



# Protection of reinforcement steel corrosion by phenylphosphonic acid pre-treatment PART II: Tests in mortar medium



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## ABSTRACT

A pre-treatment of steel reinforcement in mortar by a 72 h immersion in 0.1 M phenyl-phosphonic acid ( $C_6H_5P(O)(OH)_2$ ; PPA) was investigated. Then effectiveness of this procedure for protection against the corrosion of steel bars embedded in pre- or post-addition of sodium chloride mortar was evaluated by electrochemical impedance spectroscopy, visual inspection, SEM, and EDS analyses.

The results showed that for non-treated steel reinforcement, the charge transfer resistance ( $R_t$ ) decreases considerably with time indicating a very advanced state of corrosion after 54 months corrosion test. In contrast, for pre-treated steel rebar, this resistance remains high reflecting the effectiveness of the pre-treatment method against corrosion. The corrosion rate evaluated from the charge transfer resistance at 54 months corrosion in chloride containing medium was  $0.5 \mu m year^{-1}$ .

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## 1. Introduction

The high alkalinity of the concrete ensures chemical protection by the formation of protective passive layer on the reinforcements. However, in the presence of water, of oxygen, and various aggressive agents such as chlorides and carbon dioxide, this film will be damaged. Corrosion process thus starts, and as consequences in one hand the formation of expansive corrosion products leading to the loss of adherence to the concrete, and on the one hand, the reduction of the steel reinforcements sections is resulting in a loss of mechanical resistance. The former will involve the cracking of the concrete and will accelerate thus the process of corrosion by facilitating the diffusion of oxygen and the corrosive species.

Several methods are used to mitigate the corrosion effect of the reinforcements in the concrete; they include cathodic protection [1], metallic, inorganic, or organic coatings, and addition of inhibitors to the freshly mixed concrete like additives [2] as preventive methods, or by surface impregnation on hardened concrete

[3] as curative application.

The calcium nitrites as additives in the concrete was proposed as the first inhibitors [4], and revealed a significant decrease of the corrosion rate of the steel reinforcements. However, their toxicity led several researchers to propose other inhibitors innocuous and friendly to the environments [5].

The use of the phosphates ( $Na_2PO_3F$  [6–9],  $Na_3PO_4$  [10–13]), recognized not toxic, gave promising results. They indeed showed, a remarkable effectiveness in alkaline medium simulating the interstitial solution of the concrete. However, its efficiency is partially lost in concrete [12]. The loss of efficiency of these compounds when added to fresh concrete is attributed to the reaction with Ca forming  $Ca_3(PO_4)_2$  that precipitates in the pores reducing the content of soluble  $PO_4^{3-}$  in the pore solution able to actuate as inhibitor, as happens with  $Na_2PO_3F$  [6–9].

Phosphonates are molecules that contain one or more groups  $R-PO(OH)_2$ . Their good chemical stability and solubility in water make possible their use in oil production, in the formulation of detergents, and in the inhibition of corrosion and scale formation [14]. Among all these properties, their ability to form strong complexes with metals plays a very important role in the rate of transports of these compounds in the environment.

The use of phosphonic acids to protect carbon steel against

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corrosion was studied by various work [15–24]. In part I of the present paper [25], Etteyeb et al. reported the viability of the pre-treatment of carbon steel in phenylphosphonic acid solution, against the corrosion in alkaline chloride solution. Moreover, they showed that the protective film formed by the pre-treatment procedure is stable in air. In this paper, authors will investigate the long-term effectiveness of the pre-treatment against the corrosion of reinforcement steel when embedded in mortars, especially by Electrochemical Impedance Spectroscopy (EIS) because this method is considered as non-invasive. Indeed, in the last decades, several authors have studied the reinforcement corrosion activity in concrete by Electrochemical Impedance Spectroscopy [26–29].

## 2. Experimental conditions

### 2.1. Mortar specimens

For this study, to ensure uniform distribution of electric field, cylindrical mortar specimens, 70 mm in height and 46 mm in diameter, were prepared. The overall aspect and the dimension of mortar specimen are presented in Fig. 1. The mortar was fabricated with Ordinary Portland Cement (OPC). A cement/sand (c/s) ratio of 1/3 and water/cement (w/c) ratio of 1/2 was used [30,31]. The steel reinforcement was set at the centre of the mortar cylinder. The steel reinforcement was made of a plain carbon steel rod of 6 mm in diameter and 70 mm in length. The bars were chemically cleaned, then rinsed with acetone and distilled water. The test specimens were prepared with non-treated steel rebars and with steel rebars treated by continuous immersion in 0.1 M Phenylphosphonic acid (PPA) solution during 72 h.

