



# Shrinkage characteristics of alkali-activated fly ash/slag paste and mortar at early ages



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## ABSTRACT

The purpose of this study is to investigate the shrinkage characteristics of alkali-activated fly ash/slag (henceforth simply AFS) and the factors affecting it. A series of tests were conducted to determine the chemical shrinkage, autogenous shrinkage and drying shrinkage. The microstructures and reaction products were also characterized through XRD and SEM/EDS analyses. An increase in the slag content from 10% to 30% resulted in a denser matrix and showed a higher Ca/Si ratio of C–N–A–S–H in the microstructure. Higher sodium silicate and slag contents in a mixture caused more chemical, autogenous, and drying shrinkage, but led to a higher compressive strength. From the test results, it can be concluded that the autogenous shrinkage of AFS mortar occurs mainly due to self-desiccation in hardened state rather than volume contraction by chemical shrinkage in fresh state. The AFS paste showed higher drying shrinkage than ordinary Portland cement (OPC), which may be caused by the higher mesopore volume of the AFS paste compared to that of OPC paste.

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## 1. Introduction

A large amount of CO<sub>2</sub> is generated during the manufacturing process of cement, and this has significantly affected global warming and atmospheric pollution [29]. This problem has been gradually on the rise in recent years. As an alternative to this, many studies of new materials instead of cement have been conducted over the last 10 years: particularly, geopolymer and alkali-activated slag (AAS), which are synthesized using an alkaline activator with fly ash, metakaolin or slag, have been studied by many researchers [8,33,26,21,9].

It is known that geopolymer concrete cured at a relatively high temperature, above 60 °C, has good mechanical properties. Some researchers have shown that the curing temperature plays an important role in the polymerization process of a fly ash-based geopolymer [16,43,35]. In addition, some researchers have studied the effect of calcium on geopolymers in order to improve the mechanical properties, discovering that calcium is effective in increasing compressive strength of geopolymers [48,50,25]. Kumar et al. [22] improved the low reactivity of alkali-activated fly ash/slag by adding granulated blast furnace slag (GBFS) to a fly ash/slag mixture.

Most studies of alkali-activated fly ash/slag mixture have focused on microstructure and mechanical properties [27,22,28,24,18], whereas shrinkage characteristics of alkali-activated fly ash/slag blended mortar and concrete have been investigated very little. Slag addition to a fly ash-based geopolymer may also affect the shrinkage of a fly ash/slag binder. The lack of research on shrinkage may actually result in some problems for practical applications.

Many more studies on the shrinkage of fly ash-based geopolymers and AAS have been conducted compared to alkali-activated fly ash/slag binders. According to the previous research [46,47,15], geopolymers show relatively low shrinkage and have good mechanical properties compared to ordinary Portland cement (OPC). However, geopolymers require elevated curing temperatures to obtain low shrinkage and high compressive strength, which may result in significant energy consumption, thus having an environmental problem. On the other hand, AAS has been shown to exhibit a higher rate of shrinkage compared with OPC [7,44,6], which can cause severe defects when it is practically applied. Bakharev et al. [2] and Palacios and Puertas [34] studied the effect of a shrinkage-reduced admixture (SRA) to reduce the shrinkage of AAS, and Collins and Sanjayan [5] studied the effect of the pore size distribution on the shrinkage of AAS to determine the causes of shrinkage. It was also found that the shrinkage of AAS was dependent upon certain parameters, including the type and contents of the alkaline activator [39,40,1] and the curing conditions [51]. Recently, Sakulich and Bentz [37] showed that the autogenous shrinkage of alkali-activated

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slag mortar could be reduced by using pre-wetted lightweight aggregate due to its internal curing effect.

Fly ash-based geopolymers, AAS and alkali-activated fly ash/slag are expected to have different shrinkage characteristics from each other since their shrinkage characteristics primarily depend on the type of alkali-activator used and the main reaction sources such as fly ash and slag. In addition, given that fly ash-based geopolymer, AAS and alkali-activated fly ash/slag represent different reaction products depending on their own characteristics and properties [23], the shrinkage properties of alkali-activated fly ash/slag blends may be more complex than those of fly ash-based geopolymers or AAS.

The purpose of this study is to investigate the shrinkage characteristics of alkali-activated fly ash/slag (henceforth denoted simply as AFS) and the factors affecting it. A series of tests were conducted to determine the chemical shrinkage, autogenous shrinkage and drying shrinkage. The microstructures and reaction products were characterized through X-ray powder diffraction (XRD) and scanning electron microscopy (SEM)/energy dispersive spectroscopy (EDS) analyses. A mercury intrusion porosimetry (MIP) test was conducted to investigate the relationship between the mesopore volume and shrinkage.

## 2. Experimental program

### 2.1. Materials

In the present study, class F fly ash and blast furnace slag (BFS) were used as binder materials, and their chemical compositions are listed in Table 1. Sodium silicate powder was used as an alkali-activator. The chemical composition of sodium silicate powder (Duksan Chemicals Co., Korea) is SiO<sub>2</sub> (53.4 wt.%) and Na<sub>2</sub>O (25.2 wt.%) and its bulk density 0.62 g/cm<sup>3</sup>. The molar ratio of silica to sodium in the sodium silicate powder ( $M_s = \text{SiO}_2/\text{Na}_2\text{O}$ ) is 2.18. River sand was used as fine aggregates. The specific gravity and water absorption of the sand were 2.55 (saturated surface dry condition) and 0.08%, respectively.

