

# Effect of different curing temperatures on alkali activated palm oil fuel ash paste



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## HIGHLIGHTS

- This study showed that oven curing accelerate the geopolymerization.
- The threshold temperature was 70 °C for the alkali activation of POFA.
- Ambient temperature can be applied for curing of alkali activated POFA.

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## ABSTRACT

This study investigated effects of curing temperature on alkali activation of palm oil fuel ash. Compression test was conducted on samples activated with a mixture of sodium silicate and sodium hydroxide at different curing temperatures; ambient, 60 °C, 70 °C, and 80 °C. Also, XRD, TGA, FTIR, and SEM/EDX tests were performed to trace changes due to geopolymerization. Results showed that the ambient temperature can be applied to activate mixes with 100% POFA. Compressive strength of up to 36.8 MPa was obtained when samples cured at ambient temperature. The threshold temperature for activation of POFA was 70 °C.

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## 1. Introduction

Alkali-activated materials and geopolymer binders are scientific and technological developments being made at a rapid pace due to their low energy consumption and environmentally friendly qualities [1–3]. The alkali activated materials can be manufactured through reaction of aluminosilicate materials, mostly industrial wastes and by-products with an alkaline activator [4]. The alkaline activators are mainly concentrated H<sub>2</sub>O aqueous solutions of alkali hydroxide, silicate, carbonate or sulfate [5,6]. The Alkali activation process was first introduced by Kühl in 1908 [5,7] and later the term geopolymer was introduced by Davidovits [8,9].

So far, two major categories are defined to explain alkali activated materials: “low calcium alkali activated materials” namely fly ash class F and Metakaolin and “higher calcium alkali activated

materials” such as ground granulated blast slag (GGBS) and fly ash class C [5]. Type of resource material, its fineness, size, and type of activator are governing factors on geopolymerization process [10,11]. One of the key parameters is curing regime by which mechanical properties and durability of geopolymer binder are influenced [12,13].

Alkali-activated binders can be classified into two categories with respect to the final hardened gel; first one is represented by activation of slag with relatively high amount of Ca defined as higher calcium alkali activated materials. Provis [5] stated that the reaction product of the higher calcium binders such as GGBS and alkali metal silicates or hydroxide solutions is “aluminum substituted calcium silicate hydrate (C–A–S–H)”. This gel is similar to tobermorite in its structure and generally can be compared to the C–S–H gel resulted from the hydration of Portland cement.

Substitution of Al in the C–S–H structure leads to a higher degree of polymerization and crosslinking between tobermorite chains. However, in a recent work by Moslih et al. [14] it was revealed that in precursors with low alumina, the dominant formed gel is C–S–H.

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The second category includes the activation of Metakaolin and fly ash, defined as low calcium alkali activated materials [5,15]. The low calcium alkali activated materials are most commonly activated with sodium hydroxide or sodium silicates with relatively high pH. The formed geopolymeric gel has a calcium free structure which can be viewed as a disordered analogue of an aluminosilicate zeolitic structure [16]. However, co-existence of geopolymeric gel and calcium silicate hydrate was also reported in previous works [17,18]. Formation of C–S–H was mainly regarded to dissolved calcium from surface of the source with presence of available silicate species in alkali ambient [17]. It was also proposed that the simultaneous formation of the geopolymeric gel and the C–S–H may help to bridge gaps in the matrix and hence increase the compressive strength especially at early ages [17].

One of the important factors in the geopolymerization process is curing regime at fresh state of activation. In most cases the curing for alkali activated materials is carried out at elevated temperatures ranging from 40 °C to 80 °C for a period of 4–48 h [19–22]. It is reported that an excessive curing temperature and its duration can negatively affect both setting time and mechanical properties. In a study presented by Al Bakri et al. [23], it was reported that elevated temperatures resulted in higher strength development in fly ash geopolymer paste although temperatures above 60 °C led to strength depletion due to moisture loss from the matrix causing microcracks. Guo et al. [24] also observed that compressive strength began to decrease after applying the curing regime for more than 8 h at temperature of 75 °C. They found that prolonged curing can break down the granular structure of the matrix and also result in dehydration and excessive shrinkage due to contraction of the gel. In a study by Rovnanik et al., formation of large pores in the structure of fly ash geopolymer was observed and consequently the increase of cumulative pore volume disadvantageously affected the mechanical properties of the geopolymeric product [25]. Nonetheless, type of aluminosilicate source is also important in the curing temperature and duration. Previous studies on GGBS as a single or binary binder showed that the optimum curing regime to alkali activate GGBS is from 30 °C to 80 °C within a period of 2 h to 14 days [26–29]. However, in studies by Bernal et al. [30,31] ambient temperature (25 ± 5) °C and 90% relative humidity were reported to be beneficial and enough for hardening the GGBS binder at which high compressive strength was obtained at early ages through formation of C–S–H gel which needs lower temperatures to form.

