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Compressive strength and microstructural analysis of fly ash/palm oil fuel ash based geopolymer mortar under elevated temperatures

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HIGHLIGHTS

- POFA/FA based geopolymer mortar gain strength by subjecting to elevated temperature up to 500 °C.
- Increasing of POFA content expedite the start of micro-pores formation due to high temperatures.
- Increasing in POFA content delays the temperature of maximum strength.

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ABSTRACT

This paper presents the effects of exposing palm oil fuel ash (POFA)/fly ash (FA) based geopolymer mortar to elevated temperatures at early stage in terms of microstructural and compressive strength. Combination of sodium silicate and sodium hydroxide is used as activator. The development of compressive strength of POFA/FA based geopolymers mortar was investigated using XRF, XRD, TGA/DTG, and FESEM. It was observed that replacement of the 0–100% of POFA in FA based geopolymer mortar expedite the start of micro-pore formation due to exposure to high temperatures and shift the strength peak from 300 °C to 500 °C.

1. Introduction

Although ordinary Portland cement has adequate strength to resist fire in normal cases and can even be used as fire resistant cover for steel constructional elements, at elevated temperature it shows physical and chemical changes followed by deterioration in mechanical properties of the material [1,2]. One of the products of ordinary Portland cement hydration is calcium hydroxide which will decompose into calcium oxide and water at about 400 °C. Above 400 °C, loss in total strength is due to dehydration of calcium hydroxide and rehydration of calcium oxide [3]. Therefore, seeking a new constructional material which has improved properties at elevated temperature is certainly worth considering.

Geopolymer is a new constructional binder with a lower CO_2 footprint than traditional Portland cement. Geopolymer is an inorganic polymer based on aluminosilicates which is produced by synthesizing pozzolanic compound materials with highly alkaline hydroxide and/or alkaline silicate [4]. Industrial solid wastes and by-products containing silica and/or alumina, such as fly ash, can be applied as pozzolanic components for geopolymerization [5]. Geopolymers are environmental friendly and their production consumes a moderate amount of energy. In recent years, a lot of investigation has been undertaken to find the mechanical and environmental properties based on the utilization of different activators, pozzolanic material and methods of curing [6–11]. As energy efficient ceramic like materials, aluminosilicate geopolymers which harden at ambient temperature or above, have stable and durable high temperature characteristics, and can be applicable as fireproof insulating material have attracted considerable attention [12,13]. Geopolymers have been the subject of much research as a cementitious binder; however, the study of its performance at elevated temperature is still needed.

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POFA is a waste and byproduct of the palm oil industry which is one of the most important agro industries in South-East Asia and in the African Sub-Sahara region. Due to the burning of empty fruit bunches, fibers and shells as fuel to generate electricity, the waste, which is collected as ash, becomes POFA. It has been estimated that the total solid waste generated by the industry in Malaysia alone has amounted to about ten million tons a year [14]. About three million tons of POFA was produced in Malaysia in 2007 and hundred thousand tons of POFA is produced annually in Thailand, and this production rate is likely to increase as palm oil plantation areas have increased [15–17]. POFA disposal causes environmental pollution and a recent research on the use of silica rich POFA as cementitious material paves the way for the development of sustainable material.

Global annual ash production is about five hundred million tons, of which 75–80% is FA; environmental issues are due to disposal problems associated with FA which has led researchers to utilize it in the production of Portland cement. Therefore, many researchers have focused their attention on the use of FA as a source material for the development of cementless concrete, widely known as geopolymer concrete [18–20]. The consumption of POFA and FA as construction material causes a decrease in demand and production of conventional Portland cement, while POFA, due to its limited use, is currently disposed of in landfills which could lead to environmental problems [21].

Synthesis of geopolymer can be approximately partitioned into dissolution-hydrolysis and hydrolysis-polycondensation parts which might happen simultaneously. Release of aluminate and silicate monomers is followed by the formation of dissolved species that cross-link to form oligomers, which in turn produce sodium silicoaluminate [18]. In the next stage, the gel formation is simultaneous with setting and hardening which is attributed to polycondensation and the formation of a three dimensional aluminosilicate network. The Si/Al ratio is constant during geopolymerization and increased by passing time. The low SiO_2/Al_2O_3 of FA based geopolymer result in high initial strength. The geopolymers with lower Si content make it possible that more $Al(OH)_4^{-4}$ species were available for condensation in these materials at early stages. Further, the Al component tends to dissolve easier than the silicon components, and this enables a higher rate of condensation between silicate and aluminate species than the condensation between just silicate species, resulting in high initial compressive strength.

