



Corrosion performance of steel reinforcement in simulated concrete pore solutions in the presence of imidazoline quaternary ammonium salt corrosion inhibitor



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HIGHLIGHTS

- The IQS was used as corrosion inhibitor for reinforced concrete for the first time.
- The IQS presented a superior corrosion inhibition performance in SCPs.
- The mechanism is due to its adsorption on steel surface, forming a protecting film.

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ABSTRACT

In this study, the corrosion inhibition performance of steel reinforcement in the simulated concrete pore solution containing 3.5 wt.% NaCl in the presence of a tailored cationic type of imidazoline quaternary ammonium salt corrosion inhibitor was characterized by electrochemical measurements and surface analysis. The results indicate that the IQS can significantly improve the polarization resistance and pitting potential and reduce the corrosion current density of reinforcement. The main inhibition mechanism is most likely due to the adsorption of the corrosion inhibitor on the steel surface, leading to a reduced corrosion current density of the steel reinforcement.

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1. Introduction

It is generally accepted that concrete is one of the most widely used engineering materials for constructions, and its durability is the major problem affecting the service life of the engineering structures. Corrosion of the steel reinforcement is one of the main reasons causing the premature deterioration of reinforced concrete [1–2], and leading to a significant economic loss [3].

In order to improve the durability of reinforced concrete, various technologies and methods were applied [4], e.g. patch repairs [5], coatings [6], sealing and membranes for concrete surface [7], special steel bar (stainless steel bar, epoxy coated steel reinforcement, et al.) [8,9], electrochemical protection (including cathodic protection, electrochemical realkalization and electrochemical chloride extraction (ECE)) [10–14] and corrosion inhibitors [15–18]). Due to its excellent inhibition performance, low cost and labor saving, corrosion inhibitor is considered as one of the

most effective, long-term corrosion protection methods for steel reinforcement [19]. Traditional corrosion inhibitors are divided into inorganic (mainly nitrites) and organic (alkanolamine and their inorganic, organic acid salt mixtures) substances. Although nitrites were widely reported as an efficient corrosion inhibitor [20,21], due to its carcinogenicity and biological toxicity, it is forbidden in many European countries, i.e. Germany and Switzerland [22]. Alcoholamine [23,24] is a widely used organic corrosion inhibitor, however, there are still conflicting opinions about its effectiveness and different mechanisms were also reported: Kern and Landolt [25] suggested that organic inhibitors adsorbed on the steel surface, forming a barrier layer to prevent steel dissolution. Violetta et al. [26] revealed that this inhibitors can both halt the anodic and cathodic reactions, reducing the corrosion rate of reinforcing steel. Heiyantuduwa [27] proved that the alcoholamine presented a corrosion inhibition effect only for the chloride-induced corrosion of reinforcement. In recent years, the electrochemical migrating corrosion inhibitors were also investigated [28–30], which can be applied in the electrochemical salt extraction process. However, the available electrochemical migrating

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corrosion inhibitors are limited, and the related corrosion inhibition mechanism is still not clear. Therefore, it is very important to further investigate the exact mechanisms and develop a tailored type of high efficient organic corrosion inhibitor.

Imidazoline quaternary ammonium salt (IQS) is a type of organic corrosion inhibitor which is widely used for corrosion protection in petroleum industry [31,32]. Its molecule contains both hydrophilic and hydrophobic groups: the hydrophilic group (imidazoline heterocycle) adsorbs on the active sites of the steel, forming a single molecule adsorption film; the hydrophobic group covers the steel surface, acting as a barrier against iron ions diffusing outward and aggressive ions diffusing inward on the steel surface [33]. Due to the special molecule structures (two nitrogen atom with its lonely sp^2 electron pair and the imidazoline heterocyclic), the adsorption of IQS is more pronounced on the steel surface, compared to alkanolamine, leading to an enhanced inhibition efficiency. Although as a high efficient corrosion inhibitor, to our best knowledge, the IQS based corrosion inhibitors were never used in reinforced concrete. A well-known fact is that after the ECE treatment in reinforced concrete, there are a large amount of excess electrons remained on the surface of steel reinforcement, and the reinforcing steel is still in an active state. In this condition, if the chloride ions still exist, the “secondary corrosion” of reinforcing steel is easy to take place [34]. Therefore, the IQS corrosion inhibitor is designed as a surface applied and electrochemical migrating corrosion inhibitor for hardened concrete structures. If the IQS inhibitor is utilized during the ECE process, it can quickly penetrate into concrete matrix and absorb on the reinforcement surface, combining with the “excess electrons” and effectively preventing the “secondary corrosion”. In this way, the efficiency of ECE can be stabilized and improved, and the IQS migrating corrosion inhibitor can easily penetrate into concrete matrix under the electric field for ECE process.

In this study, the corrosion inhibition performance of steel reinforcement in the simulated concrete pore solution in the presence of the corrosion inhibitor was evaluated. Its corrosion inhibition efficiency was also compared with sodium nitrite and an alcohol amine based corrosion inhibitor. The corrosion performance of the steel reinforcement in the simulated concrete pore solutions containing different types of corrosion inhibitors was investigated by electrochemical measurements (potentio-dynamic polarization (PD), electrochemical impedance spectroscopy (EIS) and potentiostatic); and the influence of different corrosion inhibitors on the threshold chloride concentration for corrosion initiation of steel reinforcement was evaluated by potentiostatic polarization. Further, the surface of the reinforcing steel was analyzed by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

2. Experimental

2.1. Materials

2.1.1. Synthesis of IQS corrosion inhibitor

The IQS corrosion inhibitor used in this study was synthesized by solvent method, which was completed by two steps: the acylation–cyclization reaction and cationized reaction. First, the imidazoline was synthesized by an acylation–cyclization reaction. The imidazoline was then converted to a cationic type of imidazoline quaternary ammonium salt by a cationized reaction. The synthesis process is as followed.