Palm oil fuel ash (POFA) is one of the agro waste materials from palm oil industry in Southeast Asia. It is estimated that only in Malaysia the wastes generated from palm oil industry is 24.99 million tons per year [32]. So far, POFA, categorized as a pozzolanic material [33–36], and used as a partial replacement for ordinary Portland cement in conventional concrete for the purpose of enhancing strength and durability [37–43]. Recently, some works have utilized POFA as a supplementary material in mixes with other aluminosilicate materials to make geopolymer cement paste, mortars, and concrete. In most researches a low content of POFA (almost 30%) mixed with slag, fly ash, and rice husk ash was used and cured at temperature, 65 °C, and 75 °C for a duration of 24–48 h [28,44–47]. In general, the range of compressive strength was reportedly between 28 and 66 MPa when a low content of POFA was used. However, in a recent study by Salih et al. [14,48], 100% POFA with relatively high content of Ca was used and cured at 60 °C for 2 h, and a compressive strength of up to 32 MPa was achieved. In this study, the POFA was regarded as a higher calcium alkali activated material [5] and the strength was related to formation of C–S–H from available Ca and Si species in the POFA and the silicates from the activator.

So far, there is no comprehensive study on the effect of curing temperature on alkali activation of mixes with 100% POFA. So,

the overarching purpose of this study is to investigate the microstructure and chemical composition of POFA geopolymer binder cured at ambient temperature, 60 °C, 70 °C, and 80 °C. This study proposes the applicability of ambient temperature as a curing regime in POFA geopolymer binders. Investigating the use of ambient temperature to produce a geopolymer binder from agro waste material such as POFA can lower the cost and energy consumption; meanwhile enhances construction pace.

## 2. Experimental method

### 2.1. Materials

#### 2.1.1. Palm oil fuel ash (POFA)

Palm oil fuel ash was collected from a palm oil mill in Johor state, south of Malaysia. The raw POFA was first dried at 105 ± 5 °C for 24 h. Then, POFA was sieved through a 300- $\mu$ m sieve in order to remove the debris and extraneous materials such as unburned fibers and shells [49,50]. Scanning electron microscope (SEM) was used to investigate the particles morphology of the raw POFA as illustrated in Fig. 1. As can be seen, the raw POFA consisted of very irregular shaped particles with porous cellular surfaces [37,40]. The sieved POFA was then grinded by a modified Los Angeles machine for 12 h [42]. The grinding process was an effective way to turn the particles into smaller size and homogeneous powder. The final shape of particle after grinding was spherical in a crushed form with rough surface [51]. Fig. 2 illustrates the shape of POFA particles after grinding. The specific surface area of POFA after grinding was 0.915 m<sup>2</sup>/g.

Table 1 shows the chemical composition of raw POFA after grinding. The major components were SiO<sub>2</sub> and CaO with concentrations of 47.37% and 11.83%, respectively. Alumina Oxide was found in a low amount of 3.53%. The high calcium oxide available in the POFA is most likely from lime and fertilizer [41]. Fig. 3 shows the XRD patterns of ground POFA with a dominant phase of SiO<sub>2</sub> based on Cu radiation. Location of the highest hump was also detected in the XRD profile ranging from 15° to 40° (2 $\theta$ ), representing an amorphous phase [50].

#### 2.1.2. Alkaline activators

In this study, sodium hydroxide and sodium silicate were chosen as the alkali activators. The sodium hydroxide (NaOH) was in industrial-grade with minimum 99% purity. Also, an Industrial grade sodium silicate (Na<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>) solution was chosen with a chemical composition of 15.33% Na<sub>2</sub>O, 31.28% SiO<sub>2</sub>, and 53% H<sub>2</sub>O. The alkaline activator selection was based on the recommendations in [26,48,52].

#### 2.2. Mix proportions of POFA geopolymer paste

POFA was activated by mixing with alkaline activator at a solid to liquid ratio of 1.32 by mass. The alkaline activator consisted of sodium silicate and sodium hydroxide with a mixing ratio of 2.5 prepared 24 h prior to use. These mixing ratios were used based on the optimum activation results from the previous study [14]. Table 2 shows the mix proportion for all series of samples. In this study, the ash was discharged to the mixer pan, then the alkaline was added and mixed for 1.0 min at a normal speed rate. The mixer stopped for 10–20 s in order to scrape the un-mixed ash on the sides of the paddle and the pan, then mixing continued with medium speed for another 1.0 min in accordance to ASTM C305 [53]. All the geopolymer paste mixes were blended and produced with a small blender (HOBART Mixer). The fresh geopolymer paste then was cast into 50 × 50 × 50 mm iron molds in two layers immediately after mixing. For compaction, vibrating table was used for 25–60 s and applied for two layers of the paste. Four different curing temperatures were applied including 25–30 °C as ambient

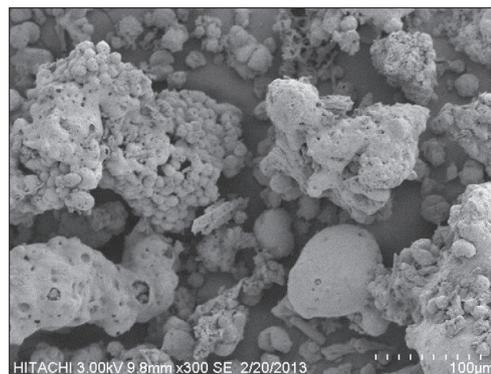


Fig. 1. Raw POFA before grinding.

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