



Effect of alkali dosage on alkali-silica reaction in sodium hydroxide activated slag mortars



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HIGHLIGHTS

- ASR expansion decreases with increasing the alkali dosage for the studied mortars.
- Higher Al concentration mitigates the dissolution of reactive silica from the sand.
- The external OH⁻ concentration is relatively lower than that of the pore solution.
- No major difference in ASR products for the mortars with different alkali dosages.

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ABSTRACT

Alkali-silica reaction (ASR) is one of the durability factors limiting the commercial use of alkali-activated slag (AAS) concrete. The effect of alkali dosage is of great importance and barely studied for ASR in AAS system. This study investigates the effect of alkali dosage on ASR in sodium hydroxide (NaOH) activated slag mortars. In addition to ASR expansion, the changes in sample weight, compressive strength, pore structure, and pore solution chemistry are also investigated for better understanding the factors controlling the ASR in AAS mortars. The present study shows that the pore solution alkalinity increases with increasing the alkali dosage of the activator. However, the ASR expansion decreases with increasing the alkali dosage. The SEM/EDX results show no significant difference in both morphology and chemical composition of the ASR products. The lower ASR expansion at higher alkali dosage may be attributed to the relatively higher concentration of aluminum in the pore solution due to higher degree of slag hydration, which is expected to mitigate the dissolution of silica from the reactive aggregate.

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1. Introduction

Alkali-activated slag (AAS) representing one of the alternatives to Portland cements has received considerable attentions since it provides not only high compressive strength for concrete but also lower environmental impact compared to Portland cement [1]. The AAS is generally produced by reaction between slag and alkali activators such as sodium hydroxide, sodium carbonate and sodium silicate etc., which are usually pre-dissolved in the mixing water [2]. The major part of the previous studies on this type of materials has focused on the mechanical properties and hydration [3–6], whereas relatively few studies focus on their durability that limiting the use of these materials [7].

Alkali-silica reaction (ASR) takes place between hydroxyl ions in concrete pore solution and reactive silica in aggregates. The formation of the ASR products can cause expansion and cracking of concrete. It is one of the key durability issues that care must be taken prior to industrial application for the AAS concretes, since the high alkaline dosage is suspected to promote ASR when AAS is used together with alkali reactive aggregates. Several studies [8–11] have shown that AAS systems perform better than Portland cement systems under accelerated ASR. Nevertheless, Bakharev et al. [12] reported that AAS concrete had lower resistance to alkali-aggregate attack than that of ordinary Portland cement concrete of similar grade. Thus, it is unclear that whether AAS concrete has lower potential to ASR in field condition. Shi et al. [13] have recently reviewed the previous studies on ASR in alkali-activated systems showing that ASR in alkali-activated materials is affected by many factors, such as the nature and dosage of activators, binders, reactive aggregates, testing methods. However, there is only

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few studies focusing on the impact of alkali dosages on ASR in alkali-activated systems [14,15]. It is not clear that how the alkali content affects the ASR in these systems, although a consistent conclusion of this impact has been made for Portland cement system where the ASR expansion usually increases with increasing alkali content [16]. Understanding this impact is particular important for the alkali-activated system due to its intrinsically high alkali content compared to Portland cement system.

The aim of the present study is to investigate the impact of alkali dosages on ASR in sodium hydroxide activated slag mortars. The accelerated mortar bar test following the procedure described in ASTM C1260 [17] was conducted to determine the ASR expansion of the mortars. The weight change as a result of ASR was also monitored along with the expansion measurements. The impact of ASR on changes in compressive strength and pore structure was investigated before and after exposure. The reaction products were examined by the SEM/EDX to confirm the occurrence of ASR. The pore solution chemistry was analyzed to explore the key factor(s) that controlling the ASR in the studied mortars.

2. Experimental

2.1. Raw materials

A granulated blast-furnace slag (SL) was used in this study as binder material. The chemical composition of the slag was determined by X-ray fluorescence and is given in Table 1. An analytical grade sodium hydroxide (NaOH, purity 99% ± 1) was used as alkali activator. The fine aggregate used for preparing mortars was natural sand from Xiangjiang River, Hunan, China, which contained significant fraction of reactive siliceous competent, i.e., cryptocrystalline and microcrystalline quartz in greywacke fragments as reported in the previous study [18]. The sand was sieved to the particle size distribution as stipulated in ASTM C 1260 [17].

