



# Shrinkage and strength of alkaline activated ground steel slag/ultrafine palm oil fuel ash pastes and mortars



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## ABSTRACT

This study evaluated the contributions of steel slag and activators ratio to the shrinkage of the alkali-activated ground steel slag (G)/ultrafine palm oil fuel ash (U) or AAGU pastes and mortars. The base materials were combined such that G/U+G varied from 0 to 0.8 (pastes) and 0–0.6 (mortars) with the use of 10M-NaOH<sub>aq</sub> and Na<sub>2</sub>SiO<sub>3aq</sub> (M<sub>s</sub> = SiO<sub>2</sub>/Na<sub>2</sub>O of 3.3) as activators whose ratios (Na<sub>2</sub>SiO<sub>3aq</sub>/10M NaOH<sub>aq</sub>) were varied as 1.0/1.0 and 2.5/1.0. The findings revealed that steel slag reduced the AAGU shrinkage through pore-refinement, elimination of microcracks, and improvement in the microstructural density and strength. The changing of Na<sub>2</sub>SiO<sub>3</sub>/10NaOH ratio in the synthesis of AAGU products from 2.5 to 1.0 slightly reduced the shrinkage through the modification of amorphousness and nature of the products (C-A-S-H/C-S-H). The maximum 90-day slag-free AAGU paste and mortar shrinkages were 60.80 × 10<sup>3</sup> με and 11.82 × 10<sup>3</sup> με but reduced to 25.88 × 10<sup>3</sup> and 2.71 × 10<sup>3</sup> με, respectively as G/(U+G) = 0.4 in AAGU<sub>0.4</sub>.

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## 1. Introduction

Palm oil fuel ash (POFA) is an agro-waste that is produced in large quantities in East-Asian (Indonesia, Thailand and Malaysia) and West African countries (Benin Republic, Ghana and Nigeria). POFA is generated from oil palm wastes that include fibers, nutshells, and empty fruit bunches whose contributions to the environmental solid wastes are very substantial. The quantity of POFA is so enormous that about 70% of the raw materials used in the production of palm oil are stockpiled as landfills [1]. For instance, about 3 million tons of POFA was generated in Malaysia in 2007 while about 0.1 million ton of POFA is generated on annual basis in Thailand [2,3]. Hence, POFA may constitute environmental challenges if not properly disposed, recycled or utilized in a productive manner. The increase in POFA organic impurities and carbon content together with its coarse particle sizes have limited its utilization either as a partial supplementary cementitious material (SCM) for ordinary Portland cement (OPC) or as a base material in

the synthesis of alkaline activated products [4–10] despite its high pozzolanicity and rich silica content [1,11,12].

Further, the pozzolanic properties of raw POFA could be improved when subjected to the pre-treatment such as grinding and calcination at an elevated temperature. These treatments reduce its carbon content and increase its surface area [11]. Similarly, steel slag is also an industrial waste generated from ladle wastes during steel production. It has higher CaO/SiO<sub>2</sub> ratio that could contribute to strength development and hydration process of in the synthesis of alkaline activated ground slag/ultrafine palm oil fuel ash (AAGU) products [6,8].

The possibility of attaining a substantial early strength, reducing energy utilization and minimizing the proliferation of CO<sub>2</sub> that accompanied OPC manufacturing processes have made an alkaline activated binder (AAB) a viable alternative to OPC for the construction and other civil engineering applications. There have been no studies on the durability of AAGU paste/mortar despite the various studies in the recent times on its strength and microstructural characteristics [4–8]. Shrinkage is defined as the volumetric change in the geometry of a matrix due to the removal of water from the surface (plastic shrinkage) and gel (drying shrinkage) or due to self-desiccation (autogenous) and replacement of heavier molecules with lighter ones (carbonation) within the matrix of a binder.

The distinction of autogenous and drying shrinkage of an oven-cured AAB or geopolymer is very challenging due to its method of

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synthesis. This is unlike OPC paste/mortar [13–16] or alkaline activated slag (AAS) [17–20] where various studies have been conducted on their autogenous shrinkage characteristics. Autogenous shrinkage (AS) is caused by a reduction in the pore relative humidity (RH) due to the formation of hydration products that lead to the development of tensile stresses, which relate inversely to the pore diameter. The developed internal tensile stresses were reported to lead to the development of a concomitant deformation-induced compressive stress that caused cracks within the product matrices [13,19,21].

In another vein, non-autogenous shrinkage includes the carbonation shrinkage, drying shrinkage, thermal deformation, and creep shrinkage [22,23]. Drying shrinkage is a measure of total deformation of the samples due to its exposure to a specific relative humidity (RH) and ambient temperature. In other words, drying shrinkage is a macroscopic dimensional reduction of hardened binders due to the evaporation of water or moisture within the matrices of the products [24]. However, plastic shrinkage is caused by an imbalance in the moisture exchanges between a specimen surface and its environment [25].

There had been very few studies on the shrinkage of AABs or geopolymers. To this end, Kani and Allahverdi investigated alkaline activated natural pozzolans geopolymer binder, and asserted that curing type and chemical compositions of the base materials affected the shrinkage of the product. They added that the Si/Na ratio had a direct correlation with shrinkage [26]. The oven-cured fly ash-based geopolymer concrete had been affirmed to have a lower drying shrinkage that was far lesser than the Gilbert's shrinkage prediction model [15,27]. The oven-dry curing of AAB accelerated the initial drying shrinkage of fly-ash geopolymer concrete while the connected capillary networks emanated from the formation of aluminosilicate product reduced the total shrinkage of the sample [10,23].

