



Compressive strength of treated palm oil fuel ash based geopolymer mortar containing calcium hydroxide, aluminum hydroxide and silica fume as mineral additives



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ABSTRACT

In this work, the compressive strength of treated palm oil fuel ash (TPOFA) based geopolymer mortar was studied. The geopolymer mortar was prepared using mixtures of TPOFA and mineral additives ((Ca(OH)₂, Al(OH)₃, and silica fume (SF)). The alkaline activator was prepared from NaOH and Na₂SiO₃. The samples with different total oxide molar ratios (Al₂O₃:SiO₂, CaO:SiO₂, Al₂O₃:Na₂O, and SiO₂:H₂O) were tested for compressive strength. Moreover, the influence of unreacted mineral additives in the final geopolymer products was also evaluated. The results showed that the early age compressive strength of TPOFA-based geopolymer mortar improved significantly when the source material (TPOFA) was substituted with Ca(OH)₂, exhibiting a relative compressive strength of 188.45% at 3 days. Furthermore, the modification of TPOFA with Al(OH)₃ and SF caused significant improvement, demonstrating a relative compressive strength of 200.90% and 175.22% at 3 and 28 days, respectively. The C–S–H and N–A–S–H binding gels formed at 28 days were transformed over time to C–A–S–H type gel at 120 days which was the most stable product.

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1. Introduction

The cement industry is the second largest producer of the green house gas [1]. On an average, approximately 1 ton of cement is being produced each year for every human being in the world [2]. Hence, in order to protect the environment, the main concern of minimizing CO₂ emission can be realized by reducing the percentage of cement used in making concrete. Therefore, it has been suggested to deploy new technology materials like geopolymers. Geopolymer technology is a generic term coined by Joseph Davidovits in the 1970s and describes all alkali activated materials [3]. These alkali-activated materials are produced through the reaction of an aluminosilicate material rich in (Si + Al) but has low CaO content which is normally supplied in powder form as an industrial by-product or other inexpensive material with an alkaline activator which is usually in the form of a concentrated aqueous solution of alkali hydroxide, silicate, carbonate or sulfate [3,4]. The general

example is the alkali-activated metakaolin or Class F fly ash with medium to high alkaline solutions. The main reaction product formed in this case is a three-dimensional alkaline inorganic polymer, a N–A–S–H or alkaline silicoaluminate hydrate gel regarded as a zeolite precursor [5]. The reaction mechanism involves the dissolution of Si and Al from the starting materials. This results in the formation of polymeric structures such as the poly(sialate) [–Si–O–Al–O], poly(sialate-siloxo) [–Si–O–Al–O–Si–O] or poly(sialate-disiloxo) [–Si–O–Al–O–Si–O], cross-linked [AlO₄][–] and [SiO₄] tetrahedral units, with charge balance ensured by Na⁺ or Ca²⁺ ions [6]. The alkali activation technology term coined by Kuehl in 1908 [7] entails activation of materials particularly rich in calcium and silicon (Me₂O–MeO–Al₂O₃–SiO₂–H₂O system) i.e. Me = Ca and Me₂ = Na⁺ or K⁺. An example is the activation of blast furnace slag (Si + Ca) with a mild alkaline solution, which generates C–S–H and C–A–S–H as main reaction products [8].

The source of aluminosilicate used for the synthesis of geopolymer must contain Al which is readily soluble with an overall molar ratio of Al₂O₃:SiO₂ lying between 1:3.3 and 1:6.5 [9]. Industrial waste products/pozzolanic materials such as treated POFA (TPOFA) which possibly has a wide range of chemical compositions usually contain high amounts of SiO₂. Deficiencies in Al₂O₃ and

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CaO content in a natural pozzolan might be compensated for by adding mineral additives such as $\text{Al}(\text{OH})_3$ or $\text{Ca}(\text{OH})_2$, enabling them to be used as a geopolymer resource [10,11]. $\text{Al}(\text{OH})_3$ could be used as the source of Al_2O_3 for geopolymer systems, resulting in increased aluminate [$\text{Al}(\text{OH})_4$]-species which leads to a faster rate of condensation [11]. Additionally, the $\text{Ca}(\text{OH})_2$ content is also an important factor affecting the setting time and final strength in geopolymer mortar. There are indications that it may also affect other properties of geopolymers [10]. Therefore, the substitution of natural aluminosilicate with an optimum amount of $\text{Ca}(\text{OH})_2$ content may also increase the strength of an alkali-activated industrial waste pozzolan [10]. However, TPOFA is rich in silica with an overall molar ratio of $\text{Al}_2\text{O}_3:\text{SiO}_2$ lying between 1:14.83 and 1:19.27 [12,13], which is too low compared to other source materials previously used [9].

Nevertheless, no research has thoroughly discussed the influence of the unreacted mineral additives i.e. $\text{Al}(\text{OH})_3$ and $\text{Ca}(\text{OH})_2$ combined with the source material which promotes reaction. The use of $\text{Al}(\text{OH})_3$ and $\text{Ca}(\text{OH})_2$ as mineral additives is somehow insufficient to produce a gel of the desired composition. Previous investigations [10,11,14–16] studied the optimum quantity of these additives for better performance, without addressing the influence of the unreacted mineral additives in the final geopolymeric products. In another study [14], the dissolution of $\text{Al}(\text{OH})_3$ with source materials containing high amounts of SiO_2 and CaO with an alkaline activator (NaOH and Na_2SiO_3) were discussed, without evaluating the effect of CaO-promoted reactions. Besides, the presence of the calcium compounds (CaSO_4 and CaO) in the source material was not taken into account. These compounds react with NaOH, promoting gypsum dissolution and the formation of $\text{Ca}(\text{OH})_2$ via the double decomposition reaction [14]. Moreover, the reaction of CaO with water causes the formation of $\text{Ca}(\text{OH})_2$, which in turn can lead to water deficiency on its precipitation. This can further raise the alkalinity [17] causing the dissolution of $\text{Al}(\text{OH})_3$ to form aluminate ions. It is also well known that the rate of gibbsite dissolution is a function of the increase in pH [18].

