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Thermal stability and mechanic al properties of alkaline activated mortars synthesized from POFA after exposure to elevated temperatures

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Abstract: This paper presents results from experimental studies on the Thermal stability and mechanical properties of alkaline-activated mortars, which that prepared by alkaline solution activating POFA.The compressive strength test and characterization, employing XRD and, FESEM were conducted on geopolymer mortar specimens at ambient temperature and after exposure to elevated temperatures from 200℃ - 1000°C. Results from these tests show that geopolymer mortar exhibits higher temperature-induced degradation in compressive strength and body at1000°C. the mixture of POFA experienced no thermal cracks, its deformation occurred at 1000℃ which resulted in extensive shape changes. measure the compressive strength of the exposed alkaline-activated mortars. The relative residual compressive load for the mixture (a) decreased from 77% to 40% after exposure to temperatures from 200°C to 800°C

Keywords: POFA; Compressive strength ; Thermal stability ; XRD ; FESEM

Introduction

Geopolymer or alkaline activated is a type of new cement, and a new environment-friendly inorganic binder, obtained by alkaline solution activating aluminosilicate source material (such as POFA, fly ash, and slag), has attracted significant interest in recent years as a practical alternative to Portland cement [1–4]. Issues related with the utilization of conventional supplementary materials such as (FA), slag (GGBFS), (SF), etc. are due to restricted accessibility locally and higher transport costs imported from somewhere else [5-7]. Palm oil produces plenteous waste squander in Malaysia. The palm oil industry produces about 80% of squandered materials such as product fruit bunches and shells which make genuine issues for the environment [8]. This agricultural squander has been utilized as a substitute for fuel [9]. to turn on generators for electrical energy technology in palm oil mills. After the burning process, the closing residues are both dumped into the soil or left in open land. This waste fabric leads to environmental air pollution and contaminates groundwater [10]. and exchange the normal composition of the soil. Moreover, the addendum of waste as a supplementary material in concrete or mortar reduces the compressive power as a result of decreasing the concentration of portlandite due to the dilution effect [11 –13]. The effect of thermal stability on geopolymer mortar from properties of physical, chemical, and morphology change of minerals, and their influence on compressive strength development is a motivation for this study. some researchers incorporated fly ash, slag, and metakaolin in concretes at ratios of 10% and 20% in the form of either cement added or replaced without affecting the cement content and concrete samples to which they are exposed to 200, 400, 600, and 800 ℃ for 3

hours. The results indicated that FA concretes exhibited the highest relative strength at temperatures of 600 and 800℃. On the other hand, the neat MK system showed a much higher residual strength upon cooling from 1000°C to room temperature, indicating that the extent of glass formation from the geopolymer gel at 1000°C is reduced by the incorporation of Ca into the gel, which occurs as a consequence of the formation of C-S-H type gel that coexisted with the aluminosilicate geopolymer gel [14]. Ranjbar et al. (2014a [15] reported low thermal stability of mixtures of POFA/FA-based geopolymer mortar when this mixture is exposed to above 300 ℃. However, in the case of the increased content of fly ash, the results revealed an increase in the high-temperature thermal stability in strength rather than increasing in POFA content (decreased compressive strength) in the mixtures investigated. This was linked to the beginning of pore formation upon exposure to elevated temperature. This finding is basically due to the combustible compounds such as carbon in the untreated POFA which decomposed in the vicinity of 500°C. The objective of this research is to investigate the effect of thermal stability on physical properties, chemical composition, morphology, and their influences on the compressive strength development of geopolymer mortar. The resulting of geopolymer were extensively characterized, employing XRD and, FESEM

2. Materials and methods

2.1 Materials

2.1.1 Palm oil fuel ash

Palm oil fuel ash (POFA) was obtained from United Oil Palm Industry in the state of Penang, Malaysia. The first step involves drying the ashes within an oven for 24 h at 105°C to take out the POFA's inherent moisture since the POFA was placed outside the mill. POFA was thereafter sieved through 300 mesh to take out coarser particles of partially burnt ashes from fibers and palm kernel shells, following the work of Elbasir et al. (2017). Then the POFA was grounded into particles of size 10 μm by utilizing a ball mill fitted with 150 steel balls that rotate about 180 rpm for 8 hours. The unburnt carbon was then removed by heating the grounded POFA in a furnace at 550 °C for 90 minutes. The resulting POFA was specified as treated t-POFA. Fine POFA, designated as f-POFA, was fabricated by grounding the t-POFA for an additional 8 hours to enhance its fineness. Then, the f-POFA was grounded another 8 hours to obtain u-POFA (ultrafine POFA) with an average particle size of 1.1 μm.

