



# Effect of $\text{Ca}(\text{OH})_2$ on shrinkage characteristics and microstructures of alkali-activated slag concrete

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## HIGHLIGHTS

- Use of  $\text{Ca}(\text{OH})_2$  coarsens AAS concrete pores.
- Autogenous and drying shrinkage were significantly reduced by adding  $\text{Ca}(\text{OH})_2$ .
- Ca-rich hydration products were found in AAS concrete with  $\text{Ca}(\text{OH})_2$ .
- $\text{Ca}(\text{OH})_2$  can accelerate the strength and elastic modulus development of AAS concrete.

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## ABSTRACT

The study presents the shrinkage behaviour of  $\text{Ca}(\text{OH})_2$ -added alkali-activated slag (AAS) concrete. A series of tests were performed to determine the plastic shrinkage, autogenous shrinkage and drying shrinkage. In order to explain shrinkage mechanisms, the moisture loss was monitored; the pore structure was examined by nitrogen adsorption test; hydration process was assessed by isothermal calorimetry; hydration products and microstructure were characterised by XRD, SEM/EDS and TG/DTG. It was found that the use of  $\text{Ca}(\text{OH})_2$  increased the plastic shrinkage of AAS concrete, but reduced autogenous and drying shrinkage. This is because the addition of  $\text{Ca}(\text{OH})_2$  accelerated the hydration at very early stage, while non-noticeable impact could be identified after 120 h. The results from nitrogen adsorption test showed a decrease in mesopores by adding  $\text{Ca}(\text{OH})_2$ , which was responsible for the less drying and autogenous shrinkage. Meanwhile, the Ca/Si of C-S-H (I) increased from 0.89 to around 1.40 at 28 d when adding 5% and 10%  $\text{Ca}(\text{OH})_2$ . XRD patterns indicated  $\text{C}_2\text{ASH}_8$  and  $\text{C}_4\text{AH}_{13}$  were found in  $\text{Ca}(\text{OH})_2$ -added samples. These changes in hydration products also contribute less drying and autogenous shrinkage in  $\text{Ca}(\text{OH})_2$ -added samples.

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## 1. Introduction

Reducing  $\text{CO}_2$  emission is a worldwide challenge that has to be overcome to protect environment. Around 5% of anthropogenic  $\text{CO}_2$  emissions were caused by the manufacture of Portland cement [1,2]. An alternative to Portland cement (PC) is highly desirable in order to reduce the relatively high carbon footprint of normal concrete [3–6]. One promising approach is to use alkali-activated slag (AAS), which offers the possibility of achieving significant reductions in greenhouse emissions.

Compared with PC, AAS has been found to have some excellent properties, e.g. rapid development of compressive strength [7], high resistance against sulfate [8] and acid attack [8,9]. However, some drawbacks of AAS concrete, such as high shrinkage [10], fast setting [11] and high rate of carbonation [12], have limited its application in practice.

High shrinkage causes a high risk of cracking, which provides additional paths for aggressive substances to move in and could accelerate corrosion of reinforcement in structures. From results in previous literatures [10–15], AAS concrete showed about 3 times higher drying shrinkage than PC concrete and the greater proportion of mesopores (1.25–25 nm) was believed to be one main reason for this behaviour [10,14,15]. Furthermore, hydration products of AAS cement were mainly C-A-S-H gel with a lower

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Ca/Si ratio [15], the structure of this type C-S-H gel was similar to that of 1.1 nm tobermorite, which has a poor water-retaining property [16]. Therefore, moisture inside AAS paste would be removed easily and rapidly, leading to high shrinkage. Numerous researches were carried out to study the drying shrinkage of AAS concrete [17–21,23]. Palacios et al. [17,18] and Bakharev et al. [19] found that shrinkage-reducing agent (SRA) could effectively reduce drying shrinkage and autogenous shrinkage of AAS concrete. Nano-MgO [20] and reactive MgO [21] showed an encouraging effect on controlling shrinkage of AAS, as MgO could decrease the amount of C-A-S-H gel and formed more hydrotalcite-like phase [22]. Yang [23] found that the drying shrinkage was reduced by adding nano-TiO<sub>2</sub> into AAS paste. Despite of these efforts, it is found that the AAS concrete shrinkage problem was still unavoidable [13]. Techniques to control shrinkage of AAS Concrete are still not fully developed yet.