In this study, POFA was used to replace conventional binders in geopolymer concrete, namely FA in five different percentages. The POFA contents were varied at 0%, 25%, 50%, 75% and 100% to develop geopolymer mortar. The mortars were subjected to four elevated temperatures at 300 °C, 500 °C, 800 °C and 1000 °C as well as room temperature, as reference, to investigate the density, volume change, compressive strength and microstructural characteristics. In addition, the effect of ambient curing on the above said parameters was also investigated and reported. Analytical methods used to investigate the microstructure of the POFA/FA geopolymer mortar include X-ray diffraction (XRD), Thermogravimetric analysis (TGA) and field emission scanning electron microscopy (FESEM). Geopolymers in this study are produced by mixing class F FA and POFA as pozzolanic component, with alkali activated aluminosilicate binders based on sodium hydroxide and sodium silicate.

2. Experimental program

2.1. Material characterization

POFA was obtained from factories in the vicinity of Kuala Lumpur where palm fiber, shells and empty fruit bunches were combusted at a temperature of about 700–1000 °C. The raw POFA is not usable due to impurities of foreign materials and uncombusted palm fibers, large particle size and unknown moisture content. Oven heating which causes the evaporation of moisture followed by grinding in a Los Angeles abrasion machine for 30,000 cycles using a mild steel bar (12 mm diameter and 800 mm long) makes the POFA suitable for constructional usage. The batch of low calcium FA (class F) used in this research had been collected from Lafarge Malayan Cement Bhd, Malaysia. Fig. 1 shows both ashes. Particle size distributions were measured with Mastersizer Malvern Instruments and results are shown in Fig. 2. POFA and FA have a median particle size of 22.78 μm and 16.23 μm respectively. The physical properties of the materials are given in Table 1. The chemical composition of the materials as determined by X-ray florescence by PANalytical Axios mAX instrument and LOI value are provided in Table 2. SiO_2 to Al_2O_3 ratio is 1.97 and 17.16 for the Fly ash and POFA respectively. Local fine aggregates were found in oven dry condition with a maximum particle size of 1.19 mm. Alkaline activators in the investigation consisted of alkali silicate and hydroxide solutions. Sodium hydroxide (NaOH) was prepared in pellet form which had 99% purity while sodium silicate was used in liquid form as activators with about 1.5gr water per milliliter at 20 °C with a modulus ratio of 2.5 (SiO_2/Na_2O, SiO_2 = 30% and Na_2O = 12%).

2.2. Mixture design, specimen preparation and testing

The alkali activator solution was prepared by mixing 16 molar NaOH with Na_2SiO_3 solution with Na_2SiO_3 to NaOH ratio of 2.5. The activator to binder ratio was kept at 0.5 for all the mixes. The mixture proportions of mixes are given in Table 3. Dry FA, POFA and mining sand were mixed with the sand to binder ratio of 1.5 in a cake mixer for about 2 min at a low speed to blend the source materials and the sand uniformly. The sand binder ratio remained constant for all specimens. The alkali activator solution was then gradually added into the mix for about 7 min simultaneous with water to improve workability and homogeneity of the mortar. The mortar specimens were cast in 50 mm cube steel molds in three layers of equal height and compacted. The samples were vibrated to remove entrained air and bubbles. Immediately after vibration, all samples were covered and kept in the oven for hot curing for 24 h at a temperature of 65 °C; the specimens were then taken out of the oven and kept in ambient condition with an average temperature and humidity of 28 °C and 70%, respectively, until testing day.

Fig. 1. Fly ash (left side) and POFA (right side).

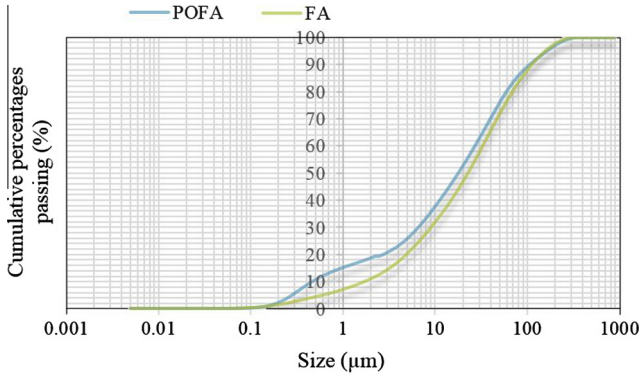

Fig. 2. Particle size distribution of the fly ash and POFA.