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Impact of added water and superplasticizer on early compressive strength of selected mixtures of palm oil fuel ash-based engineered geopolymer composites

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HIGHLIGHTS

- Engineered geopolymer composites (EGC) was developed using 100% POFA.
- H_2O and naphthalene-based superplasticizer effect on POFA-EGC strength was studied.
- POFA-EGC performed better with added water than naphthalene-based superplasticizer.

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ABSTRACT

This paper investigates and presents a study on the comparative effects of added water and naphthalene-based superplasticizer (SP) on the compressive strength and microstructure of the developed palm oil fuel ash engineered geopolymer composites (POFA-EGC). Three differently prepared 50 mm × 50 mm × 50 mm specimens (with 10% water, with 10% SP and with 5% SP & 5% water) were used to study the synthesis of POFA and alkali-activating solutions (8 M $NaOH_{(aq)}$ + Na_2SiO_3 [Ms = 3.3]). 2% volume fraction of PVA fibres were added to engineer the cementitious composite mixture. All the specimens were cured in an oven for 24 h at 60 ± 5 °C to accelerate the geopolymer reaction process to generate the binder. After testing, the obtained results showed that while specimens with added water only gave the compressive strength of 29.4 MPa, there was a 19% decrease in compressive strength for samples with SP only at 28 days. The specimens with water and SP combined have the least strength. Microstructural examinations (SEM) and material characterization (XRD, FTIR) of the alkali-activated composite also revealed the superior performance of the specimens with water over SP. The significant finding of this research work is the better performance of POFA-EGC prepared with only water in both fresh and hardened states. It is recommended that water be given precedence over SP in the development of POFA-EGC.

1. Introduction

Recently, the environmental impact of Portland cement (PC) production has reached its record peak and still growing. Another class of binder produced from the reaction between aluminosilicate precursor and high alkaline solution has been developed to mitigate the environmental impact of PC. This new cementitious binder is referred to as the geopolymer binder. Geopolymer binder is a member of the inorganic polymer family, which has a microstructural semblance with zeolites. Unlike zeolite with a crystalline microstructure, it has an amorphous one. In addition, in contrast to PC concrete where water is chemically involved in the hydration reaction, water plays no part in the geopolymer chemical reactions and thus expelled. However physically, this water provides the needed fluidity for easy handling and placement. Geopolymer binder comes with many advantages, chiefly of which are reduction of greenhouse gases and improved efficiency in its utilization for geopolymer concrete. However, vibrator is employed in its production for proper compaction due

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to the problem of high resistance to flow because of the source materials. High water demand has been reported with some materials used in the development of geopolymer concrete [1] in order to achieve the needed workability of the mixture. The use of large amount of POFA in concrete for instance has been reported to give lower slump and lower degree of compaction [2]. Megat Johari et al. [3] observed that by reducing the amount of unburnt carbon in the ultrafine POFA, slump higher than that of OPC high strength concrete was achieved. This is because the presence of more paste will give more lubricating effect to the constituents of the mixture, hence improved mobility.

Engineered geopolymer composites (EGC), the geopolymer version of normal engineered cementitious composite (ECC) have joined the ranks of new cementitious binder. EGC is a member of the fibre-reinforced cementitious composite family where short fibres are used to improve the tensile capacity of the brittle geopolymer concrete. The presence of fibres in the fresh geopolymer mixture increases the resistance previously offered by the precursor material to flow. PVA fibres have been reported by Yew et al. [1] and Shaikh [2] to adversely affect the workability of fibre-reinforced geopolymer composites. Low workability leads to a non-uniform dispersion of PVA fibres in fresh EGC mixture, which reduces the effectiveness of the fibres [3,4]. The increase in the quantity of POFA in the EGC mixture leads to a decrease in workability [6,7]. This is attributed to the highly porous nature [8] and fineness [2] of the POFA particles, hence increasing its high water absorption characteristics. Flowability properties of the mixture can be improved by careful and controlled addition of either water or superplasticizer (SP) to the composite mixture. It has been established by previous researchers [5–7] that fresh and hardened properties of concrete or mortar is greatly affected by the amount of water added to the mixture. In a study on the effect of SP on properties of high volume POFA on concrete, Awal and Hussin [9] reported a satisfactory workability performance but recorded a lower strength development compared to OPC concrete in all the ages. For effectiveness of fresh EGC, a high slump mixture is imperative to the resistance offered from precursor material and PVA fibres. Water and SP play important role in achieving the needed fluidity but there is a need to balance economy and performance of the developed geopolymer mortar.

In the present study, the resistance to flow is offered by two important materials, 100% POFA (the only source material in this study) and PVA fibres. Hence, there is need for a more plastic mixture of a stiffened POFA-EGC mixture because of high surface area of the base material (POFA) used and the presence of a hydrophilic material (PVA fibre) to achieve compaction. In many of the research works, water and SP [10–13] were combined in geopolymer mixture; little or no literature have addressed the comparative performances of water and SP in studying the efficacy of each in the mixture, hence there is a need to fill this knowledge gap.

