



Characterization of mechanical and microstructural properties of palm oil fuel ash geopolymer cement paste



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HIGHLIGHTS

- POFA was geopolymerized effectively by alkali activators.
- Qualitative observations confirmed POFA viability to be used as a geopolymer binder.
- Compressive strength achieved by POFA geopolymerization is comparable to OPC paste.

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ABSTRACT

This study delineates activation of palm oil fuel ash (POFA) by a combination of sodium silicate and sodium hydroxide at 60 °C to be used as a geopolymer binder. Qualitative observations as well as compressive strength were recorded to assess the viability of POFA utilization. Also, XRD, SEM/EDX, DSC, FTIR tests were conducted to investigate underlying mechanisms of geopolymerization. The post-test observations revealed that activation of POFA is applicable and compressive strength of up to 32.48 MPa at the age of 28 days was achieved. Chemical tests indicated that formation of calcium silicate hydrate was the dominant cause of geopolymerization.

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1. Introduction

One potential replacement for cement is alkali activated materials (geopolymer binders) which seem to yield similar mechanical properties as Portland cement [1] although these materials are still at the beginning stages of development [2]. According to a recent rigorous and useful definition by Provis [3] “Alkali activated materials are produced through the reaction of an aluminosilicate—normally supplied in powder form as an industrial by-product or other inexpensive material—with an alkaline activator, which is usually a concentrated aqueous solution of alkali hydroxide, silicate, carbonate or sulfate.” Five groups for alkali-activated cements are categorized by Shi et al. [4]: Alkali-activated slag-based cements, alkali-activated pozzolan cements, alkali-activated lime-pozzolan/slag cements, alkali-activated calcium aluminate blended cement, and alkali-activated Portland blended cement (hybrid cements).

So far, different types of aluminosilicate materials such as Metakaolin [5–9], fly ash [10–12], and slag [13–15] were applied as alkali-activated cements. Palm oil fuel ash, an agro-waste produced in massive amounts in Malaysia, is another potential alkali-activated cement which is categorized as a pozzolanic material [16–19]. POFA has been lately used in binary mixes with other aluminosilicate materials such as ground granulated blast furnace slag (GGBS), rice husk ash (RHA) and fly ash in order to produce geopolymer concrete [20,21].

In general, alkali activation process was first introduced by Kuhl in 1908 and later the geopolymer terminology was proposed by Davidovits [3]. It was reported that poly-condensation of hydrolyzed aluminate and silicate was the main reason for hardening of the geopolymer binder in the form of zeolitic crystalline structure [22,23]. However, co-existence of geopolymeric gel and calcium silicate hydrate was also reported in previous works [24,25]. Formation of C–S–H was mainly regarded to dissolved calcium species from surface of the source with presence of available silicate species in alkali ambient [24]. It was also proposed that the simultaneous formation of geopolymeric gel and C–S–H may help to bridge the gaps in the matrix and hence increase the compressive strength especially at early ages [24].

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One of governing factors on properties of the gel and its formation is type and dosage of alkali activators [26]. So far, liquid sodium silicate (water glass) (Na_2SiO_3) and liquid sodium hydroxide (NaOH) were the most used activating solutions in geopolymers [27]. According to Komnitsas and Zaharaki [28] alkali hydroxide is required for dissolution of aluminosilicate sources while water glass solution acts as a binder, alkali activator and dispersant or plasticizer. However, because of the soluble silicate available in the liquid water glass, it is a preferred activating solution, which tends to increase the rate of the polymerization reaction [29]. Alkaline solutions induce a certain amount of Si and Al atoms to dissolve the aluminosilicate sources, forming monomers in solutions, and then poly-condense to form a rigid framework [30].

Previous works have shown that solid to liquid (S/L) ratio and Na_2SiO_3 to NaOH ratio (SS/SH) have tremendous effect on mechanical properties of geopolymer binders [31]. Xu and Van Deventer [32] reported that the solid to liquid ratio by mass should be around 3.0 for activation of fly ash in order to allow the geopolymerization process to take place. Nonetheless, the shape of particles has an effect on the required quantity of the alkali activator to result in advanced dissolution. Fly ash has better workability than Metakaolin in alkali activation process because of the spherical shape of its particle which reduces the demand for liquid as compared to the plate-like structure of Metakaolin particles. As such, Kong et al. [33] suggested a very low ratio of solid to liquid (0.8) by mass in order to activate the Metakaolin with optimum strength. In a few studies the effect of sodium silicate to sodium hydroxide ratio was also investigated. Hadjito and Rangan [34] reported that the combination of the alkaline activator ratio had a clear effect on the compressive strength of low-calcium fly ash based in geopolymer concrete. The findings of their research showed an increase in the compressive strength of the geopolymer concrete by 28% with an increase in the ratio of sodium silicate to sodium hydroxide. The recommended ratio by mass was 2.5, while Wang et al. [35] reported a lower ratio of 0.24 for Metakaolin based geopolymer.

