



Performance of slag blended alkaline activated palm oil fuel ash mortar in sulfate environments



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HIGHLIGHTS

- Combination of slag and POFA in AAGU mortar provide good resistance to sulfate attack.
- Sulfate attack of AAGU mortar depends on the cations present.
- Slag content provides better resistance to sulfate attack than activators ratio.
- $\text{MgSO}_{4\text{aq}}$ attack causes decalcification of C–S–H in AAGU mortar to form M–S–H.
- AAGU mortar has a higher resistance to $\text{MgSO}_{4\text{aq}}$ than $\text{Na}_2\text{SO}_{4\text{aq}}$ attack.

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ABSTRACT

This study investigated the performance of alkaline activated ground steel slag-ultrafine palm oil fuel ash (AAGU) mortar exposed to 5% Na_2SO_4 and 5% $\text{MgSO}_{4\text{aq}}$ for 6 months. The samples cured at 60 °C for 24 h were prepared with 10 M- NaOH_{aq} and $\text{Na}_2\text{SiO}_{3\text{aq}}$ ($\text{Ms-SiO}_2/\text{Na}_2\text{O} = 3.3$) activators. Microstructural and characterization tools were employed to examine the type and nature of the resulting products. The findings showed that there was a better strength retention in the $\text{MgSO}_{4\text{aq}}$ -exposed samples compared to the $\text{Na}_2\text{SO}_{4\text{aq}}$ exposed due to the formation of surficial deposits and intra-microstructurally crystallized anhydrite (CaSO_4). The loss of weight/strength and skeletal disintegrations in $\text{Na}_2\text{SO}_{4\text{aq}}$ exposure resulted from the leaching of active elements (Ca, Al and Mg). Slag significantly contributed to the sulfate resistance of AAGU mortars, while there was an insignificant difference in strength retention when the activator ratios ($\text{Na}_2\text{SiO}_{3\text{aq}}:\text{NaOH}_{\text{aq}}$) varied within 1.0–2.5 for the samples prepared with a workable mixture.

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1. Introduction

The release of excess greenhouse gases (GHGs) and the need for the reduction in the environmental agro-industrial solid wastes have recently prompted the synthesis of alkaline activated ground slag/ultrafine palm oil fuel ash (AAGU) binding products [1,2]. Ultrafine palm oil fuel ash (UPOFA) is the post treated palm oil fuel ash (POFA) solid wastes obtained from burnt palm kernel shell and bunches used in electricity generation in an oil mill plant.

Further, POFA contributes to the solid wastes whose disposal poses an environmental challenge. For instance, 3 million tons of POFA was generated in Malaysia in 2007, while the annual POFA generation in Thailand amounted to about 0.1 million ton [3,4]. The strength of AAGU binder was reported to be better than that of ordinary Portland cement (OPC) [2,5]. This paper addresses the response of AAGU mortars to the sulfate solutions (Mg, Na) as a

way of studying its durability performances with a view to complementing the available studies on the response of alkaline activated binders (AAB) prepared from solid waste products such as slag and fly-ash to sulfate environment [6–8]. Other studies previously conducted on the durability of AAB or geopolymer include their responses to sulfuric acid [9–11] and the elevated temperature [12,13].

Sulfate attack on concrete products could cause both internal and external defects due to the contamination of the constituent materials and their exposure to contaminated environments, respectively [14,15]. Various factors affect the sulfate resistance of mortar or concrete. These include the type of the cations (Mg, Na) carried by the sulfate ions (SO_4^{2-}) in the salt solutions, the concentration of the sulfate solution, and the permeability coefficient of the exposed mortar samples [16]. The chemistry of the solid skeleton of AAB, which in turn depends on the type of activators, mineralogy of the aggregates and base materials used in the synthesis are also among the factors controlling the density of their

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microstructures [17]. Some of the failures that accompany the sulfate attack on mortar or concrete include the loss of strength, microstructural expansion, formation of products such as gypsum (calcium sulfate-dihydrate, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$) [14]. The more aggressive nature of $\text{MgSO}_{4\text{aq}}$ compared to Na_2SO_4 solution has been reported [18,19], especially for the OPC products exposed to the sulfate solutions, while the products like brucite ($\text{Mg}(\text{OH})_2$) and gypsum are also identified in the aftermath of the attack.

