



Corrosion behavior of steel bars immersed in simulated pore solutions of alkali-activated slag mortar



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HIGHLIGHTS

- Simulated pore solutions of AAS mortar were first applied in this work.
- Both aluminate and silicate in AAS pore solution had corrosion inhibition effect.
- AAS had stronger capacity to passivate and protect steel bar than PC.

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ABSTRACT

To study the corrosion behavior of steel bars in alkali-activated slag (AAS) system, simulated pore solutions of AAS mortar were first applied in this work. The effects of the three major species in AAS pore solutions, namely sulfur-containing species, aluminate, and silicate, were investigated. Electrochemical impedance spectroscopy and potentiodynamic polarization results showed that the simulated pore solution of AAS mortar had a stronger capacity to passivate and protect steel bars than the simulated pore solution of Portland cement (PC) mortar, mainly due to the higher concentration of silicate in the former, as confirmed by surface analysis of SEM-EDXS.

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1. Introduction

To meet the construction industry's demands for cement and to ensure sustainable development of the environment, a 'greener' and energy-efficient cement, alkali-activated materials (AAMs), has been proposed [1]. Its CO₂ emission rate is approximately 6 times less than that of ordinary Portland cement (OPC) [2,3]. Because of the application of industrial by-products such as slag [4], fly ash [5], and clay minerals [6], AAMs have the superior attributes of a rich raw materials source, low price, and energy conservation. They also have higher chemical resistance, higher resistance to freeze-thaw cycles, and higher resistance to high temperature and fire [7]. These excellent properties make AAMs a competitive alternative for OPC [7,8]. At present, however, our understanding of the durability of AAMs, especially the corrosion behavior of steel in AAM concrete, is insufficient, due to the lack

of long-term performance monitoring compared to that available from the several hundred years' applications of OPC [8]. This lack is particularly true of AAMs based on blast furnace slag (BFS), denoted as alkali-activated slag (AAS), containing sulfide, that generates a reduction environment within the binder and causes electrochemical complexities that are not yet well understood [8]. Some researchers have studied the corrosion behavior of steel bars embedded in AAS concretes under chloride ingress or carbonation environment. The corrosion of reinforcing bars embedded in AAS concrete subjected to chloride attack was studied by Chaparro et al. [9]. Samples were immersed in 3.5% NaCl solution by weight of the slag. It was found that AAS concrete presented passive corrosion behavior in the first 3 months, after which it presented decreased corrosion resistance, lower than that of OPC concrete. Holloway et al. [10] applied detailed electrochemical characterization to the analysis of mild steel in AAS mortars containing NaCl admixtures, and found that the highest chloride levels produced the lowest corrosion rates, a trend that could not be clearly explained from a fundamental chemical perspective. Aperador

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et al. [2] found that the carbonation rate was higher in AAS concrete than in the respective OPC concrete under both accelerated carbonation (3% CO₂, 65% RH, and 25 °C) and a laboratory environment (0.03% CO₂, 65% RH, and 25 °C). In the authors' view, the high carbonation rate of AAS might be attributed to the low calcium content in AAS concrete that leads to a low capacity to buffer the pH of the pore solution in AAS and also leads to rare deposits of CaCO₃. Moreover, reduced carbonation resistance in AAS binders can also be caused by the chemistry of the pore solution and the pore structure of the binder matrix. The absence of calcium hydroxide in AAS binders is also relevant. Though many studies have been conducted, they cannot straightforwardly explain the corrosion chemistry of steel embedded in AAS binders. Thus there is a strong need for further detailed scientific and analytical work to determine the mechanisms in order to better understand and control steel corrosion within AAS [8].

Simulated pore solutions of AAS were applied in this work to study the corrosion behavior of steel bar in AAS binders, because the chemistry of pore solution around steel is closely related to the corrosion behavior of rebar in concretes and directly influences the passivation and the permanence of the passive state in the steel surface. Few researchers have studied the corrosion of steel immersed in simulated pore solutions of AAS binders. Chemical analysis of pore solutions in AAS paste by Puertas et al. [11] and Gruskovnjak et al. [12] showed that pore solutions of AAS had higher concentrations of Na, Al, Si, and S elements than those in OPC, with lower concentrations of K and Ca. It is interesting to find that several sulfur-containing species, such as S²⁻, S₂O₃²⁻, SO₃²⁻, and SO₄²⁻, existed in the AAS system, whereas only sulfate was available in the OPC system [12]. These differences in the chemistry of the pore solutions may lead to considerable differences between the corrosion behaviors of steel bars in these two cements.

