



# Testing phosphate ions as corrosion inhibitors for construction steel in mortars



L. Yohai, M.B. Valcarce<sup>1</sup>, M. Vázquez<sup>1,\*</sup>

División Electroquímica y Corrosión, Facultad de Ingeniería, INTEMA, UNMDP-CONICET, J. B. Justo 4302, (B7608FDQ) Mar del Plata, Argentina

## ARTICLE INFO

### Article history:

Received 18 June 2015

Received in revised form 16 December 2015

Accepted 19 December 2015

Available online 22 December 2015

### Keywords:

mortars  
corrosion inhibition  
phosphate ions  
chloride

## ABSTRACT

The purpose of this investigation is to analyze the effectiveness of phosphates as inhibiting agents for steel bars embedded in mortars. Three mix designs were selected for this study: mix A had no admixed chlorides and was used as reference; mix designs B and C were contaminated with 1% in weight of chlorides/weight of cement and mix C also incorporated 7%  $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ . After setting for 48 h, the samples were cured for 7 days immersed in water. Three specimens of each group were then immersed in aerated saline solutions  $0.5 \text{ mol L}^{-1} \text{ NaCl}$  during 720 days. The corrosion potential ( $E_{\text{corr}}$ ), the polarization resistance ( $R_p$ ) and the electrochemical impedance spectra were recorded regularly. After 720 days of immersion one set of mortars was anodically polarized and another set cathodically polarized. After that, Raman spectra of corrosion products were registered. Additional specimens were used to evaluate porosity and chloride profiles. The presence of phosphate ions as inhibitors has no effect on  $E_{\text{corr}}$  values and suggests mixed-type inhibition. Mix C remains passive until 180 days of exposure, with  $R_t > 100 \text{ k}\Omega \text{ cm}^2$ . At longer times,  $R_t$  decreases in time but the inhibition percentage is always higher than 95% when evaluated by impedance spectra. Using  $R_p$ , inhibition stays above 70%. Anodic and cathodic polarization curves, together with corrosion potential values, suggest that phosphates behave as a mixed-type inhibitor.

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## 1. Introduction

Reinforced concrete is one of the most widely used structural materials in the construction industry worldwide. However, faulty concrete formulations or an aggressive service environment promote premature deterioration and failure. Among the most frequent problems, reinforcing steel bar (rebar) corrosion is at the top of the list.

Concrete, and the pore solution in contact with steel, provide a highly alkaline environment which helps to develop a passive layer that protects the rebar from active corrosion [1]. There are many factors that influence the stability of the passive film such as the chloride ions content in concrete [2–4] and concrete carbonation [5–7], the mix design and curing conditions of concrete, the chemical composition of the pore solution and the thickness of the concrete cover [8,9].

Many local variables, including the mineralogy of raw materials, the exposure conditions and traditional construction practices, may also influence rebar corrosion. For example, since the early seventies, the use of sea sand for civil construction in certain cities located at the coastal region of Argentina has become quite common. As a consequence, there are many structures nowadays presenting severe damage caused by rebar corrosion [10].

Various procedures are frequently employed in an attempt to minimize rebar corrosion, such as cathodic protection [11], realkalization [12], and the application of coatings to the external concrete surface or to the reinforcing steel bars [13–15]. Another alternative is the use of inhibitors, which can be cost-effective and are easy to apply. They can be used in reinforced concrete by adding the inhibiting agent to the mixing water during the concrete preparation or by applying it to the external surface of hardened concrete. Reviews of the most commonly used corrosion inhibitor types in concrete repair systems and the various possible mechanisms of inhibition are available [16–18]. The most commonly admixed inhibitors are formulated on the basis of nitrite ions [19,20]. However, nitrites should be used with care when lixiviation can contaminate surrounding soil or water.

Other ions have been investigated as candidates to inhibit pitting corrosion of steel, namely chromates, phosphates,

\* Corresponding author at: División Electroquímica y Corrosión, Facultad de Ingeniería, Universidad Nacional de Mar del Plata, INTEMA, CONICET, Juan B. Justo 4302 – B7608FDQ Mar del Plata – Argentina. Tel.: +54 223 481 6600 ext 244; Fax: +54 223 481 0046.

E-mail address: [mvazquez@fi.mdp.edu.ar](mailto:mvazquez@fi.mdp.edu.ar) (M. Vázquez).

<sup>1</sup> ISE member.

tungstates and molybdates [21–23]. Phosphates present some interesting advantages such as low cost and low toxicity.

To evaluate the efficiency of phosphate ions as inhibitors for reinforced concrete application, some authors simulate pore solutions [24–30]. In contrast, few articles have reported evaluations of the phosphate ions effectiveness in mortars or cement paste at long exposure times [31,32]. The interaction between phosphate ions with cement paste is complex. Some authors argue that phosphates can alter the mechanical properties of concrete or modify curing times, as they decompose and precipitate as calcium phosphate, in turn decreasing the efficiency of the inhibitor [33,34]. However, other authors claim that this inhibitor does not interfere and demonstrate that it is effective in mortars [31,32,35]. Besides, the adequate phosphate to chloride ratio in mortars is controversial and the inhibition mechanism is not clear. Some authors propose a dual effect, where calcium phosphate could block the pores avoiding diffusion of aggressive species while iron phosphate could block cathodic or anodic sites [31,32,36,37].

