



Autogenous shrinkage of alkali activated slag mortars: Basic mechanisms and mitigation methods



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ABSTRACT

This paper presents various mechanisms that can explain the cause of excessive autogenous shrinkage exhibited by alkali activated slag mortars (AASM) compared to Portland cement mortars (OPC). The influence of activator concentration parameters such as Na_2O content and of the silica modulus (M_s) on the magnitude of autogenous shrinkage are evaluated, as are the proposed mechanisms. The results show that the shrinkage kinetics of AASM is strongly dependent upon the rate of reaction, internal relative humidity (RH) and surface tension of the pore solution. Pore size variation between AASM and OPC mortars and the corresponding tensile stresses based on the capillary tension approach were also calculated using experimentally measured internal RH and surface tension values. A higher amount of both Na_2O and M_s resulted in larger capillary stress resulting in greater autogenous shrinkage. Use of internal curing and of shrinkage reducing admixture were very effective in reducing the autogenous shrinkage of AASMs.

1. Introduction

Shrinkage is the leading cause of volume instability and the associated formation of cracks in concrete structures. Shrinking of concrete is either due to the moisture lost to the environment or to self-desiccation. The latter induces autogenous shrinkage at early ages, resulting in high internal stresses and early age cracking [1], which further compromises the long-term durability performance such as resistance to corrosion, freezing and thawing, and alkali-silica reaction. Autogenous shrinkage is the macroscopic volume change occurring without moisture transfer to the exterior surrounding environment. Autogenous shrinkage occurs due to the development of tensile stresses when capillary water is extracted by unhydrated binder particles for hydration [2]. In Portland cement (OPC) systems, autogenous shrinkage is a result of chemical shrinkage affiliated with the hydration of the cement particles in concrete [3].

While autogenous shrinkage is a problem in all concrete with low water to cement ratio, it is particularly problematic in alkali activated slag concrete. Despite having adequate strength for typical construction purposes, alkali activated concrete is reported to have poor shrinkage performance compared to OPC concrete [4–8]. Alkali activated slag (AAS) concrete is prepared by mixing blast furnace slag (or the slag cement), an alkaline activation solution (the activator), and the normal fine and coarse aggregates [4]. The shrinkage strains mainly depend on the composition and fineness of the slag cement, the type and dosage of

alkali activator, and the curing conditions [6,9,10]. Autogenous shrinkage of AAS due to self-desiccation is directly linked to the decrease of total porosity and the increase of the volume of mesopores when the dosage of alkali activator (sodium silicate) increases. Additionally, as the dosage of the activator increases, the degree of reaction increases and the autogenous shrinkage attributed to chemical shrinkage also increases [11]. The drying shrinkage of alkali activated slag mortar (AASM) was determined to be 3 to 6 times that of OPC mortar depending on the activator dosage used [12,13]. Collins and Sanjayan related this higher drying shrinkage of AAS concrete to the finer pore size distribution [5]. Thomas et al. suggested various curing methods to mitigate the drying shrinkage [14]. The drying shrinkage has been widely studied, but a comprehensive understanding of the causes behind the high autogenous shrinkage behaviour of AAS is not yet well established.

Another problem yet to be addressed with AAS is the need to alleviate early age cracking due to the substantial autogenous shrinkage strains in AAS. Two common methods that have been used to mitigate the autogenous shrinkage in OPC concrete with low w/cm are (1) internal curing (IC), and (2) the use of shrinkage reducing admixtures (SRA). IC can provide water for curing throughout the cross section of the concrete when normal aggregate is partially replaced with saturated lightweight aggregates (LWA) [15]. An increase in internal relative humidity and consequent reduction in autogenous shrinkage can reduce early age cracking in OPC concrete [16–19]. Adding saturated LWA to

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engineered cementitious composites has been reported to reduce autogenous shrinkage by 67% and drying shrinkage by 37% at 28 days [20]. Another commonly used method to reduce the potential for shrinkage cracking is the use of shrinkage reducing admixtures (SRA) [21–26]. The addition of SRA can reduce the drying shrinkage of concrete by reducing the surface tension of the pore solution [27]. In high performance concrete, the addition of SRA was shown to reduce the capillary stresses that are generated by autogenous shrinkage [28]. However, the effect of SRA on the activated slag pastes and mortars has not been studied widely [29].

In order to be an effective replacement of OPC concrete, AAS concrete must show similar durability properties, including shrinkage properties. Although several researchers have quantified the magnitude of the autogenous shrinkage of AAS concrete, a comprehensive reasoning for the greater shrinkage, has not been reported before. Therefore, the main objective of this study is to characterize the autogenous shrinkage in AAS mortars and to elucidate the mechanism responsible for the same. The influence of activator dosage on the rate of reaction, surface tension of pore solution and internal relative humidity of AAS mixtures and their influence on the shrinkage behaviour are also investigated. Additionally, the effectiveness of internal curing and SRA addition in reducing the autogenous shrinkage of AAS mortars is studied.

