



# The effect of carbonate and sulfate ions on chloride threshold level of reinforcement corrosion in mortar with/without fly ash



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## HIGHLIGHTS

- The coupling effect of carbonate and chloride ions on reinforcing corrosion in mortar has been studied.
- The effect of sulfate ions on chloride threshold level of reinforcing corrosion in mortar has been researched.
- The effect of carbonate or sulfate ions on chloride binding capacity has been studied.

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## ABSTRACT

The aim of the study is to evaluate the effect of carbonate and sulfate ions on chloride threshold level of reinforcement corrosion in mortar with/without fly ash. The initiation of active corrosion for the reinforcement was detected by half-cell potential and electrochemical impedance spectra methods. The free chloride or total chloride by mass of binder was used to express the chloride threshold level. The results indicate that the presence of carbonate ions in the chloride solution shortened the time to initiation of reinforcement corrosion and decreased chloride threshold level in both the Portland cement mortar and fly ash mortar specimens. And compared with the single chloride solution, the concomitant presence of chloride ions and sulfate ions decreased the chloride threshold level, but did not influence the time to initiation of chloride-induced reinforcement corrosion. Besides, although the presence of fly ash decreased the chloride threshold level, it delayed the time to initiation of reinforcement corrosion.

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## 1. Introduction

The steel in concrete is normally in a passive state against corrosion due to the physical and chemical protection provided by concrete. The physical protection is provided by its dense structure which retards the ingress of aggressive species to the steel-concrete interface, and the chemical protection is provided by the high alkaline environment of the pore solution which aids to the formation of a thin protective layer on the steel surface. However, when the chloride ions penetrate into the concrete and exceeds to a critical level, i.e. chloride threshold level, the passive film is broken down and active corrosion is initiated.

The chloride threshold level is a key parameter to evaluate the durability and service-life prediction of reinforced concrete. Based on this, numerous investigations [1–7] have been performed on the

chloride threshold level. However, the reported values do not get an agreement and scatter a wide range. The scatter of reported values is attributed to the fact that chloride threshold level is affected by many factors, such as the detection methods [1], chloride salt type [2–4], sources of chloride salt [5], blended materials [6–8], inhibitors [9] and so on.

Thousands of salt lakes are distributed in Northwest of China. Except for chloride ions, these salt lakes contain a large amount of carbonate and sulfate ions. The presence of carbonate or sulfate ions may significantly affect the chloride-induced reinforcement corrosion and chloride threshold level. Limited data developed by Holden et al. [10], Jin Zuquan et al. [11] and Cheng et al. [12] are focus on the role of sulfate ions on reinforcement corrosion, and there are few reports to study the effect of carbonate ions on the chloride-induced reinforcement corrosion. In a previous publication [13], the role of carbonate ions on the chloride-induced reinforcement corrosion had been examined. It was demonstrated that the influence of carbonate ions is related to the content of

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carbonate ions and the kind of simulated solution. Hence, there is a need to investigate the effect of carbonate and sulfate ions on chloride threshold level of reinforcement corrosion in mortar.

This study is aimed to study the effect of carbonate and sulfate ions on chloride threshold level of reinforcement corrosion in mortar with/without fly ash. The initiation of active corrosion for the reinforcement was detected by half-cell potential and electrochemical impedance spectra methods. The free chloride or total chloride by mass of binder was used to express the chloride threshold level.

## 2. Experimental

### 2.1. Materials and specimen preparation

A cylindrical steel bar with a diameter of 1 cm and length of 6 cm was used and its chemical composition (in % by mass) was 0.22% C, 0.30% Si, 0.65% Mn, 0.05% S, 0.045% P and the residual Fe. The cement was NO. 42.5 ordinary Portland cement made in China. Fly ash from Nanjing, China was used in the work. The chemical composition of cement and fly ash is shown in Table 1. River sand with a fineness modulus of 2.8 was used as fine aggregate. The mortar mix proportions are listed in Table 2.

A copper wire was welded to one end surface of the steel bar. The exposed area of each steel bar was 12.5 cm<sup>2</sup> and the remaining area was sealed by means of epoxy resin and hot-shrinkable tube.

Mortar specimens of 4 cm × 4 cm × 16 cm with a steel bar centered in the middle were cast and the cover depth of 15 mm was provided. After casting, the mortar specimens were placed in the laboratory temperature for 24 h. Then they were demoulded and cured in a 95% humidity chamber at 20 ± 2 °C for 28 days. After the curing period, 20 mortar specimens from each mortar mix were average placed in the five exposure solutions for 4 days and the composition of the exposure solutions is detailed in Table 3. Then the specimens were exposed to a temperature of 40 °C for 1 day and room temperature for 2 days. The immersion-drying cycle procedure was repeated until the onset of active corrosion for the specimens had been tested by electrochemical techniques.

### 2.2. Electrochemical measurements

During the immersion-drying cycle procedure, the corrosion potentials and corrosion current density were measured by half-cell potential and electrochemical impedance spectrum methods, respectively. The corrosion potentials provide the information on the time to initiation of pitting corrosion, while the corrosion current density provides information on the rate of corrosion.