Our previous work focused on phosphate ions as inhibitors for highly alkaline pore solution contaminated with chlorides. A mixed-type corrosion mechanism was found when phosphate to chloride ratio is 1 [29]. That study in simulating pore solutions is being extended to carbonated solution and is currently in press [38].

In parallel, the purpose of this investigation addresses the effectiveness of phosphates as inhibiting agents but now for steel bars embedded in mortars after long exposure times. Papers published earlier by other authors, that address the effect of phosphates in mortar, are either accelerated tests [36], deal with short term exposures [26,37], use monofluorophosphates [35], or study pre-oxidized or as-received rebars [32].

## 2. Experimental

### 2.1. Samples design

The study was performed using cylindrical mortar test specimens (2.8 cm in diameter by 8 cm high) containing one rebar segment and leaving a 1 cm mortar cover (see Fig. 1). The rebar segments presented an exposed area of 12.6 cm<sup>2</sup>. Table 1 presents the chemical composition of the reinforcing steel bars used in the study. The bars were abraded with 1000 grade emery paper just before embedding them in the mortar paste.

**Table 1**  
Chemical composition of the steel rebars.

% (w/w)	Mn	C	Si	Cu	impurities
	0.63	0.30	0.26	0.23	0.24

Portland cement was used with water to cement ratio of 0.6 and sand to cement ratio of 3 (ASTM C-305). Three mix designs were selected for this study. Mix **A** had no admixed chlorides and was used as reference. Mix designs **B** and **C** reproduced the case of reinforced concrete structures heavily contaminated with a known amount of chlorides (1%, expressed in weight of chlorides/weight of cement). Mix **C** also incorporated 7% Na<sub>3</sub>PO<sub>4</sub>·12H<sub>2</sub>O, where the phosphate to chloride ratio is 0.6. The mortars were prepared using river sand and Portland cement containing less than 0.1% per weight of chloride ions.

After setting for 48 h, the samples were cured for 7 days immersed in water. Three specimens of each group were then immersed in aerated 0.5 mol/L NaCl solutions (equivalent to 2.92% w/V).

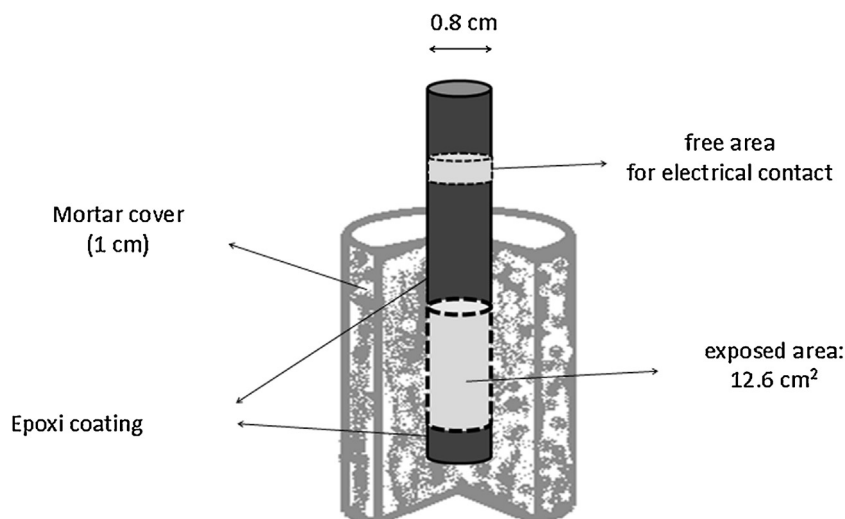
Additional specimens with the same composition but without rebars were casted in parallel. These were used to evaluate physical properties, as described below. The size of these samples is the same as those described above.

### 2.2. Electrical and electrochemical measurements

Typical electrochemical parameters normally used to characterize the corrosion behavior of reinforcing steel in concrete were monitored periodically during 720 days. These included the corrosion potential ( $E_{corr}$ ), the polarization resistance ( $R_p$ ) and polarization curves. Also, the electrochemical impedance spectra were recorded regularly. All these tests were carried out with the mortars immersed in 0.5 mol/L NaCl solutions.

The corrosion potential was measured against a mercury/mercuric oxide reference electrode, Hg/HgO in KOH 1 mol/L ( $E = 0.123$  V vs. NHE) using a Gamry Reference 600. The reference electrode was positioned inside a Luggin capillar, with the tip touching the mortar external surface. The counter electrode was a platinum wire of large area.

Using the same set-up, polarization resistance ( $R_p$ ) was evaluated as  $\Delta V/\Delta i$ , from potential sweeps up to  $\pm 0.015$  V from  $E_{corr}$  at a scan rate of  $10^{-4}$  V s<sup>-1</sup>. The results were corrected so as to



**Fig. 1.** Cylindrical mortar test specimens including one rebar segment.

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