaline earth metals (Na⁺, K⁺, Ca²⁺) in the network [19]. The replacement of Si by Al generates different type of aluminosilicate (polysialate) products, such as polysialate (-Si-Al-O-), poly-sialate-siloxo (-Si-O-Al-O-Si-O-) and poly-sialate-disoloxo (-Si-O-Al-O-Si-O-) depending on SiO₂/Al₂O₃ ratio. The polysialate empirical formula is given as follows [19]:

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$$M_n$$
-{(SiO₂)z-AlO₂} $n \cdot wH_2O$

where *n* is the degree of polymerization and *z* is 1, 2, 3 and *w* is the water molecule while *M* is alkaline or alkaline earth metals which plays the charge balancing roles in the formation [20,21].

According to Palomo et al. [22] the process of alkaline activation is defined as chemical process that transforms the vitreous or amorphous raw materials into well compacted cementitious composite matrix. They [22] established two models of alkali-activated binding systems. The first one is the case of alkali activation of **Si + Ca** systems, such as GBFS with mild alkaline to form calcium silicate hydrate (C—S—H) as main product and the second model is alkaline activation of **Si + Al** system, such as fly ash and metakaolin which requires a medium to strong alkaline to form sodium aluminosilicate hydrate (NASH) as the main product [23,24].

To derive the economic benefits of this technology, there is need to explore the locally available materials especially the materials that contribute to the volume of solid wastes such as POFA and ground blast furnace slag (GBFS). Electricity are generated in palm oil mill by burning the palm empty fruit bunches, palm fibers and palm kernel shells as fuel thereby generating ash [25] while slag is produced in the steel industry as the aftermath of purification of iron from its ore by adding coke and limestones as the slagging agents to remove the impurities. Some studies have been conducted on the alkali activated slag (AAS) but to the best of our knowledge very few studies have been conducted on the potency of alkali activated POFA in binary blended form with slag or in ternary blended form with other materials despite the fact that it is a pozzolanic material according to ASTM C 618 [26].

Some of the advantages of blending slag with aluminosilicate materials have been reported. To this end, Bernal et al. [15] have reported increase in strength in high SiO₂ content MK/slag geopolymer system in comparison with unblended MK. In addition to that Puligilla [27] reported a reduction in the setting time when the slag was blended with fly ash in a geopolymer system. Kumar et al. [28] asserted the possible dual formation of C—S—H and aluminosilicate hydrate (A—S—H) when fly ash was blended with slag. The formation of C—S—H gel together with the geopolymeric gel occurs distinctly only in a system of low alkalinity while at higher alkalinity level (NaOH >7.5 M), geopolymer gel dominates [20].

Therefore, exploring the alkaline activation of POFA-slag binary blend will not only bring environmental benefits but also increase the utilization of these materials in building construction.

2. Materials and methods

2.1. Materials

2.1.1. Pozzolanic materials (UPOFA and GBFS)

POFA and GBFS were obtained locally in Malaysia. POFA was treated to achieve UPOFA following the procedure reported in the literatures [29,30]. Slag was ball milled for 16 h after sieving through 300 μ m. The GBFS used was alkaline type [31] which has coexistence of two phases: Ca²⁺ and Si⁴⁺.

The particle sizes were determined *using Turbotrac* S360 particle size analyzer (PSA). The specific surface area of UPOFA and GBFS determined using *Micromeritics ASAP2020* BET by nitrogen gas adsorption is shown in Table 1. X-ray diffraction (XRD) was used to determine the level of amorphousity or crystalline nature of the base materials under study.

Oxides composition of UPOFA and GBFS were analyzed using X-ray fluorescence (XRF) and the results are shown in Table 2.

Table 1

Physical properties of UPOFA and GBFS.

Material	Specific gravity	Average particle size, d ₅₀ (µm)	Specific surface area (m²/g)
UPOFA	2.6	1.069	13.40
GBFS	2.9	1.098	14.92

2.1.2. Synthesis of alkaline activators (AAs)

Commercially available sodium hydroxide (NaOH) pellets of 99% purity and aqueous sodium silicate (Na₂SiO_{3aq}) of initial silica modulus (Ms = SiO₂/Na₂O) of 3.3 were used as alkaline activators. The chemical compositions of Na₂SiO_{3aq} are as follows: H₂O: 62.11%, SiO₂: 29.13% and Na₂O: 8.76%. Distilled water was used to prepare the aqueous NaOH solution.