One of the available aluminosilicate materials which can be widely found in Malaysia is palm oil fuel ash (POFA). Palm oil fuel ash is a by-product from the palm oil industry produced in massive amounts (approximately 4 million tons per year) [36]. So far, POFA is used as a partial replacement for ordinary Portland cement in conventional concrete for the purpose to enhance strength and durability of concrete [37–43]. Recently, some works have utilized POFA as a supplementary material in mixes with other aluminosilicate materials to make geopolymer cement paste or mortars. For example, low calcium fly ash blended with POFA [20] was used in order to produce geopolymer cement and so compressive strength of up to 28 MPa was obtained. Also, a geopolymer binder was fabricated from a ternary mix of slag, palm oil fuel ash and rice husk ash; however, a low content of POFA was used in the binder [21]. In another study by Mijrash et al. [44], treated POFA was activated to produce the geopolymer binder. Supplementary materials such as silica fume, calcium hydroxide and alumina hydroxide in addition to the alkaline activator were also used to increase the efficiency of treated POFA in the production of geopolymer products [44]. In other studies [45–47], a combination of ultrafine palm oil fuel ash and ground blast furnace slag was used to investigate the compressive strength and microstructure of geopolymer binder. Strength development for a geopolymeric binder from ground granulated blast furnace slag and palm oil fuel ash was also investigated by Islam et al. [48]. The study revealed that the binder with the binary mix of low content of POFA and GGBS achieved the highest compressive strength. In aforementioned studies sodium silicate and sodium hydroxide were used as alkali activators.

Still, no study investigated the activation mechanism and the microstructure of the resulted geopolymer from the activation of

POFA as an only aluminosilicate material source. The overarching purpose of this study is to activate POFA albeit its very low content of aluminum. The objectives include studying the process of alkali activation of POFA to produce a geopolymer binder, identifying the best ratio of solid to liquid, and sodium silicate to sodium hydroxide. The findings of this research will also explain the mechanism by which aluminosilicates with low aluminum content are involved in the geopolymerization. This study may encourage and promote further research on the use of POFA in geopolymer technology in mortar and concrete as well as the use of other aluminosilicate materials with low aluminum content which will ultimately lead to development of more environmentally friendly products with low energy consumption and very low CO_2 emissions.

2. Experimental method

2.1. Materials

2.1.1. Palm oil fuel ash

The palm oil fuel ash (POFA), obtained from burning of palm oil shells, husk and fibers, was collected from a mill at Johor State, south of Malaysia. The raw palm oil fuel ash was oven dried at $110 \pm 5^\circ\text{C}$ for 24 h, sieved with a $300\ \mu\text{m}$ sieve to remove large unwanted particles and incompletely combusted materials [49,50], and then it was ground by a modified Los Angeles machine [42]. The specific surface area after grinding was $0.915\ \text{m}^2/\text{g}$. The chemical composition of the POFA by XRF test is shown in Table 1. As can be seen, major components are SiO_2 and CaO with concentrations of 47.37% and 11.83%, respectively with a low amount of Al_2O_3 (3.53%). The relatively high amount of CaO available in the POFA is most likely from lime and fertilizer [41]. It is worth to mention that POFA is a not toxic waste material in terms of heavy metals leachability [51].

The XRD patterns of ground POFA are shown in Fig. 1. As can be seen, major phases of alpha quartz (SiO_2) and cristobalite (SiO_2) were traced [49,50]. The location of the highest hump was also detected in the XRD profile from 20° to 40° (2 theta), representing an amorphous phase [50]. The particle morphology of the raw POFA was investigated by scanning electron microscope SEM (Fig. 2). Fig. 2 indicates that raw POFA consisted of very irregular shaped particles with porous cellular surfaces [37,40]. The grinding process was effective to turn the POFA to a smaller sized and more homogeneous powder. The shape of particles was in crushed form and spherical with rough surface as can be seen from Fig. 3 [52].

2.1.2. Alkaline activators

Sodium hydroxide and sodium silicate were chosen as alkali activators in this investigation. The sodium hydroxide was in industrial-grade with minimum 99% purity. Industrial grade water glass (Na_2SiO_3) solution was chosen with a chemical composition of 15.33% Na_2O , 31.28% SiO_2 , and 53% H_2O . The alkaline activator selection was based on the recommendations in [34,53–55]. The alkali activation solution was prepared by mixing Na_2SiO_3 with NaOH within ratios ranged between 0.5 and 3.0 [34].

2.2. Preparation of POFA geopolymer paste

Preliminary experiments were conducted to study the alkali activation of POFA [56]. It revealed that raw POFA cannot be used without sieving and grinding because of the low compressive strength results which is related to the porous structure and high demands for the alkaline activator. Experimental program was designed in order to investigate the ability of POFA to be incorporated in geopolymer technology. NaOH solution was mixed with Na_2SiO_3 to produce six ratios (0.5, 1.0, 1.5, 2.0, 2.5, and 3) to prepare alkaline activator solution 24 h prior to use. Ground POFA and the alkaline activator were then mixed with two solid-to-liquid ratios (1.0, 1.32) as in [34,53,57,58]. Two groups of twelve mixes were prepared based on the solid to liquid ratio as shown in Table 2. Mix one with sodium silicate to sodium hydroxide ratio (0.5) was unable to be used; the geopolymer paste had such low workability that could not be cast in molds.

The palm oil fuel ash was mixed directly with the alkaline activator. First the ash was discharged to the mixer pan, and then the alkaline was added and mixed for 1.0 min at a normal speed rate (gear one). Then, the mixer stopped for 10–20 s in order to scrap the un-mixed ash on the sides of the paddle and the

Table 1
Chemical composition of POFA by XRF test.

Oxide	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	P_2O_5	SO_3	L.O.I
Concentration %	47.37	3.53	11.83	6.19	4.19	3.31	1.22	1.84

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