



Corrosion behavior of steel submitted to chloride and sulphate ions in simulated concrete pore solution



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HIGHLIGHTS

- Corrosion behavior of steel submitted to chloride and/or sulphate is investigated.
- Chloride threshold value and corrosion threshold value of sulphate are obtained.
- The concomitant presence of chloride and sulphate leads to higher corrosion rate.

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ABSTRACT

Electrochemical measurements of open circuit potential, linear polarization and electrochemical impedance spectroscopy (EIS) were utilized to investigate the corrosion behavior and chloride threshold value (CTV) of reinforcing steels submitted to chloride and sulphate attack in simulated concrete pore solution in this study. Determination of corrosion initiation was made by combining half-cell potential (E_{corr}) with corrosion current density (I_{corr}) as well as EIS curves. Results showed that electrochemical measurements were effective in detecting corrosion behavior of steels. CTV of steels was 0.5–0.6 mol/L in simulated concrete pore solution contaminated by chloride ions while threshold value of steels submitted to sulphate ions was 0.2–0.3 mol/L. The concomitant presence of chloride and sulphate ions led to higher corrosion current density which indicated sulphate ions accelerated the corrosion of reinforcing steels in simulated concrete pore solution.

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1. Introduction

Steel reinforcement embedded in concrete normally maintains passive due to the thin iron oxides film which is attributed to the high alkaline environment of concrete. However, once the chloride content at the steel surface reaches a certain level, i.e. chloride threshold value (CTV), when oxygen and moisture in presence, active corrosion of steel is initiated [1–3]. Thus the CTV is defined as the chloride concentration at the depth of steel which triggers active or pitting corrosion. Premature deterioration of reinforced concrete structures induced by steel corrosion has become a serious durability problem throughout the world, leading to significant economic loss [4,5]. Numerous investigations [6–11] have been performed on ingress of aggressive ions and CTV, but reported values are rather scattered. For example, the chloride threshold value presented by total chloride in concrete ranged from 0.04 to 8.34% by weight of binder and from 0.07% to 1.16% expressed by free

chloride [12]. Besides, many reinforced concrete structures are exposed to harsh environment where concentrated chloride and sulphate ions coexist like in salt lake district or salinized soil district in northwest China [13]. Previously, a lot of emphasis has been placed on sulphate attack to concrete matrix [13–15] but few reports regarding influence of sulphate ions on steel corrosion are found.

Experiments performed in cement paste, mortar and concrete to investigate CTV of steels are time consuming and are greatly affected by many influencing factors such as cement type, concrete mix proportions, moisture content, temperature and blended materials [12,16], though it may reveal more pertinent results. Therefore many authors have performed studies on steel corrosion in simulated concrete pore solution, a saturated $\text{Ca}(\text{OH})_2$ solution in most cases [17,18]. Simulated concrete pore solutions not only reproduce chemical environment in concrete but also shorten the experiment period and provide significant results for cement-based materials.

In the present work, the corrosion behavior of steels submitted to chloride and/or sulphate ions in saturated $\text{Ca}(\text{OH})_2$ solution

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(pH = 12.5) was investigated by employing open circuit potential (OCP), linear polarization (LP) and electrochemical impedance spectroscopy (EIS) methods. Corrosion initiation and the CTV were determined by combining half-cell potential (E_{corr}) with corrosion current density (I_{corr}) as well as EIS curves.

2. Experimental program

2.1. Specimen preparation and exposure conditions

Cylindrical steel specimens with a diameter of 16 mm and length of 10 mm were cut from HRB335 steel. The chemical composition of steel samples (by weight) was 0.20% C, 0.55% Si, 1.42% Mn, 0.028% S, 0.026% P and the balance of Fe. One cross-section surface was polished with grit paper to grade 1000 as exposure surface, degreased in acetone and then washed in distilled water. The remaining surface was sealed by epoxy resin. A copper wire was soldered to the other surface for electrochemical testing.

The saturated $Ca(OH)_2$ solution was prepared using distilled water, in which some insoluble $Ca(OH)_2$ remained. NaCl and/or Na_2SO_4 were added to saturated $Ca(OH)_2$ solution stepwise, 0.01 mol/L each day, as chloride and sulphate ions source. All chemical reagents applied in this study were analytical reagent grade.

2.2. Electrochemical techniques

All steel samples were immersed in saturated $Ca(OH)_2$ solution for ten days to obtain stable passive layer before experiment in order to simulate pre-passivated state in the concrete. Electrochemical tests were performed by using PARSTAT 2273 potentiostat/galvanostat immediately before adding new dosage of aggressive ions each day. With steel sample being working electrode, a saturated calomel electrode (SCE) and a platinum electrode were used as reference and counter electrodes, respectively. Half-cell potential, namely the E_{corr} , was with reference to SCE. The I_{corr} of steel reflecting corrosion rate was usually calculated by the LP method which is based on the Stern–Geary relationship [19] as in Eq. (1):

$$I_{corr} = \frac{\beta_a \beta_c}{2.303(\beta_a + \beta_c)} \frac{1}{R_p} = \frac{B}{R_p} \tag{1}$$

where I_{corr} is the corrosion current density, R_p the polarization resistance, B a constant related to β_a and β_c , the anodic and cathodic slopes of Tafel curve, respectively. The value of B is usually assumed to be from 26 mV to 52 mV [20]. The value of 26 mV was adopted in this study with the maximum error in the measurement a factor of 2. In this work, the R_p was obtained directly by built-in fitting software. EIS scan was carried out at the open-circuit potential with an AC signal of amplitude 10 mV and sweep frequency from 10 mHz to 100 kHz.

