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Electrochemical performance of reinforcing steel in alkali-activated slag extract in the presence of chlorides

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ABSTRACT

This study concerns the passivation behaviour and corrosion resistance of reinforcing steels in ordinary Portland cement (OPC) extract and alkali-activated slag (AAS) extract with chlorides. The electrochemical behaviour of steels in both extracts was monitored by various electrochemical measurements. Compared with OPC extract, less protective passive film was formed for steels in AAS extract without chlorides. However, much higher corrosion resistance was highlighted for steels exposed to AAS extract than OPC extract after the addition of 1 M NaCl. This was mainly attributed to the formation of gel-like substances in AAS extract, effectively suppressing the propagation of corrosion pits.

1. Introduction

It is well known that the production of one tonne of ordinary Portland cement (OPC) generates a total of 0.94 t of $CO₂$. As a result, about 7% of the total worldwide $CO₂$ emission comes from the cement industry [\[1\]](#page--1-0). On the other hand, with the rapid development of economy, the world is facing the serious situation of particulate pollution partially from the cement industry and the construction industry, particularly in the developing countries where the demand for cement is growing rapidly. Therefore, it is urgent to replace OPC with ecofriendly alternative binders without further compromising the atmospheric $CO₂$ levels and air quality [\[2,](#page--1-1)[3](#page--1-2)]. Of all the alternatives, alkaliactivated materials (AAMs) are very promising which can be used in construction industry and meet the requirements of workability, mechanical properties and other fundamental characteristics [[2](#page--1-1)]. However, when used in reinforced concrete structures (RCSs) in marine environments, the durability of RCSs with AAMs is of particular importance [\[1](#page--1-0)[,2,](#page--1-1)[4](#page--1-3)].

So far, carbonation, sulfate attack and chloride ingress of AAMs have been extensively investigated [\[1,](#page--1-0)[2](#page--1-1),[4](#page--1-3)–6]. However, the studies concerning the passivation and corrosion behaviour of reinforcing steels in AAMs exposed to chloride solutions were relatively scarce and the corrosion mechanism for steels in AAMs was not well understood $[7-12]$ $[7-12]$ $[7-12]$. It was confirmed that alkali-activated fly ash (AAFA), as the most frequently used low-calcium AAMs, could passivate reinforcing steels as rapidly and effectively as Portland cement. However, the passivation capability was mainly dependent upon the activators and the environmental conditions [\[7\]](#page--1-4). Monticelli et al. [[8](#page--1-5)] reported that,

due to the presence of higher concentration of inhibiting bicarbonate/ carbonate ions, steels embedded in AAFA mortars exhibited higher pitting corrosion resistance than those in OPC mortars. It was found recently that the soluble silicate ions, commonly used as a corrosion inhibitor for iron and steel, in the pore electrolytes of geopolymer also contributed to the high corrosion resistance for steels embedded in AAFA mortars during exposure to NaCl solution [[9](#page--1-6)]. In Fourier transform infrared spectroscopy (FTIR) analysis of geopolymer concrete, peaks of Si-O-Si bond and Al-OH were observed, suggesting the presence of sodium alumino silicate hydrate (N-A-S-H) gel at the rebarmatrix interface and the amorphous geopolymer mass was confirmed to be highly resistant to chloride-induced corrosion [[10\]](#page--1-7).

Alkali-activated slag (AAS) produced by calcium-rich precursor blast furnace slag (BFS) has been investigated for several decades with respect to its hydration mechanism and microstructure [[1](#page--1-0),[2](#page--1-1)[,4\]](#page--1-3). However, relatively few studies concerning the stability of passive film and the corrosion resistance of steels in AAS mortar/concrete have been reported [\[11](#page--1-8),[12\]](#page--1-9). It was found that the corrosion resistance of steels in AAS concrete was comparable to that in OPC concrete when exposed to chloride solutions. Moreover, the corrosion behaviour of steel in AAS concrete was largely affected by ionic exchange, carbonation and sulphide concentration [[11\]](#page--1-8). Holloway and Sykes found that the oxidation of sulphide anions originated from slag to sulphur hindered the formation of protective passive film by competitive adsorption between HS[−] and OH[−] anions at active sites on steel surface [\[12](#page--1-9)]. This phenomenon was well verified by a theoretical interpretation for steel in high BFS loaded BFS-OPC concrete [\[13](#page--1-10)]. On the other hand, when exposed to chloride solutions this oxidation reaction prevented the

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potential rising as high as the pitting potential and caused inhibition of the cathode by deposition of sulphur [[12\]](#page--1-9).

Most of the previous studies performed the electrochemical measurements of steels in alkali-activated mortars and concretes, thus it is difficult to observe the morphology of corrosion products and corrosion pits on steel surface. Synthetic solutions prepared with chemical reagents have been frequently used for simulating the concrete interstitial electrolytes [\[14](#page--1-11)–22]. Although testing in synthetic solutions may provide results that are more precise than in concrete or mortar [\[23](#page--1-12)], the influence of trace substances from cement hydration on the electrochemical behaviour was usually ignored [\[22](#page--1-13)]. Moreover, no universal standards for the composition of simulated concrete pore solutions have been established so far. Recently, cement extracts prepared with cement and water have been confirmed to be more suitable to simulate concrete pore environments compared to synthetic pore solutions due to the fact that the cement extracts contains Na⁺, K⁺, Al³⁺ and SO₄²⁻, etc. which presents the real environment of concrete [\[24](#page--1-14)]. Furthermore, extract solution is more appropriate for simulating AAMs since the composition of AAMs has not been well determined due to the different sources of raw materials and activator solutions [\[6,](#page--1-15)[25](#page--1-16)[,26](#page--1-17)].