Acylation–cyclization reaction: 0.20 mol diethylenetriamine, 0.22 mol lauric acid and 40 mL xylene (as solvent and diversion agent) were added to a 250 mL three-necked round bottom flask equipped with a stirrer, temperature controller, and a Barrett distilling receiver with a condenser on the top. The Barrett distilling receiver was filled with xylene. The reaction mixture was gradually heated with stirring up to the boiling point of the xylene (140–170 °C) and water collection was initiated. The amount of water azeotroped with the xylene was determined. The first 3.4 mL which was collected in the first 2–4 h of reaction indicated that the reaction was 96% complete (Fig. 1(a)).

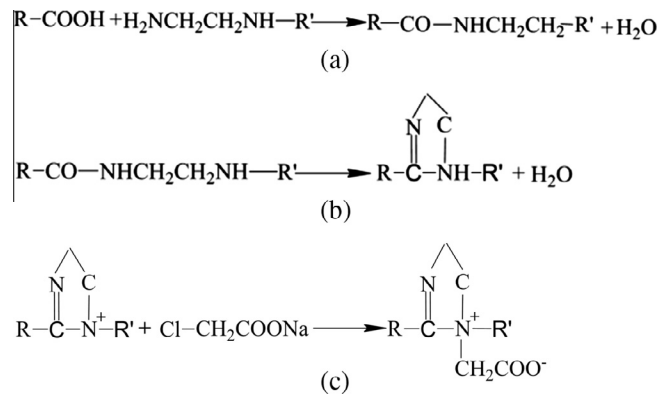


Fig. 1. Schematic of synthetic steps (a: acylation reaction, b: cyclization reaction, c: cationization reaction).

The reaction temperature was then slowly raised to 190–210 °C. The reaction was stopped after 15–18 h (Fig. 1(b)). The product was purified by rotary evaporation at 60 °C and vacuum degree of 0.09 for 30 min to remove the water and xylene impurities. IR was recorded and the results agree with the postulated structure.

Cationized reaction: further, in order to increase the stability of imidazoline and make it positively charged, the imidazoline was proceeded with a cationized reaction. 0.2 mol imidazoline and 30 ml xylene were added to a 250 mL four-necked round bottom flask equipped with stirrer, peristaltic pump, temperature control and a condenser, and aqueous solution of sodium chloroacetate (0.22 mol, 16 wt.%) was then added dropwise into the reaction mixture within 15 min, the pH of the mixture was adjusted to about 7.5–8 by NaOH solution (30%). The mixture was then heated to 70–80 °C for 6–8 h. The purification process was the same as in the first step. IR characterization of the final product confirmed the structure as that postulated in Fig. 1(c) formula.

2.1.2. Preparation of steel reinforcements and simulated concrete pore solutions

The Q 325 steel reinforcements with a diameter of 10 mm were sealed by epoxy resin with only one side exposed and the working area was about 0.785 cm². Its chemical composition was as follows: 0.18% C, 0.28% Si, 0.55% Mn, 0.04% S, 0.04% P and the residual Fe. Prior to the measurements, the steel reinforcements were polished with grades 200, 600, 1000 and 2000 silicon carbide emery papers, and rinsed with alcohol.

Based on the reported studies [35,36], the SCP solution contaminated with chlorides was prepared by dissolving the analytical grade calcium hydroxide (Ca(OH)₂, 0.003%), sodium hydroxide (NaOH, 0.008%), potassium hydroxide (KOH, 0.005%) and sodium chloride (NaCl, 3.5%) in deionized water, and the pH of SCP solution was 13.35. In this study, different types of corrosion inhibitors were added to the simulated pore solution, ending up with 4 solution types: SCP only (as reference case); SCP + sodium nitrite (SN, with a concentration of 2%); SCP + alcohol amine (AA, with a concentration of 2%); SCP + IQS (with the concentration of 0.5%, 1% and 2%, respectively). The different concentrations of IQS corrosion inhibitor employed in this test were selected with the purpose to evaluate the corrosion performance of different levels of IQS corrosion inhibitor on steel reinforcement in the simulated concrete pore solution.

2.2. Methods

2.2.1. Electrochemical measurements

Prior to the electrochemical tests, the steel reinforcements were conditioned in the relevant testing solutions with the ages of 1 d, 7 d, 14 d, and 28 d, respectively. The electrochemical measurements performed in this study (at a room temperature of 25 ± 1 °C) included PD and EIS measurements. A common three-electrode arrangement was used [3]: reinforcing steel reinforcement serving as the working electrode, titanium mesh serving as the counter electrode and a saturated calomel electrode (SCE) serving as the reference electrode (all potentials reported in this study were referred to SCE). The PD tests were performed in the potential range from –0.2 V to +1.2 V vs. open circuit potentials (OCP) with a scan rate of 0.5 mV/s, the EIS measurements were carried out at the OCP and in a frequency range from 100 kHz to 10 mHz. There were at least 3 replicates for each specimen. The equipment used was 273 A potentiostat/galvanostat (EG&G PAR, Oak Ridge, TN, USA) and 5210 Lock-in amplifier system.

2.2.2. Threshold chloride concentrations (TCC)

The threshold chloride concentrations for corrosion initiation of steel reinforcements in SCP solutions in the presence of different corrosion inhibitors were determined by potentiostatic polarization. Prior to the tests, the reinforcements were immersed in SCP solutions without chlorides for 5 days to form a passive layer on

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