The possibility of Ca–Mg ionic exchange that resulted in the formation of serpentine or magnesium silicate hydrate (M–S–H) from calcium–silicate–hydrate (C–S–H) has been adduced to more aggressive nature of $\text{MgSO}_{4\text{aq}}$ compared to $\text{Na}_2\text{SO}_{4\text{aq}}$ [20]. In fact, the M–S–H ($\text{Mg}_8\text{Si}_8\text{O}_{20}(\text{OH})_8 \cdot 12\text{H}_2\text{O}$) formed could be the combinations of poorly crystallized sepiolite ($\text{Mg}_2\text{H}_2\text{Si}_3\text{O}_9 \cdot x\text{H}_2\text{O}$) and serpentine ($\text{Mg}_3(\text{OH})_4(\text{Si}_3\text{O}_5)$) [21]. Besides, Aye and Oguchi established that $\text{MgSO}_{4\text{aq}}$ deleterious effect emanated from chemical attack or ionic exchange, while $\text{Na}_2\text{SO}_{4\text{aq}}$ exposure of OPC products was observed to be more harmful compared to $\text{MgSO}_{4\text{aq}}$ from physical attack standpoint [20].

Further, Thokchom et al. [22] studied the performance of fly-ash based geopolymer mortar in 10 wt.% MgSO_4 solution within 24 weeks. The effects of such exposure were found to be a deposition of white crystal on the sample surface with additional gain in weight. Besides, changes in pH and reduction in the residual compressive strength were also identified among other consequences of sulfate attacks. The deterioration of fly ash-based geopolymer exposed to 5% of Na_2SO_4 and MgSO_4 salts was also reported to be caused by leaching of alkali and penetration of sulfate ions through diffusion whose degree was more in the former than the latter [23]. The least deterioration, however, was observed when samples were exposed to the combined sulfate solutions ($\text{Na}_2\text{SO}_{4\text{aq}}$ + $\text{MgSO}_{4\text{aq}}$) [23].

In addition, [7] observed that durability of the alkaline activated water cooled slag exposed to 5% MgSO_4 displayed a better microstructural matrix and high resistivity such that, the performance of the product prepared with the quantities of $\text{NaOH}_{3\text{aq}}$ and $\text{Na}_2\text{SiO}_{3\text{aq}}$ of 3:3 (wt.%), surpassed that prepared with only 2 or 6% of $\text{NaOH}_{3\text{aq}}$ and 4 or 0% of $\text{Na}_2\text{SiO}_{3\text{aq}}$, respectively. Moreover, the lesser susceptibility of bottom ash (BA) geopolymer to sulfate and sulfuric acid attacks compared to OPC was also reported [10]. Investigation on fly-ash/slag geopolymer binder performance in the sulfate (5% MgSO_4 and Na_2SO_4 solutions) environments revealed more extensive physical deteriorations in $\text{MgSO}_{4\text{aq}}$ due to the formation of an expansive gypsum product compared to $\text{Na}_2\text{SO}_{4\text{aq}}$ exposure [16]. They also identified that low water/binder ratio was one of the factors that mitigated against the sulfate attack of geopolymer or alkaline activated binder [16].

Be that as it may, this study provides a better understanding of durability performance of AAGU mortar in the sulfate environments of different cations (Mg, Na), and also enhances the practical application of AAGU mortar as an alternative material of construction to OPC. It also promotes the utilization of solid wastes that constitute landfill and environmental pollution as construction materials for building structures or other civil engineering applications.

2. Materials and methods

2.1. Materials

2.1.1. Ultrafine palm fuel ash and ground steel slag

Slag and palm oil fuel ash (POFA) were collected from Malaysian Southern Steel Company and United Palm Oil Industry, respectively. These pozzolanic materials (PMs) – original raw POFA and slag – were subjected to oven drying at the temperature of $105 \pm 5 \text{ }^\circ\text{C}$ for 24 h to get rid of their inherent moistures. The samples were sieved through 300- μm sieve to remove stones and debris. Slag was then

ground for 16 h to become ground slag (GS). POFA was first ground for 8 h before subjecting it to heat treatment in a gas-powered clay furnace at $500 \text{ }^\circ\text{C} \pm 50 \text{ }^\circ\text{C}$ for 90 min to remove carbon content, and any volatile substances [17]. The treated POFA was further ground for 8 h to become ultrafine palm oil fuel ash (UPOFA). The milling of the raw materials was done by using a ball-milling machine rotating at a speed of 180 rev/min.

The particle size distributions (PSDs) of the PMs were determined with Microtrac (Turbotrac) model S3500 particle size analyzer. The oxide compositions of the PMs were obtained by using X-ray fluorescence (XRF) technique while the surface areas (BET) of the PMs were determined with Micromeritics ASAP2020 by using nitrogen gas adsorption.

