



Understanding the drying shrinkage performance of alkali-activated slag mortars



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ABSTRACT

In this work, drying shrinkage of four alkali-activated slag (AAS) mortars, prepared using various types/dosages of activator, was characterized at four different levels of relative humidity (RH) and two drying regimes (i.e. direct and step-wise drying). The results show that drying shrinkage values of AAS are significantly dependent on the drying rate, as AAS shrinks more when the RH is decreased gradually, instead of directly. At high RH, the drying shrinkage of AAS exhibits a considerable visco-elastic/visco-plastic behavior, in comparison to ordinary portland cement (OPC). It is concluded that the cause of high-magnitude shrinkage in AAS mortar is due to the high visco-elastic/visco-plastic compliance (low creep modulus) of its solid skeleton. Furthermore, the activator affects the shrinkage behaviors of AAS by influencing the pore structure and mechanical properties.

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1. Introduction

Ground granulated blast-furnace slag is an amorphous by-product of iron industry. Research has shown that blast-furnace slag can be alkaline activated to potentially substitute the ordinary Portland cement (OPC) as an alternative binder in concrete [1,2]. Alkali activation of slag is the process of promoting the dissolution of aluminosilicate vitreous structures in slag in a high pH environment. The dissolved ionic species, including aluminates, silicates, calcium, and alkali ions, can then form various types of amorphous phases, including calcium-silicate-hydrate (C-S-H), calcium-aluminosilicate-hydrate (C-A-S-H), and alkali-aluminosilicate-hydrate (N-A-S-H), which provide the binding capabilities in concrete [3–5].

Alkali-activated slag (AAS) concrete can potentially offer high compressive strength and excellent performance against fire and chemical (acid, sulfate) attack [2,6]. Recent environmental life-cycle assessment studies show that AAS concrete provide 25–50% reductions in CO₂ emission and more than 40% reductions in embodied energy relative to OPC concrete [1,7,8]. Despite their favorable performance, the marketability and acceptance of AAS concretes in the industry have been hindered partly due to

concerns related to their long-term durability [2], such as extensive shrinkage and micro-cracking [9,10], rapid carbonation [11], and potential alkali-aggregate reaction [6]. Perhaps one of the most significant conundrums impeding the implementation of AAS concrete is their volumetric instability.

Many researchers have reported that AAS has a significantly higher drying shrinkage than OPC [9,10,12–14], and investigated the shrinkage characteristics of AAS as a function of activator type/dosage [13,15], curing conditions [16,17], and aggregate properties [18,19]. For instance, it was reported that the increased silicate content and dosage of activator can potentially increase the shrinkage of AAS [13,15]. However, the scientific reasoning and mechanisms of the extensive large shrinkage in AAS have not yet been comprehensively understood. Instead, many researchers attributed the large shrinkage of AAS merely to its refined pore structure, as a high capillary force builds in the materials with a refined pore structure [12,13]. There is no doubt that the difference of pore structure between AAS and OPC can considerably affect the shrinkage behaviors. Nevertheless, previous investigations ignore the potential intrinsic difference between the main hydration products of AAS and OPC in term of shrinkage responses.

Previous experiments mainly characterized the drying shrinkage of AAS at merely 50% relative humidity (RH) atmosphere. Former studies have shown that AAS is likely vulnerable to carbonation [20], which may cause decalcification shrinkage of C-A-S-H during the tests. To eliminate the potential influence of

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carbon dioxide in the atmosphere, the drying shrinkage of AAS should be measured in an inert gaseous condition. Furthermore, the data about drying shrinkage of AAS over a broad range of RH is limited. Characterizing the shrinkage of AAS at various RH is important since it is generally believed that different shrinkage mechanisms may dominate at different RH ranges and over various sizes of pores [21]. A comprehensive characterization of the shrinkage can contribute to an in-depth understanding of the properties of AAS and guide development of shrinkage mitigating strategies, which can improve the long-term durability of AAS concrete. The aforementioned knowledge gap needs to be bridged before AAS concrete can become a marketable and viable alternative to the energy intensive OPC concrete.

The main objectives of this research are to understand the shrinkage mechanism of AAS, and to illuminate the intrinsic reasons that are responsible for its large shrinkage and volumetric instability. In this paper, the shrinkage performance of four AAS mortars under various drying conditions was investigated. The influence of activator type/dosage and pore structure on the shrinkage performance of AAS was elaborated.

2. Experimental programs

2.1. Materials

Grade 120 ground granulated blast-furnace slag with a density of 2.89 g/cm^3 and Type I portland cement with a density of 3.14 g/cm^3 were used in this study. The chemical compositions of slag and cement are shown in Table 1.