One interesting finding reported by Aydin [25] and Gao et al. [26] shows that some drawbacks of AAS concrete, such as high carbonation and shrinkage, can be improved by using calcium-rich precursors [25,26]. This can be explained by three main reasons. The first one is that addition of Ca(OH)<sub>2</sub> may increase the Ca/Si ratio of hydration products in AAS concrete [30] that has a better water-retaining property [32]. Meanwhile, the redundant Ca(OH)<sub>2</sub> could be regarded as the supplement of crystal phases in AAS concrete which is helpful to reduce the shrinkage. Thirdly, the Ca(OH)<sub>2</sub> had an elastic modulus of 35.4 GPa according to Manzano's calculation [33], whereas the C-S-H gel generally exhibits an elastic modulus of 16–25 GPa. Thus, the redundant Ca(OH)<sub>2</sub> may improve the elastic modulus of AAS concrete and limit the shrinkage of AAS concrete.

To systematically investigate the role of Ca(OH)<sub>2</sub> on shrinkage characteristics of AAS concrete, this study examines shrinkage behaviours of AAS concrete with and without Ca(OH)<sub>2</sub>, along with general quality control properties including slump, setting time, compressive strength and static elastic modulus. The microstructures of AAS concrete and characteristics of its hydration products were assessed by BET, scanning electron microscope (SEM), X-ray diffraction (XRD), which are used to analyse the shrinkage mechanisms of AAS concrete.

## 2. Experimental details

### 2.1. Materials

Granulated blast-furnace slag (GGBS) used in this study was supplied by Chongqing Iron and Steel Company. The slag has a specific surface area of 435 m<sup>2</sup>/kg and its density is 2.95 g/cm<sup>3</sup>. The alkalinity coefficient ( $M_0 = \frac{\omega_{CaO} + \omega_{MgO}}{\omega_{SiO_2} + \omega_{Al_2O_3}}$ ) and activity coefficient ( $M_a = \frac{\omega_{Al_2O_3}}{\omega_{SiO_2}}$ ) of slag were 1.01 and 0.45 respectively.

Inorganic mineral admixture (a combination of clinker and chemicals: Chinese patent-CN1071401A) was added on a basis of 3% mass of slag in order to control the setting time of AAS concrete [24]. The density of clinker is 3.17 g/cm<sup>3</sup>, which was manufactured by Chongqing Runjiang Cement Plant. The chemical compositions of slag and PC clinker are summarised in Table 1.

Sodium silicate from Chongqing Jingkou Chemical Plant was used as the activator. The physical and chemical properties are given in Table 2. NaOH from Xinjiang Tianye Chemical Plant, Technical Pure, was used to adjust the modulus of water glass ( $M_s$ , defined as the molar ratio of SiO<sub>2</sub> to Na<sub>2</sub>O).

The calcium hydroxide was industrial lime powder and the composition was analysed by XRD, as shown in Fig. 1. Its density is 2.21 g/cm<sup>3</sup> and the specific area is 625 m<sup>2</sup>/kg.

The coarse aggregate used was crushed limestone with two grades (5–10 mm and 10–20 mm), mixed with a ratio of 4:6 by mass. The fine aggregate was manufactured sand with fineness modulus of 2.87 and the powder content was 7.44%.

