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Effect of alkali dosage and silicate modulus on carbonation of alkali-activated slag mortars

Zhenguo Shi^{a,b}, Caijun Shi^{a,*}, Shu Wan^a, Ning Li^a, Zuhua Zhang^{a,c,*}

^a Key Laboratory for Green & Advanced Civil Engineering Materials and Application Technology of Hunan Province, College of Civil Engineering, Hunan University, Changsha 410082, China

^b Laboratory for Concrete & Construction Chemistry, Swiss Federal Laboratories for Materials Science and Technology (Empa), 8600 Dübendorf, Switzerland

^c Centre for Future Materials, University of Southern Queensland, Toowoomba, QLD 4350, Australia

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ABSTRACT

The long-term durability and their mechanisms of alkali-activated cement based materials have remained largely elusive. In this paper, carbonation of alkali-activated slag (AAS) mortars activated by NaOH and waterglass with different alkali dosages and silicate moduli has been investigated after exposure to $3 \pm 0.2\%$ (v/v) CO₂ at $20 \pm 2^\circ\text{C}/65 \pm 5\%$ RH for 56 days. The results show that carbonation resistance of the AAS mortars increases with increase of not only alkali dosage but also silicate modulus. In addition to the higher pore solution alkalinity and slag reaction extent, the relatively higher carbonation resistance of the AAS mortars is attributed to the lower porosity and average pore size. The loss of compressive strength for the waterglass activated slag mortars after carbonation is due to decalcification of C-A-S-H phase, whereas the carbonation of katoite contributes to the increase of compressive strength of the NaOH activated slag mortars.

1. Introduction

Development of clinker-free binder materials is a promising strategy to produce sustainable concrete, by which CO₂ emissions associated with Portland cement clinker production can be significantly reduced [1]. The use of granulated blast furnace slag (GBFS), an industrial by-product, for the purpose of producing alkali-activated cement concretes has been developed for many decades. Considerable number of studies on their mechanical properties and activation reaction kinetics have been conducted [2,3]. However, depending on the types of the activators and alkali dosages, the mechanical properties and activation reaction products may vary significantly, which may cause uncertainties of their long-term durability [4].

Carbonation is one of the important durability issues of reinforced concrete, since this process can lead to decalcification of the cement hydrates and reduction in the alkalinity of concrete pore solution. A reduced pore solution alkalinity of concrete will result in an increased susceptibility of reinforcement to corrosion as a result of the increased risk to breakdown the passive layer formed at high alkaline environment in non-carbonated concrete [5]. The carbonation of Portland cement-based materials has been widely investigated, and the factors controlling the carbonation resistance of these blends are well

understood. The CO₂ binding capacity, porosity and capillary condensation were found to be the decisive parameters governing the carbonation rate of these blends [6]. In contrast, carbonation of alkali-activated slag (AAS) mortars and concretes are much less understood due to the complexity of the chemistry of pore solution and activation reaction products. Several studies [7–11] have reported that AAS mortars or concretes were more susceptible to carbonation compared to Portland cement blends under accelerated carbonation conditions, whereas higher carbonation resistance was also reported for alkali-activated slag mortars or concretes under natural carbonation environments [12,13]. Bernal [14] explained that the changes in the pore solution equilibria caused formation of sodium bicarbonates during accelerated carbonation, which shifted the carbonation mechanism to favor more rapid reaction progress.

Carbonation of pore solution results in reduction of its alkalinity and thus decalcification of calcium bearing phases such as C-(A)-S-H phase, katoite (C₃AH₆) and/or strätlingite (C₂ASH₈) formed in alkali-activated slag mortars or concretes. The types and quantities of these activation reaction products vary significantly with the dosages and types of alkali activators, which may account for the differences in carbonation resistance. Bernal [14] reported that the carbonation depth reduced in the AAS mortars as the concentration of alkalis (wt% Na₂O) increased from

* Corresponding authors at: Key Laboratory for Green & Advanced Civil Engineering Materials and Application Technology of Hunan Province, College of Civil Engineering, Hunan University, Changsha 410082, China.

E-mail addresses: zhenguo.shi@empa.ch (Z. Shi), cshi@hnu.edu.cn (C. Shi), Zuhuzhang@hnu.edu.cn (Z. Zhang).

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9.1 to 10.6 wt%. Several studies reported that carbonation rate increased with the decrease of silicate modulus [8,15,16]. A possible explanation may be related to a denser pore structure with increasing alkali dosage and silicate modulus, which needs to be investigated in more details. In addition to the calcium bearing phases, hydrotalcite-like phase ($\text{Mg}_6\text{Al}_2\text{CO}_3(\text{OH})_{16}\cdot 4\text{H}_2\text{O}$) may also form in alkali-activated slag when the Mg/Al ratio of slag is within the range from 1.92 to 4.35. It has also been proved that this phase acted as a CO_2 sorbent for improving carbonation resistance [17]. Based on these discussions, it is suggested that more systematic studies are needed by looking into the effect of alkali dosage and silicate modulus to better understand the carbonation of the AAS mortars and concretes, since they are the important factors determining the pore structures, mechanical properties, pore solution chemistry and activation reaction products of AAS mortars and concretes.