According to ASTM C-876 [14], half-cell potentials were measured in the drying procedure of the immersion-drying cycle with a copper-copper sulfate reference electrode (CSE). The electrochemical impedance spectrum was gotten by utilizing a Partstat 2273 Advanced Potentiostat/Galvanostat/FRA system in the immersion procedure of the immersion-drying cycle. A saturated calomel electrode and a platinum electrode were connected to work as a reference and auxiliary electrode, respectively. The electrochemical impedance spectrum was obtained with the frequency range from 10 mHz to 100 kHz and a sinusoidal potential perturbation of 5 mV at the open circuit potentials. In this case, the equivalent-circuit model shown in Fig. 1 is applied to fit the EIS curve in order to obtain the ideal charge transfer resistance (i.e. the polarization resistance  $R_p$ ), which has also been used in some literatures [1,15–17]. In the equivalent-circuit  $R_s$  is the electric resistance of mortar. The parallel circuit ( $R_f$ ,  $Q_f$ ) is applied to account for the high-frequency loops and represents the dielectric properties of the passive film of steel bars. The double layer capacitance ( $Q_{dl}$ ) and the charge transfer resistance ( $R_p$ ) describe the dissolution processes at the steel/mortar interface, while the Warburg impedance ( $W$ ) is associated with the transport of charged species. Then the polarization resistance  $R_p$  was introduced to the Stern-Geary formula to calculate the corrosion current density  $I_{corr}$ . The Stern-Geary formula is  $I_{corr} = B/R_p$ , where  $B$  is Stern-Geary constant, assumed to be 26 mV [18,19].

**Table 1**  
Chemical composition of cement and fly ash.

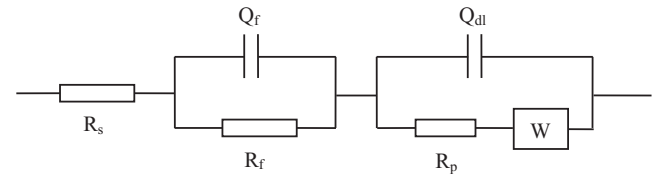
Constituent (wt%)	Cement	Fly ash
SiO <sub>2</sub>	22.97	53.93
Al <sub>2</sub> O <sub>3</sub>	9.34	31.31
CaO	52.28	2.99
Fe <sub>2</sub> O <sub>3</sub>	3.10	3.56
MgO	1.35	0.58
K <sub>2</sub> O	1.03	0.29
Na <sub>2</sub> O	0.15	1.82
SO <sub>3</sub>	2.12	0.94
Ignition loss	4.16	2.82

**Table 2**  
Mix proportions of mortar.

Sample	Mix proportion wt/ %		Binder-sand ratio	Water-binder ratio
	Cement	Fly ash		
B0	100	0	1:3	0.53
F1	85	15	1:3	0.53
F2	70	30	1:3	0.53

**Table 3**  
Composition of the exposure solutions.

Solution	Concentration of the exposure solution
1	5%NaCl
2	5%NaCl + 2% Na <sub>2</sub> CO <sub>3</sub>
3	5%NaCl + 4% Na <sub>2</sub> CO <sub>3</sub>
4	5%NaCl + 2% Na <sub>2</sub> SO <sub>4</sub>
5	5%NaCl + 5% Na <sub>2</sub> SO <sub>4</sub>



**Fig. 1.** Equivalent circuit applied to fit the electrochemical impedance spectra curve.

### 2.3. Chemical analysis

Once the active corrosion had been confirmed by both half-cell potential and corrosion current density, the mortar specimens were broken to analyze the free chloride and total chloride of the mortar adjacent to the steel bar. Specific operation to measure the free chloride as follows: the cement paste was gotten by removing the aggregates, grinded until it can pass through a sieve of 0.63 mm and dried at 40 °C to constant weight. Then 5 g powder was added to 50 ml distilled water in a sealed container. After vigorous stirring and waiting for 24 h, the solution was filtered and analysed by potentiometric titration to obtain the free chloride. Moreover, the total chloride was evaluated by dissolving the powder in dilute nitric acid. Both free chloride and total chloride were expressed in percent by mass of binder.

## 3. Results

### 3.1. Half-cell potential

The variations of half-cell potentials for the Portland cement mortar specimens exposed to 5% NaCl adding 0%, 2% and 4% CO<sub>3</sub><sup>2-</sup> solution are indicated in Fig. 2. The initial half-cell potentials were high and decreased with the period of the immersion-drying cycle. The half-cell potential-time curves were applied to evaluate the time to initiation of reinforcement corrosion based on the ASTM C876 criterion of -350 mV CSE. The corrosion initiation time of the Portland cement mortar specimens exposed to solution 1, 2 and 3 is 112d, 91d and 77d, respectively.

Fig. 3 shows the variations of half-cell potentials for the fly ash mortar specimens exposed to 5% NaCl adding 0%, 2% and 4% CO<sub>3</sub><sup>2-</sup> solution. The half-cell potentials displayed a similar variation trend to that of the Portland cement mortar specimens. The order of initiation of corrosion in three test solutions is: 5%NaCl + 4% Na<sub>2</sub>CO<sub>3</sub> → 5%NaCl + 2% Na<sub>2</sub>CO<sub>3</sub> → 5%NaCl. Hence, the carbonate ions shortened the time-to-initiation of reinforcement corrosion significantly.

Fig. 4 depicts the variations of half-cell potentials for the Portland cement mortar specimens exposed to 5% NaCl adding 0%, 2% and 5% SO<sub>4</sub><sup>2-</sup> solution. Initially, the half-cell potentials were around

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