

Effect of phosphate based inhibitor on the threshold chloride to initiate steel corrosion in saturated hydroxide solution



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HIGHLIGHTS

- In alkaline medium, Na₃PO₄ is an efficient inhibitor against the Cl⁻ ions aggressivity.
- Na₃PO₄ increases the resistance to localized corrosion of reinforcement in concrete.
- Na₃PO₄ increases the threshold ([Cl⁻]/[OH⁻]) of steel corrosion from 0.6 to 15.
- The acoustic emission technique is a powerful tool to monitor the corrosion process.

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ABSTRACT

In this paper, a phosphate-based inhibitor was used to increase the well-known critical ratio [Cl⁻]/[OH⁻] (*R*), inducing the localized corrosion of steel reinforcements in saturated Ca(OH)₂ solution. For this purpose, electrochemical measurements and characterization methods (SEM, EDS, XRD, etc.) were first employed to identify the phosphate layer formation, and then to evaluate the inhibition effectiveness for the localized corrosion of steel reinforcements. The process of pitting corrosion is evaluated by cyclic polarization and is followed by acoustic emission.

The experimental results show that electrochemical techniques can evaluate the pitting corrosion and reveal that the introduction of phosphate-based inhibitor increases strongly the critical ratio [Cl⁻]/[OH⁻] from which the depassivation and pitting corrosion can be initiated. This clearly confirms its positive effect on the localized corrosion resistance of steel reinforcements in saturated Ca(OH)₂ solution. A perfect correlation with the evolution of the acoustic activity is also demonstrated. Thus, it was significantly possible to highlight the acoustic signature of depassivation damage and to chloride concentration.

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1. Introduction

Steel corrosion in reinforced concrete is one of the main pathologies in civil engineering. Observed predominantly in marine structures and chemical industries, it is also very present in civil and/or nuclear structures: buildings, bridges, seawater pipelines, reactors and nuclear waste containers. In concrete, corrosion of steel reinforcements reduces the life and the durability of structures. Localized corrosions are induced by chlorides present in the surrounding medium [1,2], therefore they are the major cause of its degradation. The chloride ions eventually reach the steel and accumulate beyond a certain concentration level at which the protective film is destroyed and the steel begins to corrode, if oxygen and moisture are present in the steel–concrete interface. Besides, a vital parameter for chloride threshold value is the pH of the pore

solution, since the amount of chloride ions required for corrosion initiation increases in the presence of hydroxide ions. The [Cl⁻]/[OH⁻] (*R*) ratio seems to be the most accurate parameter to take into account when testing the corrosion onset in reinforced concrete [3–5].

Many investigations have been conducted in order to limit this phenomenon and to protect reinforced steel against corrosion, such as addition of inhibitors and surface treatment [6–9]. Two approaches are generally used to evaluate the efficiency of inhibitors for concrete application; one uses simulated pore solution testing, and the other uses actual concrete or mortar [10–13]. The approach in simulated pore solutions generally employed a saturated calcium hydroxide solution (pH 12–13), hydroxide solutions, and cement extract solution, with or without alkaline addition [14,15]. Those kinds of model solutions allow one to verify the inhibitor efficiency and to control some parameters that otherwise would be very difficult to achieve, such as natural and *in situ* testing.

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Table 1
Composition of carbon steel (RB 500).

Elements	C	S	P	N	Cu
wt.%	≤0.22	≤0.050	≤0.050	≤0.012	≤0.80

The objective of this work is to perform a simple and efficient treatment on steel to prevent corrosion by increasing the critical ratio R in saturated $\text{Ca}(\text{OH})_2$ solution. To do so, a new processing is developed in a bath containing phosphate. Steel with and without pretreatment will be investigated to determine the value of the critical ratio R . Electrochemical techniques associated to acoustic emission were applied and completed by SEM observations, EDS analysis and X-ray diffraction. Acoustic emission technique is used to *in situ* monitor, characterize and anticipate localized corrosion in its early stages [16–18].

2. Experiments

2.1. Working electrode

The working electrode is a carbon steel rod of 6 mm in diameter normally used as concrete reinforcement. Chemical composition of this rebar is presented in Table 1. For electrochemical tests, the specimen is embedded into an epoxy resin to form a disk electrode. So, the electrode surface area is only the cross-section, and is equal to 0.28 cm^2 .

Before each experiment, the electrodes are polished using a series of silicon carbide emery papers of grades between 120 and 4000 under a fountain to remove surface defects.

2.2. Treatment method

The treatment method consists on an immersion of the steel electrode during 7 h in 0.2% $\text{Ca}(\text{OH})_2$ solution and in 0.2% $\text{Ca}(\text{OH})_2$ with different contents of Na_3PO_4 (1%, 3% and 5%) solutions. The treatment study is conducted at room temperature around 22°C and without stirring.