This research study was intended to explore the feasibility of producing EGC made with POFA by examining its basic physical and mechanical properties. The experimental part of the work studies the effects of extra water and superplasticizer on the workability and compressive strength of POFA-based EGC.

2. Experimental investigations

2.1. Materials

2.1.1. Palm oil fuel ash

POFA was collected from a nearby palm oil mill, United Oil Palm Industries Sdn. Bhd. in Nibong Tebal, Penang, Malaysia. The raw POFA was dried in an oven at 100 ± 5 °C to remove its moisture and then sieved using a set of sieves (600 and 300 μm) in order to exclude coarser and unwanted materials. The ash obtained after sieving was ground by a mechanical ball mill containing of 150 steel balls of different sizes (6 mm to 32 mm) and rotating at speed of 180 rpm. This was done to reduce the POFA particle size and to increase its surface area, which ultimately led to improved reactivity. In order to remove unburned carbon and prevent glassy phase crystallization, in addition to agglomeration of particles, the POFA was calcined at 550 °C in a gas-powered furnace for 90 min. To further improve the surface area, the calcined POFA underwent another round of grinding in the ball mill. The loss on ignition (LOI) values after heat treatment is 2.3%. The reduction in the LOI value is compensated for by the increase in mass percentages of other oxide components. With total oxides of Silicon, Aluminium and Iron of 78.07%, it complies with the specification of ASTM C618 [14] class F. The particle size distribution of the final stage of preparation of POFA was determined using *Turbotrac S360* particle size analyser (PSA). The surface areas were determined using *Micromeritics ASAP2020 BET* using nitrogen gas adsorption. Table 1 shows the oxide compositions of POFA, which were determined using X-ray fluorescence (XRF) technique.

2.1.2. Aggregates

The fine aggregate used in the study is dune sand with fineness modulus of 1.85 and specific gravity in the saturated and surface dry (SSD) condition of 2.62.

2.1.3. Synthesis of alkaline activators

Commercially available $Na_2SiO_{3(aq)}$ with an initial silica modulus ($Ms = SiO_2/Na_2O$) of 3.3 and 8 M $NaOH_{(aq)}$ were used as alkaline activators (AA).

2.1.4. Superplasticizer

A commercially available sulphonated naphthalene polymers superplasticizer (SP), satisfying the ASTM C494 [15] Type F was used in this study to modify the workability and to achieve adequate rheological properties in the fresh geopolymer mortar for appropriate fibre dispersion. It is a chloride free superplasticizing admixture.

2.1.5. Polyvinyl alcohol fibre

Polyvinyl alcohol (PVA) fibres, "REC15" was used in this study with data provided by the manufacturer as shown in Table 2. The surface of the PVA fibres is coated with a proprietary hydrophobic oiling agent of 1.2% by weight to control the interfacial bonding properties between the fibre and matrix for composite performance [16].

2.2. Experimental design

2.2.1. Mixture constituents and proportion for POFA-EGC

An approximate unit weight of between 2300–2400 kg/m^3 for the POFA-EGC is quite similar to that of typical Portland cement concrete. The mortar was prepared with 100% POFA as the binder, a ratio of Sand/POFA is 1.8 and the percentage of PVA fibre used is 2% volume fraction. The total silica modulus ($Ms = SiO_2/Na_2O$) was obtained from activator's relative proportion – ($Na_2SiO_{3(aq)}$/8 M $NaOH_{(aq)}$) such that the ratio is 2.5:1. The SP and water was added such that (SP/Water) to POFA ratio was maintained at 10 wt.%. Table 3 shows the proportions of the constituent materials in the POFA-EGC mixtures.

2.2.2. Sample preparation and curing

The POFA and the sand were mixed together thoroughly in dry condition using a *Hobart* planetary bench mixer model *N50-60* to get a uniform mixture of dry materials. Subsequently, the alkaline activators (mixture of $Na_2SiO_{3(aq)}$ and $NaOH_{(aq)}$) were added to the dry materials, mixed for five mins and then followed by the addition of water and SP, after which mixing was continued for another 4–5 min. After making sure there are no solid materials left sticking to the base of the bowl, the PVA fibres were gradually spread into the fresh mortar matrix by hands as the mixture is mixed at slow speed until all fibres were evenly distributed. The fibres were added slowly to ensure proper dispersion with no agglomeration. This mixing procedure was considered by many authors [17–19] as required to attain good compressive strength. The overall mixing time was 14 min and the flowability of the

Table 1
Chemical compositions and physical properties of POFA.

Chemical composition (mass%)	POFA
Silicon dioxide (SiO_2)	66.91
Aluminium oxide (Al_2O_3)	6.44
Ferric oxide (Fe_2O_3)	5.72
Calcium oxide (CaO)	5.56
Magnesium oxide (MgO)	3.13
Sodium oxide (Na_2O)	0.19
Potassium oxide (K_2O)	5.20
Sulphur oxide (SO_3)	0.33
LOI	2.3
Physical properties	
Specific gravity (g/cm^3)	2.53
Median particle size d_{50} (μm)	1.068
Specific surface area (m^2/g)	1.521