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Hydration products, internal relative humidity and drying shrinkage of alkali activated slag mortar with expansion agents



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HIGHLIGHTS

• Effects of different admixtures on drying shrinkage and IRH of AAS were evaluated.

• CE with more CaO was found to have better effect than CSAE with more sulphoaluminate.

• Linear relationship between drying shrinkage and IRH was found.

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ABSTRACT

In this paper, two types of composite expansion agents, CSAE (calcium sulphoaluminate type expansion agent) with more calcium sulphoaluminate than CaO, and CE (CaO type expansion agent) with more CaO than calcium sulphoaluminate, were used to improve the drying shrinkage of AAS (alkali activated slag) system, separately. For comparison, SRA (shrinkage reducing agent) was used for the same purpose as well. Results showed that the addition of CSAE, CE and SRA in AAS mortar reduced the drying shrinkage by about 41–45%, 54–56% and 35–44% at 56 d, respectively. At late ages, the IRHs in the three groups with admixtures were all higher than the reference group, though the addition of CSAE, CE showed a better effect in drying shrinkage control due to the comprehensive effect of both low water consumption of CaO hydration and the high elastic modulus of CH in hydration products. It is found that drying shrinkage to IRH in AAS, and the incorporation of the admixtures reduced the sensitivity of drying shrinkage to IRH in AAS mortar.

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1. Introduction

Alkali activated slag (AAS) is a kind of cementitious material which has attracted many researchers for its lower energy consumption and lower CO₂ emission in the manufacturing process when compared to Portland cement. Previous studies indicate that AAS concrete possesses high early strength and good resistance to chemical attack, frost-thaw cycles and high temperature [1–7], which are superior to the performance of Portland cement concrete. However, the high drying shrinkage of AAS [8–11] is a critical factor that restricts its application. The drying shrinkage of NaOH-activated AAS paste is about 4 times higher than that of Portland cement paste at 60 days [12]. The AAS mortar generally shows a drying shrinkage 2–4 times higher [11,13,14] than that of PC

mortar and the shrinkage value can reach to about 2200 $\mu\epsilon$. The investigation about AAS concrete [8] also found that the drying shrinkage of AAS concrete is 3 times higher than that of Portland cement concrete.

Compared to Portland cement, more refined pores can be found in AAS and can result in higher capillary stress [10,12,15–18], which can considerably increase the shrinkage at drying condition. In addition to the pore structure, the hydration product of AAS is also one of the key factors that contributes to the high drying shrinkage of AAS [11,12,19]. The main hydration product in AAS is C-A-S-H, which is more amorphous than the C-S-H in Portland cement system [19]. The investigations from Ye [11,12] have explained the high drying shrinkage of AAS by understanding the behavior of hydration products at drying process. According to their research, the structural incorporation of alkali cations in C-A-S-H reduces the stacking regularity of C-A-S-H layers and make the C-A-S-H easier to collapse and redistribute upon drying, and the high drying shrinkage of AAS can be attributed to the relatively low creep modulus of the hydration products in AAS. The research of Ye help to illuminate the intrinsic reasons of the high drying shrinkage of AAS.

Some researchers have investigated the possible ways to reduce the shrinkage of AAS [14,20–23]. Yang [23] found the addition of nano-TiO₂ can help to decrease the volume fraction of pores between 1.25 and 25 nm which is responsible for the high shrinkage of AAS. T. Bakharev [20] studied the effect of different admixtures on the properties of AAS and found that the addition of gypsum reduced the shrinkage of AAS due to the compensating effect of AFt and AFm phases generated in the reaction related to gypsum. M. Palacios [14] studied the effect of shrinkage-reducing agents (SRA) on the shrinkage of AAS mortars and pastes, and the results showed that the addition of SRA reduced shrinkage in AAS mortars by up to 85% and 50%, when cured at 99% RH and 50% RH, respectively.

According to [24-27], the development of internal relative humidity (IRH) in cement-based materials has a certain relationship with shrinkage. Z.C. Grasley and D.A. Lange [26] have used an IRH measurement system to quantify the moisture gradient in early-age Portland cement concrete exposed to drying condition, and they developed a model to estimate the stress gradient in Portland cement concrete. Z. Jiang [24] studied the relationship between IRH and autogenous shrinkage of high-performance cement pastes, and the results showed that the IRH and autogenous shrinkage of high-performance cement pastes had a good linear relationship. D.P. Bentz [27] studied the effect of SRA on the IRH of cement paste and found that the addition of SRA caused a decrease in the IRH reduction and a significant reduction in autogenous shrinkage in low w/c ratio cement pastes and mortars cured under sealed conditions. C. Song [28] studied the effect of superabsorbent polymers on IRH and autogenous shrinkage of alkali-activated slag mortars and found that the increasing dosage of superabsorbent polymers in the specimens could help to increase the IRH and decrease the shrinkage. In his investigation, he found that the relationship between IRH and shrinkage in AAS was linear up to about 7 days.