2. Materials and methods

2.1. Materials and mixture proportions

The cementitious materials used in this study were ASTM C989 Grade 100 ground granulated blast furnace slag (GGBFS/slag cement) and Type I ordinary Portland cement (OPC). The oxide compositions of as-received raw materials are listed in Table 1. The alkaline activators used in this study were 8 M sodium hydroxide and an aqueous sodium silicate solution ($\text{Na}_2\text{O} + \text{Ms.SiO}_2$). The various concentrations were prepared using pre-mixed reagent grade sodium silicate, sodium hydroxide pellets and distilled water. The silica modulus Ms. refers to the mass ratio of silica to sodium oxide equivalent; the Na_2O concentration of the activator is reported relative to binder mass (% Na_2O , by mass of binder). The saturated surface dry (SSD) natural fine aggregate with specific gravity of 2.65 and fineness modulus of 2.42 was used. The sand to cementitious materials ratio used was 1.25. The mortar mixtures were designed in such a way to have a constant paste volume at 0.40 solution to binder ratio (s/b) among the different mixtures used in this study. An expanded shale light weight aggregate (LWA) with a maximum particle size of 4.75 mm and the absorption capacity of 18%, was used as the internal curing (IC) agent. A commonly used shrinkage reducing admixture (SRA) was also used.

An AAS mortar mixture with a s/b of 0.40, silica modulus of 1.5, and Na_2O content of 5% referred to as AAS1 in this study was used as the control mixture to compare the performance of AAS mixtures with LWA and SRA. The normal weight aggregate was replaced with 10, 15, 20, and 30% (by mass) LWA. The SRA dosage was varied from 2.5% to 7.5% by mass of binder material. The mixture proportions used are presented in Table 2. Typical behaviour of OPC mixtures were also compared with AAS mixtures. The s/b ratio was kept constant at 0.40 for all these mixtures. In order to find the effect of activator type, AAS mortar mixture activated with 8 M sodium hydroxide solution (AAS13) was also prepared and compared with sodium silicate activated mortar

Table 1
Oxide composition of the cementitious materials.

% by mass	CaO	SiO ₂	Al ₂ O ₃	MgO	Na ₂ O	Fe ₂ O ₃	SO ₃	K ₂ O
Slag cement	39.10	37.80	7.91	10.3	0.30	0.43	2.60	0.47
OPC	61.85	19.10	4.40	2.05	0.27	3.96	3.72	0.70

mixtures.

Activator solutions were prepared at least 24 h prior to casting to allow for complete dissolution and heat dissipation. Mixing of mortars was performed in the laboratory as per ASTM C192 specifications. The mortar mixture was then poured into corrugated tubes for shrinkage studies.

2.2. Autogenous shrinkage measurements

Linear autogenous shrinkage of AAS mortars was measured in accordance with ASTM C1698-09. This method prevents moisture loss effectively and minimizes the restraint to volume change during hardening. Slender corrugated plastic tube specimens with a length of approximately 420 mm and 29 mm in diameter were prepared for each mixture. The specimens were placed horizontally on corrugated plastic sheets to avoid any restraint to length change or damage. Change in length of the specimen was measured using a length comparator at room temperature ($21 \pm 2^\circ\text{C}$) starting at the point of final set as determined by the Vicat needle test. The values for setting time (in minutes) of all the mixtures are given in Table 2. The workability of all the mixtures was good enough to fill the corrugated tube within just a few minutes after the mixing was done. The flow measurements done on these mixtures were in the range of 55 to 115%. Stiffer mixtures were prepared by rodding as per the ASTM C1698-09. Three replicas were prepared for each mixture, and the length change was recorded frequently up to 28d and thereafter each month for one year.

2.3. Isothermal calorimetry

The heat of hydration was monitored using an isothermal conduction calorimeter (TAM AIR). Measurements of the heat of evolution were performed on paste specimens hydrated at 25°C isothermal condition. Since the s/b used in most of the cases was 0.40, to ensure complete mixing, mixing of the slag cement and the activator solution was done externally. Mixing of components was performed manually as the mass of the sample was relatively small. 5 g of paste were transferred to each ampoule. The ampoules containing sample were placed inside the calorimeter and the heat evolution data collection started after the thermal equilibrium was reached. Since the blending was done outside the calorimeter, the first exothermic peak was not recorded completely.

2.4. Thermogravimetric analysis (TGA)

The thermogravimetric analysis was performed using a Perkin Elmer Pyris TGA instrument. Specimens were cured for 1 day, crushed into small cubes, and soaked in isopropanol for 3 days to halt the hydration. Specimens were then ground to fine powders passing the 75 μm sieve and then vacuum desiccated for 1 day. Ground specimens weighing 25 ± 2 mg were heated at $10^\circ\text{C}/\text{min}$ from 30°C to 400°C in an alumina crucible under a nitrogen atmosphere with a flow rate of 40 mL/min.

2.5. Internal relative humidity

Internal relative humidity (RH) of AAS mortars was measured using in-situ RH sensor probes. Internal RH sensors were embedded in fresh mortar molds of 2×4 in. cylinders. The sensors were inserted to a depth of 2 in. from the top surface. The cylinders were cast with the sensors; the surface was immediately sealed using plastic caps. Internal RH measurements commenced immediately after setting time at a frequency of 1 h per measurement up to 24 h followed by one measurement every day over a period of 56 days. The sensors incorporated factory calibration to within $\pm 1.8\%$ RH ($\pm 3\%$ when over 90% RH). The average of two replicate measurements is reported.

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