The structure of this study is as follows. First, the chemistry of real pore solution extracted from AAS mortar was studied. Simulated pore solution was then prepared in accordance with the chemistry of the extracted pore solution. Through immersing steel bars in simulated pore solutions, various experimental methods were applied to study the effect of the main anions (S²⁻, S₂O₃²⁻, SO₃²⁻, SO₄²⁻, AlO₂⁻, and SiO₃²⁻) on the corrosion behavior of steel bars to reveal the corrosion mechanism of steel in AAS system. Simulated pore solution of Portland cement (PC) mortar was also prepared for comparative purposes. Two nondestructive monitoring techniques, electrochemical impedance spectroscopy (EIS) and linear polarization resistance (LPR), were utilized to continuously monitor the corrosion processes of steel bars. To clarify the corrosion behavior, cyclic voltammetry (CV) and potentiodynamic polarization (PDP) measurements were conducted. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDXS) were employed to observe the morphology of steel surfaces and to analyze corrosion products after the immersion experiments, in accordance with the EIS measurements.

2. Experimental

2.1. Pore solution extraction and analysis

Pore solution is usually squeezed from cylindrical samples [13] or fragments [14] of pastes or mortars using a high-pressure device. Concretes are not used, lest coarse aggregates damage the device. In this study, squeezing was performed on AAS mortars and also on PC mortars to extract pore solutions. The ground granulated blast furnace slag (GGBFS) used was S95 slag, conforming to GB/T 18046-2008. P-II 52.5 cement conforming to GB175-2007 was used to prepare the PC mortar. Chemical compositions, surface areas and specific gravity are summarized in Table 1. The basicity

(CaO + MgO)/(SiO₂ + Al₂O₃) and quality (CaO + MgO + Al₂O₃)/(SiO₂ + TiO₂ + MnO) indexes of GGBFS were 0.94 and 1.73, respectively. The waterglass used as activating solution was a mixture of commercially available sodium silicate mix (34.8% SiO₂, 11.6% Na₂O, and 63.6% water) with flaky analytical reagent NaOH to obtain a SiO₂/Na₂O ratio of 1 and a 5% concentration of Na₂O by weight of slag. The specific gravity and bulk density of river sand were 2630 kg·m⁻³ and 1480 kg·m⁻³ with a fineness modulus of 2.5. The sand/binder weight ratio was 2 for both AAS and PC mortars. Based on previous study, a water/cement ratio of 0.3 was used for AAS mortars to obtain pore solutions. PC mortars with water/cement ratio of 0.3, 0.4, and 0.5 were prepared, in order to find a suitable water content for the PC mortar to extract sufficient pore solutions for testing. Mortar samples were cast into plastic molds with the dimension Φ 50 mm × 60 mm at room temperature. After casting, the surfaces of the samples were sealed by plastic film and the samples were then cured in a curing chamber at the temperature of 20 ± 2 °C and RH >95%. Taking into account that the strength of AAS and PC mortars at age of 7 d can be almost 80% of that at 28 d, and the chemistry of pore solutions in AAS is similar from 7 d to 180 d [12], the pore solution of mortars was extracted at 7 d because continuing hydration of the specimens could lead to insufficient pore solution for squeezing out.

The pore solution extraction device in this experiment was similar to that used by Barneyback et al. [15]. Before each extraction test, all surfaces of the device were cleaned by ethanol at least twice until no residue remained [15]. An approximate 6 mm-thick Teflon seal was inserted between the top of the specimen and the bottom surface of the piston, to reduce the damage to the piston [14,15] and reduce the friction between mortar samples and piston by facilitating transfer of the applied pressure to samples.

The loading rate for extraction was about 1–2.8 MPa/s until the maximum 550 MPa stress was achieved [14,15]. To eliminate residual particles [14], the collected solution was filtered as soon as possible using filter paper with pore size 0.45 μm, then the pH of the pore solution was determined using a precision pH meter (PHS-3E, Shanghai INESA Scientific Instrument CO., LTD, China). All the liquid samples were sealed to minimize any carbonation or oxidation [16]. The concentration of elements (K, Na, Ca, Al, Si, and S) was measured by ICP-OES (700 Series, Agilent Technologies, USA), and concentrations of S²⁻, SO₃²⁻, S₂O₃²⁻, and SO₄²⁻ were tested by ion chromatography (IC) (ICS-3000, Thermo Fisher Scientific, USA), as listed in Table 2. The tested concentration of ions in AAS and PC mortars was consistent with the literature [12]. It should be noted that only PC mortars with 0.4 and 0.5 water/cement ratio could provide adequate pore solutions for chemical analysis.

2.2. Specimen preparation

The difficulty of studying the corrosion behavior of steel in pore solutions stems from the complexity of effectively replicating the chemical composition of the pore solution. For a PC system, saturated Ca(OH)₂ solution is usually applied to represent the chemical environment of the concrete's pore solution [17]. A certain amount of NaOH and KOH and even a trace of CaSO₄ may also be added [18,19], to better represent the pore solution chemistry of PC. On the other hand, simulated pore solution of the AAS system has not previously been used. As already stated, the chemical composition of the pore solution of AAS is more complex than that of PC. Therefore, we cannot use only one or a few species in pore solution to represent the real environment of AAS binder's pore solution, as was done for the PC system. Thus when preparing simulated pore solutions of AAS and PC mortars we considered all the ions that exist in real pore solutions to better represent the real environment in the binders' pore solutions. This method

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