A solution of 10 M of Sodium hydroxide (NaOH) was made by dissolving, 404.04 g pellets (99% purity) in 1 liter of NaOH solution. The reaction is exothermic; hence, the solution was allowed to cool down before use. The prepared NaOH solution was added to Na₂SiO₃ to achieve a silica modulus (Ms = SiO₂/Na₂O) of 1.59 such that the mass ratio of Na₂SiO_{3(aq}/10 M NaOH_{aq} was 2.5.

Eqs. (2)–(5) show the decompositions and percentage constituents of the AAs. Total solids comprised the Na₂O, SiO₂ and PMs. From Eq. (5), a given volume of 10 NaOH prepared contained 24.1 wt.% of Na₂O, negligible 6.79 wt.% of chemically bonded water and 68.28 wt.% of distilled water. The total water was composed of added free water content (2 wt.% of PMs) and water contained in the combined AAs (CAAs) as shown in Eqs. (4) and (5). The total oxides compositions were expressed as a percentage of CAAs as shown in Table 3. The total molar ratio of H₂O/Na₂O and SiO₂/Na₂O were 18.92 and 1.64, respectively while H₂O/solids wt. ratio was 0.22.

$$Na_2SiO_3(aq) \rightarrow Na_2O + SiO_2 + H_2O$$
 (2)

$$10\text{NaOH}_{(aq)} \rightarrow (5\text{Na}_2\text{O} + 5\text{H}_2\text{O})_{\text{pellets}} + \text{H}_2\text{O}_{\text{dist}}$$
(3)

 $38.89\% Na_2 SiO_3 + 62.11\% H_2 O \rightarrow 8.76\% Na_2 O + 29.13\% SiO_2 + 62.11\% H_2 O_{dist} \eqno(4)$

$$\begin{array}{l} (30.80\%) 10 \text{NaOH}_{\text{pellets}} + (68.28\%) \text{H}_2\text{O}_{\text{dist}} \rightarrow (24.01\%) 5 \text{Na}_2\text{O} + (6.79\%) 5 \text{H}_2\text{O}_{\text{bonded}} \\ + (68.28\%) \text{H}_2\text{O}_{\text{dist}} \end{array} \tag{5}$$

2.2. Test details

2.2.1. Specimens preparation for compressive strength

Pozzolanic materials, GBFS (G) and UPOFA (U) were proportioned such that the G/U + G ratios were 0, 0.05, 0.1, 0.15, 0.2, 0.25 and 0.3. The samples were described as AAGU_x where x is the G/U + G ratios. The weight ratio of CAAs/PMs and free water content (FWC/PMs) were 0.35 and 0.02, respectively.

2.2.2. Specimens mixing, placing and curing

The mixing of the sample mixture was done by adding the constituent materials in batches using a 4.73 L capacity planetary bench mixer. The pozzolanic materials were followed by free water, NaOH_{aq} and Na₂SiO_{3aq} sequentially. Each component was mixed for an average period of 2 min and an additional 2 min was allowed for the mixtures to enhance homogeneity.

The mixtures were cast in oil smeared steel moulds of $50 \times 50 \times 50 \text{ mm}^3$ in two layers and then vibrated for 15 s each. The samples were then covered with vinyl sheet after placing in the mould to prevent loss of moisture. All the specimens were delayed for 12 h rest period at ambient temperature (25 °C) before being demoulded. The demoulded samples were kept inside vinyl bags and then placed in the oven at 60 °C for 24 h. The compressive strength of the specimens was determined after 3, 14, 28 days utilizing universal crushing machine at a loading rate of 0.9 kN/s, while the average failure load of three specimens is reported.

2.2.3. Microstructural characterization of the specimens

In order to characterize the microstructure of the products and to determine the contribution of GBFS to the product formed, AAGU_{0.2} was compared with AAGU₀ as a control specimen. The comparison was done by observing the bond vibrations of the molecules, the morphology and the elements present in the products using Fourier transform spectroscopy (FTIR), scanning electron microscopy coupled with energy dispersive X-ray spectroscopic analysis (SEM + EDS), and X-ray diffractometer respectively.

The fragmented binder surface of the specimens tested for compressive strength was used for SEM micrographs and the EDS of the specific areas within the microstructure was then obtained. The pulverized or finely divided paste samples were used for XRD and FTIR measurement. XRD measurement was conducted using Cu Ka radiation (40 kV, 40 mA) through continuous scanning within the angle 2-tetha range of $4-80^{\circ}$ and at a scan speed of 2.5° /min. FTIR was determined using KBr pellet technique.

3. Results and discussion

3.1. Particle size and physical properties

Table 1 indicates that both materials used were less than 1.1 μ m. The average particle sizes, d_{50} , obtained from the PSA, which is equivalent to 50th percentile of particle distributions for both UPOFA and GBFS, were 1.069 and 1.098 μ m, respectively.

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