Further, the decrease in NaOH<sub>aq</sub> concentration (NC) reduced the drying shrinkage of high calcium fly-ash geopolymer paste [28], but NC sparingly affected total shrinkage in fly-ash based geopolymer mortar [29]. The shrinkage recorded in fly-ash geopolymer concrete had a value of 400  $\mu$ strain/year which was lower than the 700  $\mu$ strain limit of AS 3600 [23,30]. The strength of AAB, liquid-to-ash ratio of the mixture and the adopted curing temperature were among the identified controlling parameters of AAB shrinkage. Besides, Jin et al. [31] reported that 2.5% of highly reactive MgO mitigated drying shrinkage by accelerating early hydration of alkaline activated slag, but in the excess of this quantity, the surface cracks was developed around the surfaces of the sample.

This research aimed at studying the total shrinkage and strength behaviors of alkaline activated ground steel slag/ultrafine palm oil fuel ash pastes and mortars. The contributions of steel slag in the reduction of shrinkage in the AAGU were discussed in details. The maximum 90-d shrinkage obtained in the slag free AAGU paste and mortar specimens were found to be slightly lesser than 6.1% strain ( $6.1 \times 10^4 \mu\epsilon$ ) and 1.2% strain ( $1.2 \times 10^4 \mu\epsilon$ ), respectively. These values further reduced upon adding steel slag and changing the alkaline activators proportion or Na<sub>2</sub>SiO<sub>3aq</sub>/10M NaOH<sub>aq</sub> ratio from 1.0 to 2.5. The contributions of steel slag and the varied activators ratio ranged from the densification of the microstructure and changing of the nature/amorphousity of the product to the reduction in degree of carbonation and microcracks within the interstices of the pastes/mortars.

On the other hand, this study also promotes the utilization of solid waste materials with a view to fostering a sustainable environment and enhancing the provision of an alternative binder to OPC. This will reduce the carbon proliferation and other environmental pollutions that accompany the accumulation of solid wastes.

## 2. Materials and methods

### 2.1. Materials

#### 2.1.1. Pozzolanic base materials

Steel slag and palm oil fuel ash (POFA) were obtained from Malaysian Southern Steel Company and United Palm Oil Industries, respectively. The steel slag was ground for 16 h in the mechanical ball-milling machine containing 150 balls of different sizes ranging from 6 to 32 mm diameter at the speed of 180 rpm to produce *ground steel slag* (GSS (G)). The POFA, on the other hand, was first ground for 8 h, calcined at the temperature of 500 °C + 50 °C and then re-ground for additional 8 h to obtain *ultrafine POFA* (UPOFA(U)). The paste specimens were prepared with the combined pozzolanic materials (PMs=G+U) such that G/PMs or G/U+G varied as 0, 0.2, 0.4, 0.6, to 0.8 while the mortar specimens were prepared with G/(U+G) ratio which varied as 0, 0.2, and 0.4.

#### 2.1.2. Preparation of alkaline activators

The alkaline activators used were 10M NaOH<sub>aq</sub> (NH) and Na<sub>2</sub>SiO<sub>3aq</sub> (NS) of initial silica modulus ( $M_s$  or  $n = \text{SiO}_2/\text{Na}_2\text{O}$ ) of 3.3 such that H<sub>2</sub>O = 62.1 wt.% SiO<sub>2</sub> = 29.13 wt.% and Na<sub>2</sub>O = 8.76 wt.%. The solution of 10M NaOH<sub>aq</sub> was prepared by dissolving 404 g of NaOH pellet (99% purity) in distilled water to make 1-liter of solution. The reaction between NaOH pellet and water was exothermic so the solution was allowed to cool-down for 24 h in the laboratory (25 °C). All the paste and mortar specimens were prepared with (Na<sub>2</sub>SiO<sub>3aq</sub>+10M NaOH<sub>aq</sub>)/PMs or (NH+NS)/PM ratios of 0.35 and 0.5, respectively. The effects of NS/NH ratios of 2.5 and 1.0 were also examined on shrinkage of AAGU mortar. The mixing water expressed as free water content to pozzolanic material ratio (FWC/PMs) was kept constant at 2 wt.% in all the mixtures [5].

#### 2.1.3. Fine aggregates

The fine aggregates (FAs) were dune sand of fineness modulus of 1.85 with the specific gravity of 2.62 in the saturated and surface dry (SSD) condition, while the FA/PM = 1.8 was adopted for the preparation of all the mortar samples.

### 2.2. Analytical methods

The particle size distributions (PSDs) of the PMs – GSS and UPOFA were determined with *Microtrac (Turbotrac)* instrument model S3500 particle size analyzer (PSA). The oxide compositions of the PMs were also determined using X-ray Florescence (XRF) technique and the values were as shown in Table 1. The *Micromeritics ASAP2020* equipment was used to determine Brunauer–Emmett–Teller (BET) surface areas by using nitrogen gas adsorption at the temperature –195.8 °C (77 k). The instrument also determined the Barrett–Joyner–Halenda (BJH) pore sizes, pore areas, and adsorption pore volumes of the two PMs and the results

**Table 1**  
Oxide percentage compositions of ultrafine palm oil fuel ash (UPOFA) and ground steel slag (GSS).

Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Na <sub>2</sub> O	MgO	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	C
UPOFA	60.42	4.26	3.34	11.00	0.18	5.31	5.03	4.48	0.45	4.86
GSS	25.78	4.42	1.94	53.54	0.03	9.50	0.05	0.04	3.85	0

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