### 2.2. Mixture proportions and specimen preparation

The water-to-binder (fly ash + slag) mass ratios (*w/b*) of AFS mortar were 0.34 and 0.42 by mass, while the water-to-binder mass ratio of AFS paste was 0.34. These values were selected through a preliminary study that considered compressive strength and flowability. The sand-to-binder mass ratio of AFS mortar was 2.0. The mixture proportions for the tests are provided in Table 2 and are denoted with specific codes. The labels 'M', 'S', 'A' and 'W' represent the mortar, slag, alkali activator (sodium silicate) and water, respectively. The first numbers, '10', '20' or '30', refer to the percentage of slag replacement for fly ash. The second numbers, '12', '16' or '20', refer to the ratio of sodium silicate to total binder (fly ash + slag) by mass, which are expressed as a percentage. The third numbers, '0.42', refer to the ratio of water to total

binder by weight. The label 'GP' represents geopolymer products which consist of only fly ash, but not slag. The GP-A specimen was cured at 20 °C until testing, and the GP-H specimen was cured at 60 °C for 24 h and thereafter at 20 °C until testing.

The method of producing AFS mixtures is as follows. First, fly ash, slag, and sodium silicate powder were dry mixed for 2 min to ensure homogeneity of the mixture. Mix water was added to the mixture and the mixture was mixed for an additional 2 min. The AFS mortar specimens prepared as described above were immediately cast into 50 mm cubic molds to measure their compressive strength and into 25 mm × 25 mm × 285 mm prismatic molds to measure their autogenous and drying shrinkage.

All the specimens were cured at a temperature of 20 °C and at a relative humidity of 50% in a room with a constant temperature and humidity. After one day, all the specimens for compressive strength and drying shrinkage measurements were removed from their molds and stored in a conditioning room until the day of testing.

### 2.3. Experimental details

Compressive strength testing was conducted using a universal testing machine in accordance with ASTM C39 at 7 d, 14 d, 28 d, and 56 d. X-ray powder diffraction (XRD) data were recorded on a Rigaku D/MAX-2500 machine using Cu K $\alpha$  radiation at a scanning rate of 2°/min from 2° to 160° in 2 $\theta$ . The specimens were prepared in powder form by mechanical grinding.

Scanning electron microscopy (SEM) with back-scattered electron (BSE) images and energy dispersive spectroscopy (EDS) analyses were performed to analyze the polished surfaces of the specimens on a NOVA 230 device (FEI Company). Specimens were impregnated using a low-viscosity epoxy resin, polished with SiC paper, and then coated with gold.

The pore size distribution of the AFS mortar was measured using the mercury intrusion porosimetry (MIP) method in accordance with ASTM D4284-07 on an Autopore VI machine by Micromeritics Corp. The contact angle and the surface tension of mercury were assumed to be 130° and 485 dynes/cm, respectively.

Chemical shrinkage tests were conducted using a volumetric method according to ASTM C1608, which is based on the method described by Geiker [11]. In the present study, fly ash and slag were used as cementitious materials instead of OPC. Water is generally used to produce cement paste, while an alkali-activator containing water and sodium silicate powder was used to produce AFS paste in the present study. The alkali-activator was prepared by mixing sodium silicate powder into water for use in the chemical shrinkage test. According to ASTM C1608, chemical shrinkage is calculated as the measured weight of sorbed water (mL) per gram of cement (in the present study, the total weight of the fly ash and slag). The mass of the cement powder in the vial is given by (ASTM C1608):

$$M_{\text{cement}} = \frac{(M_{\text{vial+paste}} - M_{\text{vialempty}})}{(1.0 + \frac{w}{c})} \quad (1)$$

Here,  $M_{\text{cement}}$  is the mass of cement in the vial (g),  $M_{\text{vial+paste}}$  is the mass of the glass vial with the added cement paste (g),  $M_{\text{emptyvial}}$  is the mass of the empty vial (g),  $w/c$  is the water-to-cement ratio according to the mass of the prepared paste (in this study, 0.34) and the density of water is assumed to be 1.0 g/cm<sup>3</sup>.

The chemical shrinkage per unit mass of cement at time  $t$  is computed as (ASTM C1608):

$$cs(t) = \frac{[h(t) - h(60 \text{ min})]}{M_{\text{cement}}} \quad (2)$$

In this equation,  $cs(t)$  is the chemical shrinkage at time  $t$  (mL/g cement) and  $h(t)$  is the water level in the capillary tube at time  $t$  (mL).

**Table 1**  
Chemical composition of the binder materials (%).

	Fly ash (FA)	Blast furnace slag (BFS)
CaO	6.26	42.47
SiO <sub>2</sub>	42.1	35.17
Al <sub>2</sub> O <sub>3</sub>	28.6	13.93
Fe <sub>2</sub> O <sub>3</sub>	14.4	0.58
SO <sub>3</sub>	0.61	2.03
MgO	2.6	4.12
Na <sub>2</sub> O	–	0.15
K <sub>2</sub> O	2.4	0.46

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