It is known that calcium sulphoaluminate type expansion agent (hereafter called CSAE) [29], CaO type expansion agent (hereafter called CE) [30] and shrinkage-reducing agent (hereafter called SRA) [31,32] are admixtures which have been widely applied to Portland cement system to control shrinkage. Although these admixtures have shown significant positive effect in Portland cement system, the situation in AAS system is different, therefore the effect and mechanism of these admixtures on AAS needs to be clarified.

In this study, CSAE and CE were added into AAS mortar to see if these traditional expansion agents could have effect on AAS system. For comparison, SRA was also used to control the drying shrinkage of AAS mortar. Drying shrinkage and IRH in AAS system were tested in order to understand the effect of these admixtures on the relationship between drying shrinkage and IRH.

2. Experimental

2.1. Raw materials

GGBFS with a density of 2.87 g/cm³ and specific surface area of 431 m²/kg was used. X-ray fluorescence spectrometer (XRF) was applied to obtain the chemical composition of solids. The chemical composition of slag is summarized in Table 1.

CSAE and CE agent, commercially available expansion agents obtained by mechanical blending of various constituents, are in fact the composites with different content of calcium sulphoaluminate and CaO as main components, respectively. From Table 2, the calculated mass proportion of CaO and CaSO₄ in CSAE agent are 34.8% and 41.2%, respectively, while in CE agent, 57.0% and 22.9%, respectively. Compared to CE type agent, there are more CaSO₄ in CSAE agent. Alkyl polyether type SRA was used for comparison. The Alkyl polyether type SRA can reduce the

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Composition	of slag	(wt%)

	SiO ₂	Al_2O_3	CaO	Na ₂ O	MgO	SO ₃
Slag	31.95	14.0	37.24	5.98	6.57	2.36

Table 2

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Composition of expansion agent (wt%).

Туре	SiO ₂	Al_2O_3	CaO	MgO	SO ₃	Fe ₂ O ₃
CSAE	3.86	8.71	51.87	3.94	24.27	0.48
CE	3.28	7.86	66.54	4.86	13.51	1.22

Table 3

Mix proportion (in mass) of mortars.

Sample	Modulus	Na2O (%)	Water to slag ratio	Sand to slag ratio	Dosage of admixtures (%)
Ref	1.2	4.0	0.4	2	0
CSAE-6	1.2	4.0	0.4	2	6
CSAE-8	1.2	4.0	0.4	2	8
CSAE-10	1.2	4.0	0.4	2	10
CSAE-12	1.2	4.0	0.4	2	12
CE-6	1.2	4.0	0.4	2	6
CE-8	1.2	4.0	0.4	2	8
CE-10	1.2	4.0	0.4	2	10
SRA-1	1.2	4.0	0.4	2	1
SRA-2	1.2	4.0	0.4	2	2
SRA-3	1.2	4.0	0.4	2	3



Fig. 1. Effects of CSAE, CE and SRA on compressive strength of AAS mortar.

surface tension of pore solution by up to 52% [33]. These three types of admixtures were supplied by Jiangsu Subote new materials corporation in China.

The alkali activator was a mixture of solid NaOH and liquid water glass. The water glass was composed of 9.7 wt% Na₂O, 26.2 wt% SiO₂, and 50.0 wt% water. Solid NaOH was used to adjust the modulus (molar ratio of Na₂O/SiO₂) of the activator to a specific value. The mix proportions of alkali activated slag mortars are listed in Table 3. For each group, the water to slag ratio was 0.4, the sand to slag ratio was 2, the modulus (molar ratio of Na₂O/SiO₂) of the activator to f Na₂O was 4.0%. The only difference between each AAS group was the type and dosage of admixtures. The dosage of admixtures was determined according to the recommendation of the supplier. The number behind the admixture in Table 3 represents the dosage of this kind of admixture (percentage by mass of slag).

2.2. Test methods

2.2.1. Compressive strength

 $40~mm\times40~mm\times160~mm$ AAS mortar prisms were prepared for the compressive strength test. The specimens were demolded after 24 h and then cured in a

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