



Alkali-silica reaction in waterglass-activated slag mortars incorporating fly ash and metakaolin



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ABSTRACT

Mitigation of alkali-silica reaction (ASR) in concrete is essential for durable and sustainable constructions. In conventional Portland cement-based concrete, alumina-rich supplementary cementitious materials such as low-calcium fly ash and metakaolin have been successfully used to mitigate ASR. Such effect has not been investigated for alkali-activated slag based materials. In this study, ASR expansion, changes in mortar weight and pore solution alkalinity have been studied. XRD and SEM/EDX have been employed to examine the reaction products and degradation of aggregates. The results demonstrate that there is an optimum dosage of fly ash to reduce ASR expansion of the studied mortars, whereas ASR expansion decreases with increasing the amount of metakaolin. It is found that the amount of ASR product formed is minor in the studied mortars with highest expansion. The pore solution alkalinity of the studied mortars is the main factor which controls the ASR in the alkali-activated slag mortars.

1. Introduction

With the aim to meet the increasing cement demand and to lower the CO₂ emission associated with Portland cement clinker production, development of low environmental impact materials to replace conventional Portland cement is emerging during the past decades [1]. Alkali activated cements (AACs) are generally produced by mixing industrial by-products (e.g. fly ash and slag) and/or calcined clays (e.g. metakaolin) directly with alkaline solutions (e.g. waterglass, NaOH, Na₂CO₃ solutions etc.) [2]. They have received considerable research interests since no clinkering process is involved in the production. The resulting mortars and concretes have demonstrated some obvious advantages compared to Portland cement-based materials, such as rapid development of compressive strength and high resistance in aggressive environments [3]. However, a potential concern has been raised on the use of AACs together with alkali-reactive aggregates, since the high alkalinity required for the activation of the binders may result in alkali-silica reaction (ASR), possibly causing deleterious expansion [4].

Although numerous studies in laboratory and field conditions have focused on ASR in Portland cement-based systems after its discovery in 1940's [5], few studies have focused on ASR in alkali-activated systems.

A major debate is that whether ASR is possible to take place in alkali-activated systems, on the other hand many studies have shown that ASR did exist under accelerated conditions (i.e., elevated temperature and high alkali concentration of exposure solution), but in a less severe form than that in Portland cement systems as reviewed by Shi et al. [4]. In order to eliminate the impact of external alkalis, Shi et al. [6] investigated ASR in alkali-activated slag mortars under steam and water curing conditions at 80 °C. They confirmed that the occurrence of ASR in these mortars was due to presence of alkalis used for alkali activation. Despite an initially high alkalinity, many studies attributed the lower ASR expansion of AAM systems to the rapidly decreased pore solution alkalinity over time as a result of alkali activation. However, Shi et al. [6] showed that the alkalinity of pore solution of the AAM mortars was higher than that of the Portland cement mortars when they were exposed to water and steam curing conditions at 80 °C for 56 days, whereas the alkalinity of pore solution of the AAM mortars was lower than that of the Portland cement mortars after exposure to 1 mol/L NaOH solution. The higher pore solution alkalinity in the Portland cement mortars may be explained by the presence of coarser pores in Portland cement mortars than in AAM mortars [2].

Mitigation of ASR by incorporating alumina-rich supplementary

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cementitious materials (SCMs, e.g. fly ash, slag and metakaolin) has been widely studied in Portland cement systems [7,8]. The most plausible mechanism for the mitigation of ASR is related to high aluminum concentration in pore solution, which prevents the dissolution of reactive silica from aggregates [9,10]. In addition, low calcium concentration is also expected to be beneficial for mitigating ASR, as it may prevent gelation of silica [11]. As such, the ASR products may be innocuous due to low yield strength and viscosity [7]. Shi et al. [12] recently studied the effect of alkali dosage on ASR in NaOH-activated slag mortars using an accelerated mortar bar test. It was surprising that the ASR expansion of the studied mortars decreased with increasing the alkali dosage, contradicting to the trends normally observed in Portland cement based materials [7]. The authors attributed the lower ASR expansion at higher alkali dosage to higher aluminum concentration in pore solution generated by higher reaction extent of slag at higher alkali dosage [12]. Based on these findings and in light of the difference in chemical compositions (i.e., Al and Ca content) of different SCMs, it is expected that the behavior of ASR in alkali-activated slag (AAS) may be different from that in alkali activated low-calcium fly ash (AAFA) and alkali activated metakaolin (AAMK), i.e., the ASR expansion is expected to be lower in AAFA and AAMK than in pure AAS based materials. Although ASR has been investigated in AAS, AAFA and AAMK materials [4,13], ASR in alkali activated blended binders is still poorly understood.

In the present study, slag (SL) has been partially replaced by low-calcium fly ash (FA) or metakaolin (MK) in AACs. Given the higher Al and lower Ca contents in the low-calcium FA and MK, the aim of the present study is to investigate the possible mitigating effects of FA and MK on ASR in waterglass-activated slag mortars. The accelerated mortar bar test following the procedure described in ASTM C1260 [14] has been conducted to determine the ASR expansion of the mortars. The mass change of the studied mortars after exposure to 1 mol/L NaOH solution has also been monitored along with the expansion measurements. The occurrence of ASR has been examined by analyzing the reaction products using a scanning electron microscopy equipped with energy-dispersive X-ray analyzer (SEM/EDX). The pore solution chemistry has been analyzed to explore the main factor(s) controlling the ASR in the studied mortars.