The mixture proportions of four AAS mortars and a control OPC mortar are listed in Table 2. All five mixtures were designed to have a constant volume-based liquid (water + activator) to solid (slag or cement) ratio. Because of the difference in density between slag and cement, as well as among various type/dosage of activator, the mass-based liquid-to-solid ratio varied from 0.489 to 0.510 for AAS. For OPC mixture, the mass-based water-to-cement ratio was 0.41. In addition, a natural river sand with a fineness modulus 2.60, oven dry specific gravity 2.52, and absorption capacity 2.02% was used.

Mortars AAS1 and AAS2 were activated by a combination of sodium hydroxide (NaOH) solution and aqueous sodium silicate (water glass) solution with different values of modulus n ($n = \text{SiO}_2/\text{Na}_2\text{O}$) in activator. Mortars AAS3 and AAS4 were activated by 2 M and 4 M NaOH solution, respectively. The pH, modulus, and density of activators are listed in Table 2.

2.2. Test procedures

2.2.1. Mixing

To prepare NaOH solution, reagent sodium hydroxide pellets were dissolved in distilled water at room temperature and sealed to prevent evaporation and carbonation. The aqueous sodium silicate solution was kept separately from NaOH solution. In order to dissolve the slag properly, NaOH solution was added first, followed by sodium silicate solutions after mixing for 15 min. The mixing was conducted in a Hobart mixer according to ASTM C305-12.

2.2.2. Drying shrinkage and mass change

A non-standardized mold with the dimensions of $12.7 \text{ mm} \times 12.7 \text{ mm} \times 139.7 \text{ mm}$ was designed to cast “mini-bar” prism samples. Mini-bars (see Fig. 1) were used to expedite drying shrinkage and allow samples to dry in Vena VC-10 environmental chambers. The chambers were capable of creating environments under various RH and/or gases (e.g., nitrogen purge). The PVC mold was able to be completely disassembled and had a gauge length of 101.6 mm (4 inches). The gauge studs used were one inch black oxide alloy steel cone point socket #6–32 set screws. Length measurements were taken utilizing a modified digital comparator with an invar (low coefficient of thermal expansion) place holder and a measuring precision of 0.0001 inches. Simultaneous mass measurements were taken using a balance with a precision of 0.0001 g to determine the moisture loss upon drying. The samples were positioned on a grid panel in the chambers to enable uniform drying with minimal restrains.

Two regimes of drying were investigated in this study.

2.2.2.1. Direct drying. Environmental chambers were set at four different RH (85%, 70%, 50%, and 30%) and constant temperature ($23 \pm 0.5 \text{ }^\circ\text{C}$). The drying shrinkage (direct drying) was measured for all five mixtures (four specimens per mixture) at each RH under dry nitrogen (N_2) purge after 7 days of moist curing (100% RH, $23 \pm 0.5 \text{ }^\circ\text{C}$). The concentration of CO_2 in environmental chambers was recorded with a portable CO_2 analyzer as 0 ppm. The measurement continued until the samples reached equilibrium, defined as both the mass and length changes of less than 2.0% over one week period.

2.2.2.2. Step-wise drying. After 7 days of moist curing, the specimens were sequentially dried at 85%, 70%, 50% and finally 30% RH under dry N_2 purge. The RH was dropped in each level when the samples reached equilibrium.

2.2.3. Elastic modulus

Elastic modulus represents the resistance of materials against elastic deformation when a force (e.g. capillary pressure) is applied, which can affect the shrinkage magnitude. The elastic modulus of all five mixtures was measured according to ASTM C469, and the detailed procedure was previously reported by Cartwright et al. [10].

2.2.4. Degree of saturation

The degree of saturation is an important parameter related to shrinkage since it represents the fraction of water-filled pores which may contribute to the capillary forces. In order to calculate the degree of saturation of samples conditioned at various RH (i.e. 100%, 85%, 70%, 50%, and 30%RH), the total amount of porosity (volume of permeable pore space) and moisture content at various RH should be known. The total amount of porosity of each mortar mixture before drying (after 7 days moisture curing) was measured according to ASTM C642-13, and the detailed procedure was documented by Cartwright et al. [10]. The moisture content of samples can be calculated based on the mass data during drying (Section 2.2.2) and oven-dried at $105 \text{ }^\circ\text{C}$ until constant mass was

Table 1
Chemical compositions (by mass %) of slag and portland cement.

	CaO	SiO ₂	Al ₂ O ₃	MgO	SO ₃	S ²⁻	Fe ₂ O ₃	Na ₂ O	K ₂ O	P ₂ O ₅	MnO	TiO ₂	LOI
Slag	43.83	30.04	12.74	4.79	3.11	0.85	1.16	0.24	0.40	0.08	0.22	–	2.56
Cement	62.5	19.9	5.44	2.31	4.93	–	2.26	0.30	0.89	0.23	0.09	0.29	0.86

Note: a: The element compositions of slag were measured by ICP-AES method, the relatively high SO₃ content in this slag is primarily due to the presence of gypsum. The composition of cement was provided by the supplier.

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