2.3. Corrosion test

The reinforcement corrosion is triggered at the end of the treatment period. Electrode is kept in 0.2% $\text{Ca}(\text{OH})_2$ and in 0.2% $\text{Ca}(\text{OH})_2$ with 5% Na_3PO_4 solutions, chloride ions “in the form of NaCl ” are added up to a ratio $R = [\text{Cl}^-]/[\text{OH}^-]$ varying between 0 and 20.

The equipment used in this study is a RADIOMETER potentiostat/galvanostat using the VOLTALAB version 3.10 software. The experimental parameters of potentiodynamic polarization are carried out at a constant scan rate of 25 mV min^{-1} .

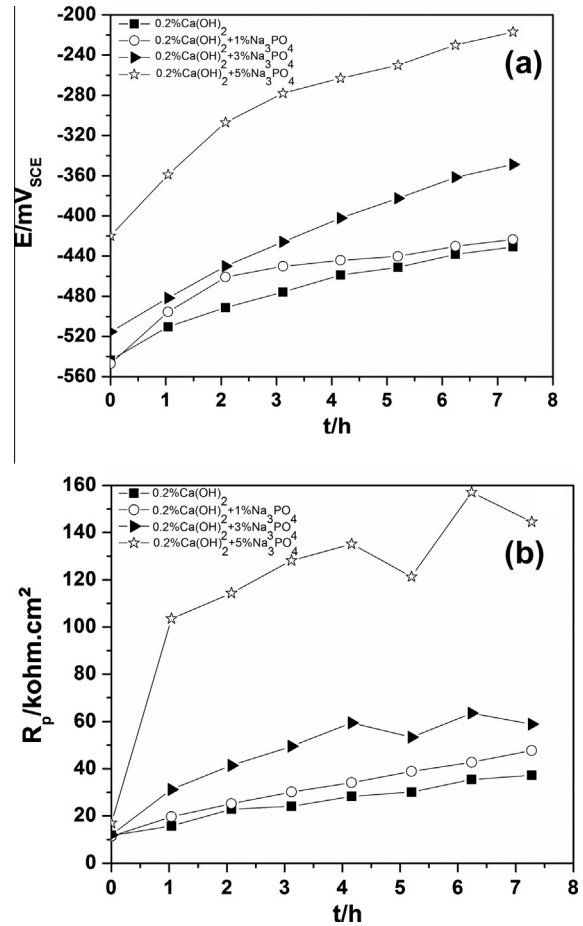


Fig. 2. Open circuit potential (E_{ocp}) (a) and polarization resistance (R_p) (b) evolution during 7 h of steel immersion in 0.2% $\text{Ca}(\text{OH})_2$ solution and 0.2% $\text{Ca}(\text{OH})_2$ containing various content of Na_3PO_4 .

2.3.1. Electrochemical measurements

The polarization resistance (R_p), the corrosion potential (E_{corr}) and the cyclic polarization are used to measure the localized corrosion tendencies of a specimen in a given metal-solution system. Indeed, in a pitting experiment, one applies at the beginning, a potential scan from cathodic to anodic region until a large increase on current occurs. The potential at which the current sharply increases is defined as the pitting potential (E_{pit}). Thus, the scan direction is reversed when the current density reaches 1 mA/cm^2 and the final potential should be less than the corrosion potential ($E_{corr(c/a)}$) as determined by a preliminary scan. The potential where the

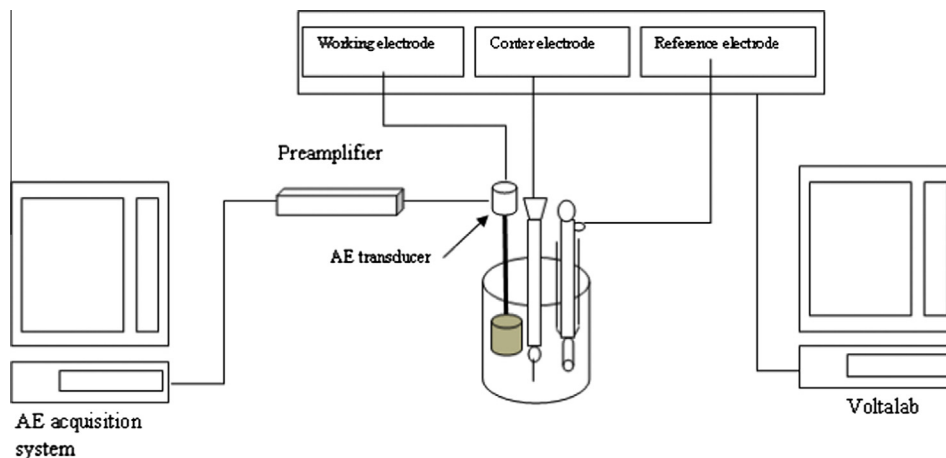


Fig. 1. Experimental device of acoustic emission instrumentation.

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