2.1.2. Preparation of alkaline activators

The commercially available $\text{Na}_2\text{SiO}_{3\text{aq}}$ was used together with 10 M- NaOH_{aq} as the alkaline activators. $\text{Na}_2\text{SiO}_{3\text{aq}}$ comprised H_2O : 62.11%, SiO_2 : 29.13% and Na_2O : 8.76% such that its initial silica modulus ($M_s = \text{SiO}_2/\text{Na}_2\text{O}$) was 3.3. The 10 M- NaOH_{aq} was prepared by dissolving 404.4 g of NaOH pellet (99% assays) into distilled water to form 1 L of alkali solution (10 M- NaOH_{aq}).

2.1.3. Preparation of sulfate solutions

The sulfate salts Na_2SO_4 (CAS Number 7757-82-6) and MgSO_4 (CAS Number 7487-88-9) of percentage purity > 99% were used. Each solution was prepared by dissolving 50 g of the solutes in distilled water to obtain 1 L (50 g/dm³) of the sulfate solution.

2.1.4. Aggregates

Mortars prepared comprised fine aggregates (FAs), which were dune sand with the specific gravity of 2.62 in the saturated and surface dry condition (SSD) and the fineness modulus of 1.85. The percentage water absorption for fine aggregates was 0.57% [24].

2.2. Analytical methods

The 14-day pulverized mortars were used for the X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy analyses. The samples were collected at an approximate depth of 10 mm from the sample surface. FTIR of the samples was conducted using Perking Elmer 880 spectrometer (KBr pellet technique), while X-ray diffraction analysis (XRD) was obtained with the semi-quantitative X-ray diffraction (XRD) instrument. Copper (Cu) K_α monochromatic radiation ($\lambda = 1.5406 \text{ \AA}$), 2θ from 10° to 90° ; with step interval 0.0341° , while a commercial software was used for the XRD mineral compound or phase analyses.

X-ray fluorescence analyses (XRF) was obtained by using XRF-spectrometer with a fully automated programmable operation of Rh/W dual X-ray tube, which is capable of analyzing liquid, solids and powder. The fragmented portions of the samples obtained from compressive strength test were used to determine the morphologies of the matrix by using field emission scanning electron microscope couple with energy dispersive spectroscopy (FESEM + EDS) equipment.

2.3. Experimental design

2.3.1. Mixture proportion for the constituents in AAGU mortar

Alkaline activated mortar has an approximate unit weight of 2400 kg/m³ [25]. The mortars were prepared with GS/PMs of 0 and 0.2. $\text{Na}_2\text{SiO}_{3\text{aq}}/\text{NaOH}_{\text{aq}}$ (NS/NH) varied as 1.0 and 2.5 as shown in Table 3. Free water content to pozzolanic materials ratio (FWC/PMs) was kept at 2 wt.% with the adjustment for the water absorption of the aggregates. The fine aggregates to pozzolanic materials (FA/PMs) and (NS + NH)/PMs were 1.8 and 0.5, respectively.

2.3.2. Mixing, placement and testing of AAGU mortar

The constituent materials were mixed in a 4.73 L planetary bench mixer. The PMs were first mixed for 2 min to remove the air pockets and this was followed by adding free (portable) water content (FWC), NaOH_{aq} , $\text{Na}_2\text{SiO}_{3\text{aq}}$ and FAs, sequentially. Each material was mixed for 2 min such that the total mixing time for the mortar was 12 min. The mixtures were cast in oil smeared prismatic steel mould of $50 \times 50 \times 50 \text{ mm}^3$ in two layers and each layer was vibrated for 15 s. The cast samples were then covered with vinyl bag to prevent moisture loss, and then later kept in the laboratory at temperature of $25 \text{ }^\circ\text{C}$ for 12 h for easy demoulding.

Subsequently, the samples were then cured in an oven at $60 \text{ }^\circ\text{C}$ for 24 h. The cured samples were exposed to the sulfate solution 30 min after removing them from the oven. The unexposed (control) samples were used for the determination of 3, 7 and 28-day compressive strength. The strength was tested with a crushing machine at a loading rate of 0.9 kN/s. The average failure stress (MPa) of three specimens was recorded as the compressive strength value. The loss in the sulfate solutions in which the samples were submerged were made up by freshly prepared solution. The weights of the samples were initially determined initially on weekly, and subsequently on monthly basis for 6 months.

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