### 2.2. Mix proportions of concrete

Table 3 provides the mix proportions of AAS concrete. The water to binder (slag) ratio (W/B) of AAS concrete was 0.45 and 0.52 respectively. Sodium silicate ( $M_s = 1.2$ ) was used as alkali activator with a constant concentration of 5.0% Na<sub>2</sub>O (by mass of slag). Ca(OH)<sub>2</sub> was added into AAS concrete to equally replace 5% or 10% of GGBS content.

The AAS paste with a W/B of 0.40 was used in isothermal calorimetry test, XRD analysis, TGA and SEM/EDXA, the paste contains the same content of Ca(OH)<sub>2</sub> as AAS concrete. Similarly, the activator of AAS paste was 5% (Na<sub>2</sub>O equivalent) sodium silicate with a modulus of 1.2 and water in sodium silicate was considered when the W/B of paste was calculated.

### 2.3. Testing methods

In order to present the experiments clearly, Table 4 summarises the testing parameters along with corresponding types of specimens, the details of which are listed in Sections 2.3.1–2.3.10.

#### 2.3.1. General properties

AAS concrete was mixed for 3 min in a double horizontal shaft forced mixer with a capacity of 100 L, the apparent density, initial slump and air content were immediately measured according to GB/T 50080-2002 [34]. Following these tests, concrete was cast into 100 mm cubic steel mould for compressive strength test and 100 mm × 100 mm × 400 mm for static elastic modulus test according to GB/T 50081-2002 [35].

#### 2.3.2. Plastic shrinkage

Plastic shrinkage was measured immediately after casting and the detailed testing process were described in Chinese standard GB/T 50080-2002 [34]. After mixing, the concrete was cast into the moulds in two layers and vibrated on a vibrating table to eliminate the air bubbles. Then the specimens were moved to a room with a constant temperature of 20 ± 2 °C and a relative humidity (RH) of 65%. Fig. 2 shows the test set-up, 100 mm × 100 mm × 515 mm steel moulds with two laser displacement sensors to measure the length changes along the longitudinal axis. To avoid the friction and seal joint, the inner sides of steel moulds were coated with Teflon tapes. Shrinkage was recorded every 10 min for 24 h. Each shrinkage value shown in this paper was an average of three replicates.

#### 2.3.3. Drying and autogenous shrinkage

Drying shrinkage was determined according to Chinese standard GB/T 50082-2009 [36]. AAS concrete was cast in 100 mm × 100 mm × 515 mm steel moulds using the same method as described in Section 2.3.2, then covered with cling film after casting to avoid the water evaporation. The concrete samples were demoulded after 1 day and cured in a standard curing room (20 ± 2 °C, 90 ± 5% RH) for 3 days. Then, the specimens were transferred into an environment-controlled room (20 ± 3 °C, 55 ± 5% RH) and a dial gauge was used to measure the linear dimension changes along the longitudinal axis [37].

Autogenous shrinkage test was performed under the same procedure with drying shrinkage test, while the specimens were sealed with the Teflon film immediately after demoulding.

#### 2.3.4. Mass loss of concrete at different stages

The moisture loss from concrete samples was recorded in parallel with studies of shrinkage at different stages. During the plastic shrinkage stage, the moisture loss was tested using 100 mm cubic plastic mould with one 100 mm × 100 mm surface exposed for evaporation. The results were transferred into water evaporation (g) per unit (m<sup>2</sup>). The test of fresh concrete can only be conducted using one exposure surface and hence, the mass loss changes were not be calculated as percentage of concrete due to the asymmetric (one-surface) evaporation. Additionally, this condition is closer to the status that plastic shrinkage happens (one-surface evaporation as shown in Fig. 2).

For the drying shrinkage stage, the mass change was determined using 100 mm concrete cubes and the mass loss was recorded as percent of concrete weight.

**Table 1**  
Chemical compositions of slag and clinker (wt.%).

Materials	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	Loss
Slag	31.63	13.42	1.32	9.12	36.35	0.34	0.46	0.61
Clinker	19.99	4.80	2.98	3.27	61.22	0.18	0.88	3.52

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