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# On drying shrinkage in alkali-activated concrete: Improving dimensional stability by aging or heat-curing

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## ARTICLE INFO

## Article history:

Received 8 April 2016

Received in revised form 1 October 2016

Accepted 5 October 2016

Available online xxx

## Keywords:

Alkali-activated concrete

Drying shrinkage

Autogenous shrinkage

Fly ash

Slag cement

## ABSTRACT

The problem of excessive drying shrinkage in alkali-activated concrete (AAC) is well-documented in the literature. The magnitude of drying shrinkage is often three or more times that in portland cement concrete. This study investigates the effects of binder type, activator concentration, strength, age, and curing method on the manifestation of drying shrinkage in alkali-activated fly ash and slag cement concrete. Early-age shrinkage strains in excess of  $1200 \mu\epsilon$  (0.12 percent strain) are observed in AAC. This is attributed to delayed hydration, microstructure refinement, and strength development. The resulting damage is far more significant than in portland cement concrete. Shrinkage and resulting damage are greatly reduced when specimens are dried at later age and after heat-curing. Alkali-activated slag cement concrete is more sensitive to water loss than portland cement or alkali-activated fly ash concrete. This results from a finer pore structure in alkali-activated slag binders.

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## 1. Introduction

Alkali-activated fly ash and slag cement concrete are promising sustainable alternatives to ordinary portland cement concrete (PCC). The improved sustainability of alkali-activated concrete (AAC) is based on: (1) the beneficiation of industrial byproducts which would otherwise be landfilled, and (2) the reduction of carbon emissions associated with portland cement production. Estimates for the reduction in carbon emissions associated with the use of alkali-activated or geopolymer binders in place of portland cement vary wildly from 9% [1] to 80% [2], but most studies estimate a reduction of at least 40% [2–7]. The reduction in emissions is not only dependent on the chemistry of the alkaline activator, but also on the availability of raw materials, fuel costs, and the efficiency of the clinkering operation in a particular geographical location. This provides some context for the lack of consensus within the literature.

Although alkaline activation of aluminosilicates is by no means a new technology [8–12], alkali-activated fly ash (AAF) and slag cement (AASC) binders have only recently emerged as viable alternatives to portland cement. A number of studies describe the activation chemistry, product formation, reaction kinetics, and mechanical

properties of AAF and AASC concrete [13–22]. Despite this, the long-term durability of AAC is not summarily proven [12,23–25]. Several questions remain with regard to the durability of AAC. These mainly relate to carbonation, chloride permeability, alkali-silica reactivity, resistance to chemical attack, and shrinkage. This study investigates the last.

### 1.1. Drying and autogenous shrinkage

Volume instability in hardened cement or concrete may occur due to carbonation, thermal effects, drying, or self-dessication. The latter two are of concern for the present study. Drying and autogenous shrinkage both result from the removal of pore water from within the hardened binder. The former is due to the loss of internal water to the external environment through evaporation. The latter is due to self-dessication, i.e., the consumption of pore water by the continuing hydration reaction. Only autogenous shrinkage can occur if the specimen is sealed to prevent moisture egress, but autogenous and drying shrinkage occur simultaneously if the specimen is unsealed.

Drying shrinkage is approximately proportional to water loss [26]. Since aggregates are highly dimensionally stable, shrinkage is dominant within the binder paste. An increase in binder content in concrete, which leads to an increase in the paste volume, naturally leads to an increase in drying shrinkage [27,28]. With sufficient shrinkage strains, cracking occurs. This can result in weakening of concrete structures, but the formation of cracks is more significant to the durability of concrete [28].

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Since autogenous shrinkage results from the consumption of water by the continuing hydration reaction, it is only of concern when the binder does not reach its full hydration potential. In the case of portland cement, complete hydration requires  $w/cm > 0.42$  [29–31]. Above this value, at which sufficient water is provided for complete hydration, autogenous shrinkage is theoretically nonexistent. It should be noted that there is some disagreement on the value of  $w/cm$  which results in complete hydration of portland cement [32,33], but this is of no consequence to the present study.

Three predominate mechanisms are known to contribute to drying shrinkage: Surface energy, capillary stress, and disjoining pressure [34–41]. The effects of surface energy (or surface tension) are explained by the Bangham equation and Gibb's free energy [35,40,42,43], which describe the shrinkage resulting from a reduction in surface energy due to the spreading of physically adsorbed water within the pore volume. Surface energy effects are significant below 45%RH [44]. Disjoining pressure arises from a cohesive interaction—mainly due to van der Waals forces—between the pore water and the surface within the pore volume [40,45–48]. Disjoining pressure is significant only above 45%RH [44].

Shrinkage induced by capillary stresses is a function of the volume of capillary pores, the capillary stress, and the compliance of the medium [49]. The volume of capillary pores in PCC is sensitive to  $w/cm$  and the degree of hydration [34,50,51]. The capillary pressure  $P_{cap}$  is related to the surface tension of the pore fluid  $\gamma$  and the pore radius  $r$  according to Eq. (1) [44,52,53].

$$P_{cap} = \frac{2\gamma}{r} \quad (1)$$

As the surface tension increases, so does the capillary pressure. Larger pores develop lower capillary stresses since the ratio of pore volume to surface area is lower. Capillary stresses are significant only above 45%RH due to instability of menisci below this humidity level [44].