In this study, the electrochemical behaviour of reinforcing steels in OPC and AAS extracts, simulating the concrete pore environment, was studied by means of various electrochemical measurements. The surface morphology of chloride-induced corrosion pits and corrosion products of steels was also obtained to support the electrochemical results and to reveal the corrosion mechanism for steels in different extracts.

2. Experimental

2.1. Raw materials

The chemical composition of ordinary Portland cement (P·II 42.5) and ground granulated blast furnace slag (GGBFS) determined by energy dispersive X-ray fluorescence (EDXRF) analysis (Thermoscientific, ARL QUANT'X) is shown in [Table 1.](#page-1-0) The chemical composition of hotrolled ribbed reinforcing steel (20MnSiV) is presented in [Table 2](#page-1-1). The cross-section of steel specimens was wet ground with SiC grinding papers successively to 1200 grits, and then polished with diamond polishing agent to eliminate visible scratches and imperfections. Afterwards, the polished steel specimens were cleaned with ethanol for the electrochemical measurements. Notably, electrochemical results with acceptable dispersion can be obtained for polished steel specimens with uniform surface condition.

2.2. Preparation of extract solutions

Two extract solutions were prepared for investigation:

- (1) Ordinary Portland cement (OPC) extract [[21,](#page--1-18)[27](#page--1-19)[,28](#page--1-20)], ordinary Portland cement and deionized water were mixed in a mass ratio of 1:1 and then stirred for 24 h, and finally the suspension was filtrated to prepare the OPC extract ($pH = 12.0$).
- (2) Alkali-activated slag (AAS) extract, GGBFS and the activator solution were mixed in a mass ratio of 1:1 and then the mixture was stirred for 24 h, and finally the AAS extract was obtained $(pH = 12.4)$ after the filtration of the obtained suspension. The activator solution consisted of commercial sodium silicate, sodium

Table 2

Chemical composition (wt.%) of reinforcing steel.

hydroxide (analytical reagent) and deionized water with a molar ratio $SiO₂/Na₂O$ of 1.2. The chemical composition of the commercial sodium silicate is 29.58% $SiO_2 + 13.15$ % Na₂O + 57.27% H₂O with a molar ratio $SiO₂/Na₂O$ of 2.32. The Na₂O concentration in the activator solution was 4% by weight of the mixed GGBFS.

The preparation of both extracts was conducted at room temperature (25 \pm 1 °C). The pH values of OPC and AAS extracts were lower than the corresponding hydrated mortars ($pH = 12.9$ for OPC mortar and $pH = 13.2$ for AAS mortar), which is due to the difference in watercement ratio (solution-slag ratio), the curing time, and the possible carbonation during extraction, etc. [\[5](#page--1-21)[,25](#page--1-16)[,29](#page--1-22)].

The photographs of the prepared OPC and AAS extracts can be seen in [Fig. 1.](#page--1-23) It is clear that flocculent precipitates were contained in OPC extract, whereas AAS extract was limpid with the color of primrose yellow. It was found that extract solutions were superior for simulating concrete pore environments than the simulated concrete pore solutions prepared by chemical reagents [[29,](#page--1-22)[30\]](#page--1-24), though both solutions are not fully representative of hydrated binders in concrete/mortar [\[31](#page--1-25)].

Analysis for the chemical composition of OPC and AAS extracts was performed using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) from Sysmex SpectroBlue ([Table 3\)](#page--1-26). Prior to analysis, the calibration was performed using standard solutions provided by the manufacturer.

2.3. Electrochemical measurements

The steel specimens were immersed in chloride-free extracts for 10 days to form a stable passive film. Afterwards, 1 M NaCl was added to both extracts to record the corrosion performance. Electrochemical measurements were performed after the stabilization of open-circuit potential (OCP) within the initial 1 h and further for time intervals of 10 days (passivation for steels), 13 days (1 M NaCl for 3 days) and 40 days (1 M NaCl for 30 days) of immersion. During the process of immersion and electrochemical measurements, the corrosion cells were all covered by a PMMA lid in order to mitigate the excessive carbonation of extracts.

The electrochemical measurements were all performed in PARSTAT 4000 Potentiostat. Steel specimen was the working electrode, and saturated calomel electrode (SCE) and platinum electrode were used as the reference electrode and the counter electrode, respectively.

Open-circuit potential (OCP), electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV) and cyclic potentiodynamic polarization (CPP) measurements were performed regularly to achieve the electrochemical performance of steels in extracts at passivation and corrosion stages. The arrangement of electrochemical measurements at various exposure times can be seen in [Table 4.](#page--1-23) Replicate steel specimens were prepared for the purpose of reproducibility and reliability of the electrochemical results. All potentials mentioned in this study were referred to the saturated calomel electrode (SCE). All experiments were conducted at room temperature (25 \pm 1 °C).

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