2.2. Mortar preparation

Alkali activated slag (AAS) mortar specimens were prepared with one part of slag activated by NaOH and 2.25 parts of graded sand by mass at water-to-slag ratio of 0.47. The dosage of the alkali activator (i.e., NaOH) contained 4, 6 and 8 wt.% Na₂O by mass of slag. The activator was pre-dissolved in a fraction of the mixing water before mixing. After casting, the mortar specimens were stored in a standard curing room at 20 ± 2 °C with relative humidity (RH) above 95% for 24 h. Finally, the specimens were carefully demolded and subsequently steam cured in an incubator at 80 °C for 22 h. Mortars with different alkali dosages were labeled as AAS4, AAS6 and AAS8, with numbers represent the weight percentage of Na₂O.

2.3. Accelerated mortar bar testing

The accelerated mortar bar testing was performed following the procedure described in ASTM C1260 [17]. Three mortar specimens (25 mm × 25 mm × 285 mm) for each mix were submerged in a 1 mol/L NaOH solution in a sealed plastic container after curing. All the mortars were then stored in an incubator at 80 °C. After 2 h of NaOH solution exposure, the initial lengths of the mortar specimens were measured using a digital length comparator. Subsequent lengths of the specimens were measured at 1, 3, 5, 7, 10, 14 and 28 days of exposure. Each reported expansion was an average of measurements from three specimens. The weight of the mortar specimens was also measured, and this was done immediately after the length measurements for each three specimens.

2.4. Compressive strength

The compressive strengths were determined on the additional mortar specimens prepared with larger size (40 mm × 40 mm × 160 mm), which were cured and exposed under the same conditions as used for the specimens for the accelerated mortar bar testing. The initial compressive strength was determined after steam curing. Additional measurements were performed after 14 and 28 days of exposure.

2.5. Pore structure

The pore structure after steam curing and 28 days of NaOH solution exposure was characterized by Mercury Intrusion Porosimetry (MIP). The samples were taken from the center of the specimens and dried in a vacuumed incubator at 60 °C for 2 days. The measurement was performed following the procedure described in ASTM C642 [19].

2.6. Elemental concentration of the extraction solution

The concentration of hydroxyl ion in the extraction solution was determined after 0, 1, 7, 14 and 28 days of exposure following the procedure described in literature [20]. Mortars were ground and passed through 0.30 mm sieve. Powered samples were collected and mixed with distilled water (50 g solid plus 50 g liquid) in sealed plastic bottles. The mixtures were stirred regularly for 3 days at 20 °C and then filtered. About 10 mL solution for each sample was collected and titrated with 0.1 mol/L HCl solution to determine the OH[−] concentration. The data can be used as an indication of alkalinity of the pore solution with its applicability has been confirmed [20]. The Ca, Si and Al of the extraction solutions obtained from mortars after 28 days of exposure were determined by ICP-AES.

2.7. SEM/EDX examination

The ASR products were examined after 28 days of exposure by a scanning electron microscopy equipped with energy-dispersive X-ray analyze (SEM/EDX). The samples used were cut off into slices with thickness of 10 mm and submerged in isopropanol for 3 days. The samples were then dried in a vacuumed incubator at 60 °C for 2 days prior to SEM/EDX examination.

3. Results and discussion

3.1. ASR expansion

The ASR expansions of the alkali-activated slag (AAS) mortars containing different alkali dosage (4, 6 and 8 wt.% Na₂O) are shown in Fig. 1. The results show that the ASR expansions for all mortars increase with increasing exposure time. All the measured ASR expansions within the testing period are below 0.1%, which is stipulated as criteria in ASTM C1260 [17] for evaluation of the risk of ASR in Portland cement mortars containing alkali reactive aggregates. Compare with previous study, it can be seen that the expansions of the NaOH-activated AAS mortars are lower than the expansion of the Portland cement mortar containing the same aggregate [18]. This indicates that the NaOH-activated AAS mortars performed better than the Portland cement mortar under accelerated mortar bar testing condition. This observation is in line with most of published studies as reviewed by Shi et al. [13]. The authors [18] also demonstrated that lower ASR expansion was related to a lower pore alkalinity for mortars exposed to 1 mol/L NaOH solution. However, it should be noted that the risk of ASR may be higher for AAS mortars under natural condition due to the high alkalinity of the pore solution of AAS mortars [18]. They also showed detectable ASR expansion for AAS mortar under steam curing condition while no expansion for Portland cement mortar was observed up to 56 days [18]. Therefore, care still need to be taken when the alkali-activated binders are used together with alkali reactive aggregate. The discrepancy of the ASR expansion and pore alkalinity observed between steam and alkaline exposure conditions suggest that the accelerated mortar bar test may not be suitable for comparison of the risk of ASR between AAS mortars and Portland cement mortar. This because the main source of alkalis in accelerated mortar bar test is from the highly alkaline exposure solution. The availability of alkalis to react with reactive silica is dependent on the transport of these alkalis through the

Table 1
Chemical composition of slag powder (wt.%).

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃
SL	33.00	13.91	0.82	39.11	10.04	1.91	–	0.16

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