2. Experimental

2.1. Raw materials

A granulated blast-furnace slag (SL), low-calcium fly ash (FA) and metakaolin (MK) were used in this study as cementing components. The chemical composition of these materials was determined by X-ray fluorescence and is given in Table 1. The densities of these materials are 2900 kg/m³ (SL), 3100 kg/m³ (FA) and 2750 kg/m³ (MK), respectively. The activator used was waterglass with a silicate modulus ($M_s = \text{SiO}_2/\text{Na}_2\text{O}$ by mass) of 1.5. It was prepared by adding analytical grade sodium hydroxide (NaOH, purity 99% ± 1) into an industry grade waterglass with M_s of 3.3 (water content of 65.2 wt%). The fine aggregates used for mortars were natural sand with density of 2530 kg/m³ from Xiangjiang River, Hunan, China. The mineralogy and microstructure of the sand were documented in previous studies [15,16], which showed that the aggregates were not porous but contained some flaws which is expected to allow hydroxide ions to penetrate into the aggregate. The

Table 1
Chemical compositions of the binder materials (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
FA	54.29	22.55	5.53	1.34	1.08	1.49	0.75	0.29
MK	53.92	41.63	1.33	0.68	0.41	0.34	0.49	–

aggregates contained a significant fraction of reactive siliceous components, i.e., cryptocrystalline and microcrystalline quartz in grey-wacke fragments [15,16]. The sand was sieved and remixed to the particle size distribution according to ASTM C1260 [14].

2.2. Mortar preparation

Alkali-activated mortar specimens (25 mm × 25 mm × 285 mm) were prepared with one part of waterglass activated binders and 2.25 parts of graded sand by mass at a water-to-binder ratio of 0.47. The cementing components were prepared by replacing slag with 0, 10, 30, 50 and 70 wt% of FA or MK. The components were activated using prepared waterglass solution containing 6 wt% Na₂O by mass of the components. The activator was pre-mixed with mixing water before being added to the dry components. The binders were mixed and then cast into 25 mm × 25 mm × 285 mm molds. After casting, the mortar specimens with molds were stored in a standard curing room at 20 ± 2 °C with relatively humidity (RH) above 95% for 24 h. Then, the specimens were carefully demolded and subsequently steam cured in a chamber at 80 °C for 22 h. Mortars were labeled as SL, FA10, FA30, FA50, FA70, MK10, MK30, MK50 and MK70, with numbers representing the percentage of replacement levels of the SL by FA or MK.

2.3. Accelerated mortar bar test

The accelerated mortar bar test was performed following the procedure described in ASTM C1260 [14]. Three mortar specimens for each mixture were submerged in 1 mol/L NaOH solution in a sealed plastic container then stored in a chamber at 80 °C following the curing procedure described in the previous section. After 2 h of exposure in the NaOH solution, the initial length of the mortar specimen was measured using a digital length comparator. Subsequent lengths of the specimens were measured after 1, 3, 5, 7, 10, 14, 21 and 28 days of exposure. Each reported expansion value was an average of measurements from three specimens.

2.4. Mass gain

The mass of the mortar specimen ($M_{\text{mortar}, t}$) was determined by weighing the specimen immediately after the length measurements for each three specimens, where t represents the exposure time, i.e., $t = 0, 1, 5, 7, 10, 14, 21$ and 28 day. The paste specimens with the same size (25 mm × 25 mm × 285 mm) and water-to-binder ratio to mortar specimens were also prepared and exposed to the same conditions. The mass of the paste specimen ($M_{\text{paste}, t}$) was determined after the same exposure time (t) compared to mortar specimens. The mass gain of the mortar ($\Delta M_{\text{mortar}} = M_{\text{mortar}, t} - M_{\text{mortar}, 0}$) and paste ($\Delta M_{\text{paste}} = M_{\text{paste}, t} - M_{\text{paste}, 0}$) specimens is reported in gram per volume of the starting specimens (g/cm³). In order to eliminate the influence of secondary alkali activation due to ingress of alkaline solution on mass gain of the studied mortars, the net mass gain as a result of alkali-silica reaction (ΔM_{ASR}) is roughly estimated by subtracting the mass gain of pastes in mortar specimens from the mass gain of the mortar specimens as follows: $\Delta M_{\text{ASR}} = \Delta M_{\text{mortar}} - \varphi \Delta M_{\text{paste}}$, where φ is the volume percentage of paste in mortar specimens that can be calculated based on the mortar constituents and densities of the raw materials.

2.5. Alkalinity of the extracted solution

The concentration of hydroxyl ion (OH⁻) in the extracted solution was determined after 0, 1, 7, 14 and 28 days of exposure following the procedure described in ref. [17]. Mortars were firstly surface dried using wet cloth, then ground and passed through 0.30 mm sieve as fast as possible to minimize the potential risk of carbonation. A powered sample of 50 g was collected and mixed with 50 g distilled water in sealed plastic bottles. The bottles were rotated for 3 days at 20 °C and

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