### 1.2. Drying and autogenous shrinkage in AAC

The problem of excessive drying shrinkage strains in AASC is well documented [12,14,54–62]. Bakharev et al. [55] showed that curing at elevated temperature effectively reduced the magnitude of drying shrinkage strain in AASC concrete. Additions of gypsum, common air-entraining admixtures (AEA), and shrinkage-reducing admixtures (SRA) have also been shown to temper the magnitude of drying shrinkage in AASC [57,59]. Atiş et al. [61] found that the use of sodium carbonate as an activator instead of sodium silicate or sodium hydroxide also resulted in reduced shrinkage. Several studies have also demonstrated increased autogenous shrinkage in AASC relative to comparable portland cement [55,57,60,62].

In accordance with the previous discussion, the pore structure has a significant effect on drying shrinkage in concrete. Collins and Sanjayan [58] attributed the increased drying shrinkage in AASC concrete to a finer overall porosity. AASC binders are known to include a higher percentage of mesopores ( $2 \text{ nm} \leq r \leq 50 \text{ nm}$ ) than portland cement [58,63–65]. Shi [63] posited that sodium silicate-activated slag cement binders resulted in the finest pore structure of any AASC binders. Bakharev et al. [55] showed that the relatively localized hydration in heat-cured AASC resulted in a coarser porosity. Reduced drying shrinkage in heat-cured AASC concrete was attributed to this change in pore structure [55]. Furthermore, Palacios and Puertas [59] attributed reduced drying shrinkage in AASC with SRA to a coarsening of the pore structure and a reduction in pore fluid surface tension.

Comparatively few studies have discussed the manifestation of drying or autogenous shrinkage in AAF. Fernández-Jiménez et al. [66] reported that the drying shrinkage in AAF mortar was less than that

observed in comparable OPC mortar. Several studies have reported similar results for fly ash-based geopolymer binders [67,68], but geopolymers are chemically and mechanically distinct from AAF binders. In a study of the properties of alkali-activated blended fly ash/slag cement mortars, Chi and Huang [69] showed that a higher proportion of fly ash led to a decrease in the observed drying shrinkage strain. That same study also indicated that an increase in the dosage of sodium oxide in blended alkali-activated binders led to a decrease in the observed shrinkage [69].

The porosity and pore size distribution in alkali-activated binders has been studied somewhat intensely using micro-computed tomography ( $\mu$ CT), mercury intrusion porosimetry (MIP), Wood's metal intrusion porosimetry (WMIP), nitrogen sorption, water sorption, and microscopy; much of this research is neatly summarized by Provis et al. [70]. The high degree of heterogeneity and chemical complexity of alkali-activated binders results in a wide distribution of pore sizes, which can range from the order 10 nm to the order 10  $\mu$ m [70]. As a result of very small pore sizes and the ink-bottle effect, standard techniques like MIP, WMIP, and water sorption give incomplete information regarding the porosity and pore structure in alkali-activated binders [70–73]. More advanced techniques like  $\mu$ CT and nitrogen adsorption using Brunauer-Emmett-Teller (BET) theory [74] give more complete information.

Very high porosity has been observed in AAF binders [75–78], which are devoid of large capillary pores but include some very large cavities [78]. In comparison, the pore structure of AASC binders is much finer and more tortuous [70]. Some conflicting results have indicated only minor differences in the porosity and pore structure with curing time [79], but most studies suggest that the porosity and pore connectivity in AAC decrease with both curing time and curing temperature [70,78,80,81].

### 1.3. Research objectives

The problem of excessive drying shrinkage in AASC concrete is well-documented. However, there is limited information about how the activator concentration in AAC affects the susceptibility to drying shrinkage. The effect of heat curing on the manifestation of drying shrinkage has been documented to some extent, but the effect of age at drying has not. Finally, the damage that occurs as a result of drying shrinkage in AAC has not been fully investigated. This study seeks to address these gaps in knowledge.

## 2. Materials and methods

### 2.1. Materials and mixture proportions

The binder materials used in this study were class C fly ash meeting the specifications of ASTM C618, grade 100 ground granulated blast-furnace slag (slag cement) meeting the specifications of ASTM C989, and Type I ordinary portland cement (OPC) meeting the specifications of ASTM C150. The oxide composition of the fly ash and slag cement is listed in Table 1.

The activator was an aqueous sodium silicate solution ( $\text{Na}_2\text{O} + m \cdot \text{SiO}_2$ ). The silica modulus  $m$  is the mass ratio of silica to sodium oxide in the activating solution. Activator concentrations were specified by the silica modulus  $m$  and the dosage of sodium oxide in percent by mass of binder. The solution-to-binder ratio  $s/b$  is the mass ratio of activator solution (or water, in the case of OPC concrete) to binder. The solution-to-binder ratio  $s/b$  was 0.40 throughout. Activator concentrations are given in Table 2. The fine aggregate was a natural quartz sand. The specific gravity was 2.71 and the fineness modulus was 2.36. The coarse aggregate was a quarried crushed stone composed mainly of rose quartz. The nominal maximum aggregate size was 12.5 mm (0.5 in.). The binder content was 570  $\text{kg/m}^3$ , the coarse aggregate content was

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