2.1.2 Aggregates

Total fine aggregates content of 100 percent river sand along with properties of 1.85 fineness modulus as well as specific gravity 2.62 under saturated and surface dry (SSD) conditions. The ratio of sand to raw materials (FA, u-POFA, and GBFS) was maintained at 1.5.

2.1.3. Alkaline activator

10 M concentration NaOH and sodium silicate with initial silica modulus 2.2 (Ms = SiO2/Na2O) were utilized to synthesize the alkali activators for the purpose of mortar mixtures). This contains sodium silicate with sodium hydroxide 10 M.

2.2 Design and preparation

Delay time before heat application had no adverse effect on compressive strength development. In addition to this, a longer delay time is required to provide ample time during the manufacturing process. In this study, selected single binders fabricated from POFA mixtures for thermal stability analyses were chosen from the base material (POFA) (as shown in Table 1).

Table 1: Mix proportions for thermal stability

Type of Mix materials (kg) **m³**	Sand	Na_2SiO_3	NaOH	Water	Added water
	(kg)/ m^3	(kg) / m^3	(kg)/ m^3	(kg) / m^3	(kg)
POFA 856	1280	293	40	80	60

2.2.1 Test Procedure

To examine the significant effect of the single binder of (POFA) on thermal stability as shon fig (1),The alkali-activated mortar specimens from the mixture were exposed to various elevated temperatures. The testing procedure was the same as previous researchers [14]. Specimens were cured at ambient temperature for 28 days, prior to exposure at various elevated temperatures (from 200°C to 1000°C) in increments of 200°C at a heating rate of 10 °C/min. The specimens were placed inside the electrical furnace, as shown in Figure (1). As soon as the target temperature was attained, it was maintained for an additional 1 hour before the furnace was shut down to allow the specimens in the furnace to cool down to room temperature.

Fig 1: Samples placed inside the electrical furnace after heating

2.2.2 Specimen Analysis

The compressive strengths after cooling were determined according to ASTM C109/C109M (ASTM, 1999a) test [16]. The residual compressive strength was measured in triplicate samples. The behaviour of the hardened geopolymer mortar mixtures after exposure to different thermal loads was evaluated quantitatively by measuring the retained residual compressive strengths after the heat exposure. Detailed XRD and physical appearance analysis were undertaken to observe the effects of high temperatures on specimens exposed at 1000°C. From the XRD analysis, only the specimen with the highest thermal stability was investigated for their phases, physical, and microstructure changes (from ambient temperature to 1000°C) using FESEM. To further confirm the results of XRD analysis and microstructure changes.

3. Results and discussion

3.1 Visual observation

The main visual observation of the selected hardened mortars before and after exposure to elevated temperatures is shown in Figure (2). The specimens (a - d) before exposure to elevated temperatures reveal that at ambient temperature all mixtures have a dark grey colour. However, after being exposed to elevated temperatures from 200℃ - 1000°C, all mixtures changed from grey to light sand and light pink. Although mixture (a) contained u-POFA experienced no thermal cracks, its deformation occurred at 1000℃ which resulted in an extensive shape changes.

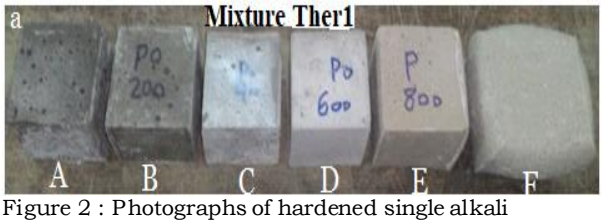

Figure 2 : Photographs of hardened single alkali activated mortars containing u-POFA Ther1, before and after being exposed to elevated temperature of (A) 28℃, (B) 200℃ (C) 400℃ (D) 600℃ (E) 800℃ (F)1000℃