Chindaprasirt et al. [14] studied the replacement level of $\text{Al}(\text{OH})_3$ and showed that replacement levels of 2.5 wt.% $\text{Al}(\text{OH})_3$ is far better than 5.0 wt.%. The incorporation of 2.5 wt.% of $\text{Al}(\text{OH})_3$ promoted the reaction and provided a dense and homogeneous matrix with the formation of additional aluminosilicate compounds. However, the high dosage of 5 wt.% of $\text{Al}(\text{OH})_3$ resulted in its excess and formed sodium aluminate which weakened the geopolymer. However, the XRD analysis was only discussed in the range of 20–50°. This range is not suitable since the peak of gibbsite ($\text{Al}(\text{OH})_3$) is dominant at around 18° and the intensity increases with the increase of $\text{Al}(\text{OH})_3$ content [11]. This observation is essential to prove the reactivity of $\text{Al}(\text{OH})_3$ with the source material. The peak intensity of gibbsite (18°) must be proven to have reduced in order to validate the research findings, since increased amounts of residual gibbsite ($\text{Al}(\text{OH})_3$) with a small amount of cross-linked geopolymer leads to lower strength matrix [11].

The pure reaction kinetics-based approach to the description of alkali activation has some shortcomings [4]. Most of the previous research only focused on the influence of the molar ratio of $\text{Na}_2\text{O}:\text{H}_2\text{O}$ derived from the alkaline activator [19]. In contrast, no information can be found in the open literature regarding the role of the total molar ratios of $\text{SiO}_2:\text{H}_2\text{O}$ or $\text{Al}_2\text{O}_3:\text{H}_2\text{O}$, and $\text{CaO}:\text{SiO}_2$ derived from alkaline activator and mineral additives such as $\text{Ca}(\text{OH})_2$, $\text{Al}(\text{OH})_3$, and SF, individually, or when they are brought into contact in the same system.

Therefore, the main purpose of this work is to elucidate the effect of the total oxides molar ratio ($\text{Al}_2\text{O}_3:\text{SiO}_2$, $\text{CaO}:\text{SiO}_2$, $\text{Al}_2\text{O}_3:\text{Na}_2\text{O}$, and $\text{SiO}_2:\text{H}_2\text{O}$) through the partial substitution of the source material (TPOFA) with mineral additives to achieve higher

compressive strength geopolymer mortar at the ages of 3, 7, and 28 days. Furthermore, it aims to clarify the influence of the unreacted mineral additives in the production of gel which will affect not only the compositional, mineralogical, and thermo-physical properties but also the compressive strength. Further, the study also investigates the changes in the main gel binder at long-term age of 120 days and quantifies their influence on compressive strength. The study also investigates the probable correlation between compressive strength development and the changes in the main gel binder at long term age at 120 days for the optimum mixture of TPOFA-based geopolymer mortar.

2. Materials and methods

2.1. Materials

The materials used in this study consist of solid materials and alkaline activators. The solid materials were the source material (TPOFA) and mineral additives ($\text{Al}(\text{OH})_3$, $\text{Ca}(\text{OH})_2$, and SF) while the alkaline activator was prepared using NaOH and Na_2SiO_3 with the addition of water.

2.1.1. Solid materials

2.1.1.1. Source material. TPOFA is the major source material used in this research. Raw POFA was collected from a palm oil mill in Nibong Tebal, Penang, Malaysia. The POFA which contained incompletely combusted fibers and palm kernel shells were sieved passing a 300 μm sieve. The POFA was then ground in a ball mill to obtain particle size of about 5 μm . To remove the unburned carbon, the POFA was heated at 500 °C for 1 h in a gas furnace to form TPOFA. The chemical compositions and physical properties of the TPOFA used in this study are provided in Table 1. The TPOFA could be classified as a class F mineral admixture according to ASTM C618 [20]. The same approach had previously been used and was reported to be effective in increasing the efficiency of TPOFA for use in high-strength green concrete [13].

2.1.1.2. Mineral additives. The mineral additives used were $\text{Ca}(\text{OH})_2$, $\text{Al}(\text{OH})_3$, and SF. The commercially available white powder $\text{Al}(\text{OH})_3$ with CAS No: 21645-51-2 and $\text{Ca}(\text{OH})_2$ with CAS No: 1305-62-0 were also used as the sources for Al and Ca, respectively. SF is another source for SiO_2 . The chemical compositions and physical properties of the SF used in this study are provided in Table 1. A laser diffraction particle size analyzer (Mastersizer/E) was used to analyze the particle size distribution and surface area of the

Table 1
Chemical compositions and physical properties of TPOFA and SF.

Compositions	TPOFA Component (%)	SF
SiO_2	61.33	92.26
Al_2O_3	7.018	0.89
Fe_2O_3	5.11	1.97
CaO	8.20	0.49
MgO	4.69	0.96
P_2O_5	4.55	–
K_2O	6.50	1.31
SO_3	0.27	0.33
TiO_2	0.25	–
MnO	0.097	–
Na_2O	0.123	0.42
C	1	0.09
<i>Physical properties</i>		
Specific surface area (m^2/g)	1.0854	0.1364
Loss on ignition (%)	2.53	4.96
Median particle size (μm)	5.30	36.45
Specific gravity (kg/m^3)	2560	2330

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