The present study investigates the carbonation of the AAS mortars under accelerated carbonation conditions, i.e., $3 \pm 0.2\%$ (v/v) CO_2 at $20 \pm 2^\circ\text{C}/65 \pm 5\%$ RH for 56 days. Although the accelerated carbonation condition may overestimate the carbonation rates of alkali-activated concretes compared to that under natural carbonation condition, this method is still used in the present study since information derived from the accelerated carbonation experiment still allows us to understand how the alkali dosage and silicate modulus would affect the carbonation resistance of the AAS mortars. To better understand the carbonation mechanisms of the AAS mortars, additional information has also been obtained, such as compressive strength, porosity and average pore size determined by mercury intrusion porosimetry (MIP), phase assemblages examined by X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FTIR) and thermal gravimetric analysis (TGA).

2. Experimental

2.1. Materials

Granulated blast furnace slag (GGBS) produced by a local steel factory in Hunan Province, China, was used. Portland cement (PC) was also used in order to compare the carbonation resistance of its corresponding mortar with that of the alkali-activated slag mortars. Their chemical compositions determined by X-ray fluorescence (XRF) are given in Table 1. Alkali-activators with silicate modulus of 0 (NaOH only), 0.5, 1.0, 1.5 and 2 were prepared by mixing industrial grade waterglass (with original modulus $M_s = \text{SiO}_2/\text{Na}_2\text{O}$ of 3.3 and concentration of 65.2%) with different amounts of NaOH pellet (with purity of $99 \pm 1\%$). River sand with a maximum size of 2.36 mm and a fineness modulus of 2.75 was used as fine aggregate to prepare mortar samples.

2.2. Mortar preparations

Both AAS and PC mortars were prepared at a constant slag (cement)-to-sand ratio of 2.25 and water-to-slag (cement) ratio of 0.47. For the AAS mortars, the water content of waterglass was included into the mixing water. The alkali dosages of the AAS mortars were 6 and 8 wt% of the slag. All the mortars were cast into $40 \times 40 \times 40 \text{ mm}^3$ molds and cured at $20 \pm 2^\circ\text{C}$ with RH of $> 90\%$ for 24 h. After demolding, the mortars were steam cured in a chamber at 80°C for 48 h. Previous study

Table 1

Chemical compositions of the granulated blast furnace slag and Portland cement, wt%.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	LOI
GBFS	33.00	13.91	0.82	39.11	10.04	1.91	–	0.16	0.08
PC	22.51	5.32	3.78	63.12	2.56	0.71	0.19	2.02	–

showed that similar mortar samples reached their highest compressive strength after curing at such high temperature for 48 h as a result of full consumption of the alkali activators [18]. This means that the alkali-activation of the studied AAS mortars is nearly completed and the influence of possible minor continuous alkali-activation during CO_2 exposure on carbonation is negligible. Thus, the potential changes in the compressive strength, porosity and average pore size after the CO_2 exposure are only attributed to carbonation. Moreover, it should be noted that a complete alkali-activation does not necessarily correspond to a 100% reaction of slag. Thus, the extent of slag reaction may be different for the AAS mortars activated with different types and dosages of alkali activators.

2.3. CO_2 exposure

After 48 h of steam curing, the mortars were firstly cooled to room temperature and then exposed to a controlled atmosphere at $20 \pm 2^\circ\text{C}$ in a carbonation chamber for 56 days. The CO_2 concentration of the carbonation chamber was $3 \pm 0.2\%$ and the relative humidity was $65 \pm 5\%$, which are similar to conditions used in several other studies as reviewed in [4].

2.4. Methods

2.4.1. Phenolphthalein spray method

Carbonation depths were measured after 1, 3, 7, 14, 28 and 56 days of CO_2 exposure. Mortars were split into two halves and the fresh surface was immediately sprayed with a 1 wt% phenolphthalein in ethanol. The carbonation depths were measured on the mortars after the color had stabilized.

2.4.2. Compressive strength

The compressive strengths were determined on the extra mortar samples prepared in parallel to carbonation experiments. These measurements were performed after 14, 28 and 56 days of CO_2 exposure. The compressive strengths were also measured for the mortars prior to CO_2 exposure, i.e., after 48 h of steam curing. A loading rate of 2.4 kN/s specified in [19] was employed for determination of the compressive strengths. An average of three measurements is reported for each mixture.

2.4.3. Mercury intrusion porosimetry (MIP)

MIP measurements were performed on both the non-carbonated mortars (i.e., after 48 h of steam curing) and mortars carbonated for 56 days in order to evaluate the impact of carbonation on the changes in total porosity and average pore size of the studied mortars. The samples were crushed to grain size of 2–3 mm and then vacuum dried at 60°C for 2 days prior to MIP measurements.

2.4.4. X-ray diffraction (XRD) analysis

Paste samples with the same binder constituents as those used for the mortar samples were prepared for XRD analysis. Same curing and exposure conditions were applied to these samples. The measurements were performed on the paste samples after 48 h of steam curing and 56 days of carbonation. The carbonated samples with thickness of about 2 mm were cut from the carbonated surface of the pastes. Both the non-carbonated and carbonated samples were crushed and dried at 60°C for 24 h followed by passing a sieve with size of $325 \mu\text{m}$. The fine powders were analyzed using Philips X-ray diffractometer with $\text{CuK}\alpha$ radiation. The samples were step-scanned from 5 to 70° (2θ) at a rate of $0.04^\circ/\text{step}$.

2.4.5. Fourier transform infrared spectroscopy (FT-IR)

FT-IR analysis was performed on selected mortar samples after 48 h of steam curing and 56 days of carbonation. The non-carbonated and carbonated mortar samples were ground into powder and dried at 60°C

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