3.2. Compressive strength (CS)

Figure 3. and Figure 4. show the residual compressive strength results of single alkaline-activated mortars containing u-POFA at 28 days (as the reference) and after being exposed to elevated temperatures ranging from 200°C to 1000°C with an increment of 200°C. Figures 3 and 4 reveal that the strength of all alkaline activated u-POFA-based mortar steadily decreased as temperature increased from 200°C to 1000°C. On the other hand, for the specimens of alkaline activated u-POFA mortar after being exposed to 1000°C, the deformation in the form of sharp swelling was indicated. As shown in sample F given in Figure 2 , there is a change in the specimen's size and shape,

which made it impossible to measure the compressive strength of the exposed alkaline activated mortars. The relative residual compressive load for the mixtures decreased from 77% to 40% after exposure to temperatures from 200°C to 800°C, respectively

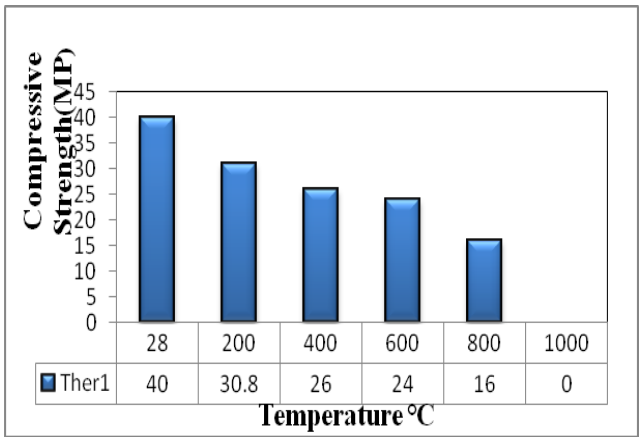

Fig 3: Residual compressive load of alkaline activated mortars

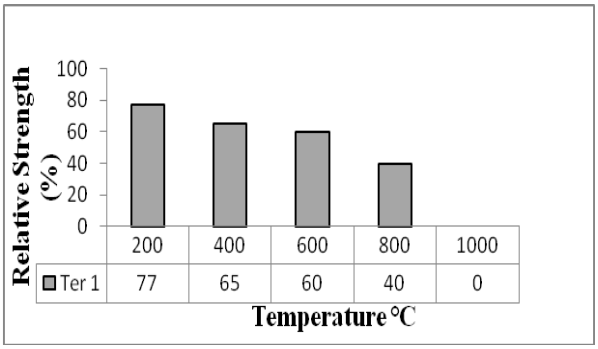

Fig 4: Relative residual compressive load alkaline activated mortars

3.3 Mineralogical analysis

X-ray diffraction tests were performed to identify the phases of the geopolymer mortar specimens.As shown in Figure 5, the XRD patterns have similar crystalline phases for mixture Ther1 made from u-POFA at 28 days and being exposed to elevated temperatures of 200℃ to 400℃. It should be noted that the calcite phases remained. After exposure to 600℃, no calcite phase was retained in the structure. While, the quartz and jadeite phases remained the same after exposure to 800℃, however, after exposure to 1000℃ no calcite phase was retained in the structure. The new phase was identified as nepheline with chemical formula NaAl(SiO4). Nepheline starts to form at high temperatures (800℃) from the decomposition of amorphous aluminosilicates [17]. The collapse of the tetrahedral framework at about 800°C, just before melting (about 1000°C), may explain the higher rate of expansion observed with nepheline).

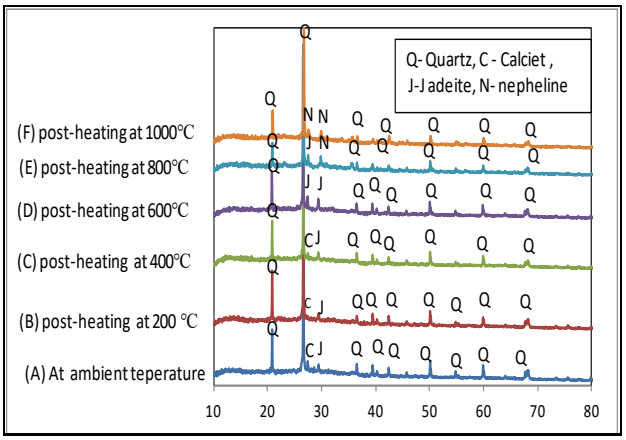

Fig 5: XRD diffractograms of u-POFA based alkali activated mortar before and after being exposed to elevated temperature of (A) 28 ℃ (B) 200 ℃ (C) 400 ℃ (D) 600 ℃ (E) 800 ℃ (F)1000 ℃