3. Results and discussion

3.1. Corrosion behavior in chloride contaminated condition

Three parallel steel samples labeled as #1, #2 and #3 are exposed to chloride added stepwise by 0.01 mol/L each day. The evolutions of E_{corr} are presented in Fig. 1. It can be found that the average E_{corr} of specimens after ten days of pre-passivation

is around -230 mV vs. SCE. With the additions of chloride, The E_{corr} decreases gradually and moves to negative values, which means a higher risk of corrosion. Then the E_{corr} drops dramatically when a certain content of chloride is added, indicating the breakdown of passive film and initiation of active corrosion. The CTV of steels can be inferred to be from 0.05 to 0.06 mol/L.

The evolutions of I_{corr} obtained from the LP and Eq. (1) are depicted in Fig. 2. With lower concentration of chloride, I_{corr} keeps a steady low value. When chloride content exceeds a certain level, I_{corr} denotes a sudden shift to greater values, indicating high corrosion rate. It should be noted that the pit growing rate (penetration depth rate) is assumed to be around 10 times higher than the average corrosion rate measured [21]. An I_{corr} of $0.1 \mu A/cm^2$ means that the pitting rate value could be higher than $1 \mu A/cm^2$, which indicates significant active corrosion. Also the CTV could be assumed to be from 0.05 to 0.06 mol/L according to I_{corr} evolution.

An abrupt change in topology of Nyquist plots of EIS can also be observed in Fig. 3. A typical Nyquist plot of steel immersed in simulated solution depicts a capacitive loop of depressed semicircles. As the concentration of chloride increases, the radius of the low-frequency semicircle gradually reduces, becoming from “linear” to “semicircular”.

An equivalent circuit shown in Fig. 4 was used to interpret EIS results, where R_s is the solution resistance and R_{ct} the charge

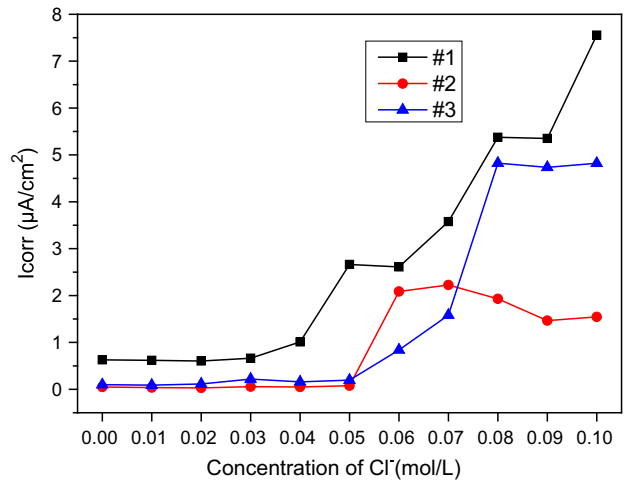


Fig. 2. Variations of I_{corr} of specimens with additions of chloride ions.

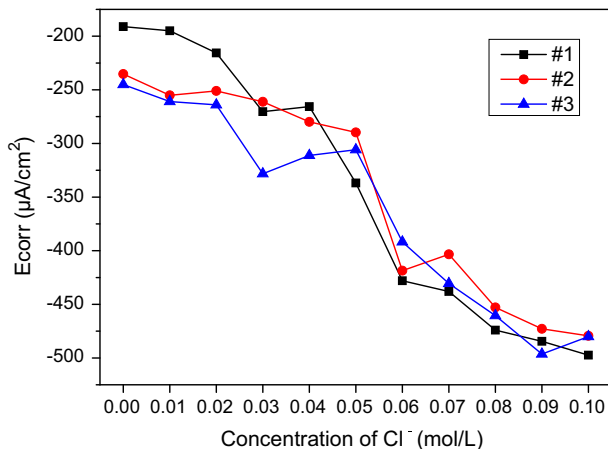


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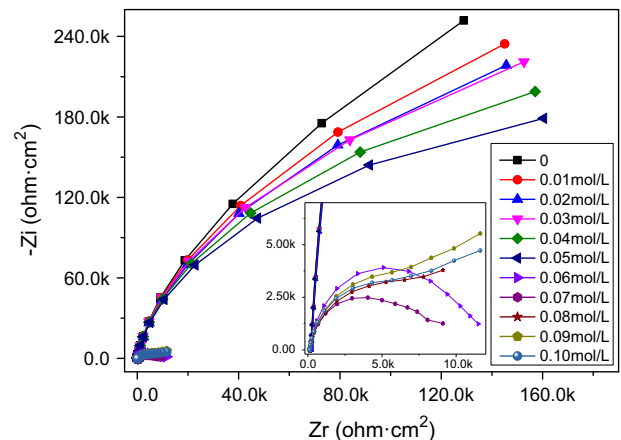


Fig. 3. Typical Nyquist plots of specimen with additions of chloride.

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