3.4 Field emission scanning electron microscopy analysis

The FESEM microscopy images of the alkali activated mortar mixtures which are prepare it from POFA waste before and after exposure to elevated temperature are shown in Fig 6. The specimens demonstrated the effects of high temperatures up to 1000℃. The FESEM micrographs (exposure temperatures from 200°C to 1000°C) for the specimens of POFA The selection of the mixture was based on physical appearance, relative residual compressive strength, and XRD analyses. The micrographs showed that when exposed to elevated temperatures from 200°C to 600°C

(Fig 6), the cracks observed on the surface were wide and larger. Similarly, the number of pores also increased. However, when heated at 800°C, a number of microcracks appeared besides the uniformly dispersed pores with similar diameters (Duxson et al., 2006). The microstructure of POFA samples before and after heating is characterized by the presence of unreacted materials in POFA samples embedded in the reaction product of geopolymerization of the POFA. As shown in Fig 5 , the results of XRD analysis of the reaction product indicate the formation of C–S–H gel with a low Ca content along with geopolymer-type gel (N–A–S–H), as observed previously by [18]. Heating from 600°C to 1000°C has formed some pores also decreased the compressive load significantly. Martin et al. (2015) [15] reported that it disappeared at 800°C, giving way to nepheline (NaAlSiO4).

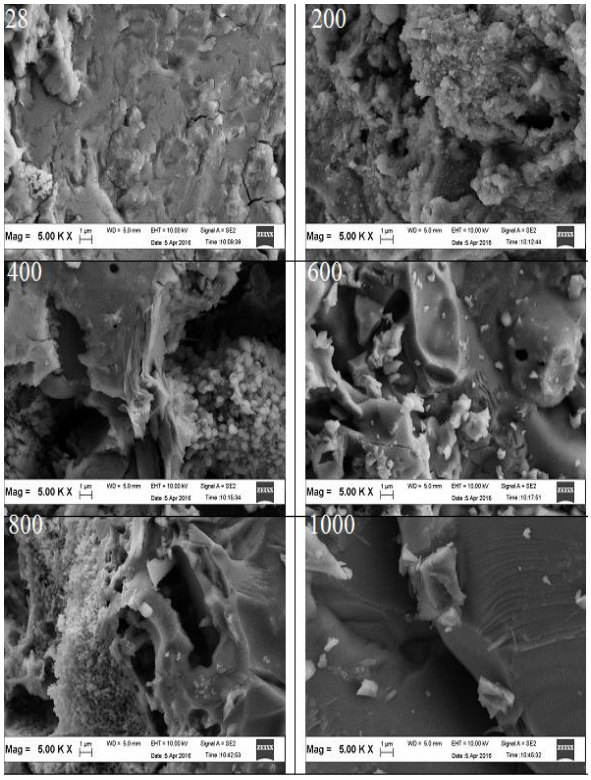

Figure 6: FESEM for mixture of u-POFA based alkali activated mortar before and after being exposed to elevated temperature of 200 C to 1000 C

Conclusion

In this research, the effect of Thermal stability and mechanical properties of alkaline-activated mortars, which that prepared from, Waste of palm oil full ashes. The change in the compressive
strength was also investigated. Based on the results, the following conclusion can be drawn;

1. The main finding of the strength of all alkaline activated POFA-based mortar steadily decreased as temperature increased from 200°C to 1000°C.
2. After being exposed to 1000°C, the deformation in the form of sharp swelling was indicated. As shown in sample F given in Figure 2 , there is a change in the specimen's size and shape, which made it impossible to measure the compressive strength of the exposed alkaline activated mortars.
3. The relative residual compressive load for the mixtures decreased from 77% to 40% after exposure to temperatures from 200°C to 800°

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