Each rebar had a protruding part in the specimen for electrical connection. To minimize differential aeration and also to determine the exposed area, the top and bottom parts of the rebar were covered with an insulating tape. The upper part, that is, the cover mortar thickness was 2 cm. The length of rebar exposed to the mortar was of 3 cm in length thus having an area of 5.7 cm<sup>2</sup>. The specimens were cured in a humidity box at 100% RH and 20 ± 2 °C for 7 days before any experiments. Three replica measurements were carried out for each series to check the reproducibility of results.

Mortar tests were divided into two groups:

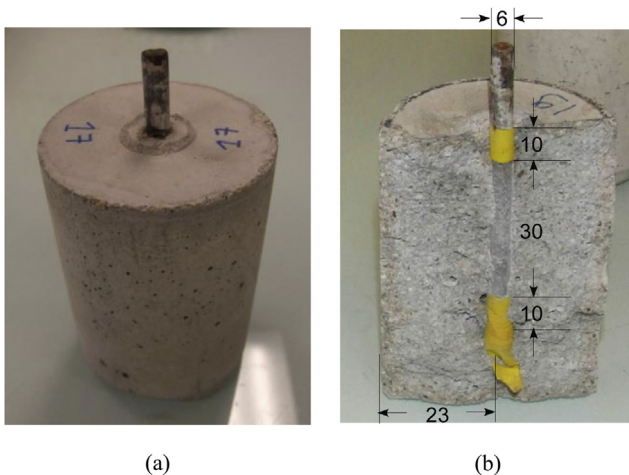


Fig. 1. Photograph of the reinforced mortar test specimens employed (a) overall view (b) the dimension.

### 2.1.1. Group I

In this group, 1% NaCl by weight of cement was added to the mixing water. These specimens were prepared with untreated steel rebars (reference specimens) or with steel rebars treated as indicated above. This group is called hereby as “pre-chlorinated mortar”. Between two collections of impedance spectra, the mortar samples were left in a humidity box. Fig. 2 illustrates the humidity box used for the ageing of pre-chlorinated mortar specimens. The specimens were set on a perforated plastic plate and the water filled in the bottom of the box to insure the humidity in the tightly covered plastic box.

### 2.1.2. Group II

Mortar specimens without chloride were prepared with untreated or treated steel bars. After a curing period, the samples were submitted to alternative cycles of immersion and emersion, (2 days drying and 1-day immersion in 0.5 M NaCl solution, thus 3 days for one cycle). This system will be called “post-chlorinated mortar”.

For the former group, the aggressive chloride ions were present initially by adding NaCl in the mortar whereas for the second group, the chloride ions were brought by immersion in NaCl solution. The alternative cycles between immersion and emersion enhanced the penetration of chloride ions inside the mortar matrix.

## 2.2. Electrochemical measurements

The experiments were performed to follow the time-change of the electrochemical impedance spectrum. Fig. 3 shows the electrochemical cell layout used for these measurements.

The electrochemical impedance spectra (EIS) were collected with a Gamry potentiostat (model PC4/300) using a 10mVrms ac-signal at the open circuit corrosion potential ( $E_{oc}$ ) from 100 kHz to 10 mHz with 5 points per decade.

The saturated calomel electrode (SCE) was used as reference and in fixed at the upper part of the mortar cylinder. The electrical contact to the mortar that consists of the electrolyte was ensured with a tiny wet synthetic sponge. The counter electrode was stainless steel wire of 0.5 mm in diameter surrounding the lateral part of the mortar cylinder. A wet synthetic sponge surrounding the lateral part of mortar cylinder and its bottom allows also the electrical contact with the counter electrode and also avoids the drying of mortar specimens during impedance measurements. After these measurements, the mortar specimens were transferred to a humidity box or an immersion – emersion cell for the next impedance measurements.

## 2.3. Characterization of steel-mortar interface

At the end of the test, specimens were broken and the surface state of steel and mortar were examined visually.

This examination made possible the characterization of the corrosion pattern. The corrosion products and steel–mortar interface were then analysed with Energy Dispersive Spectrometry

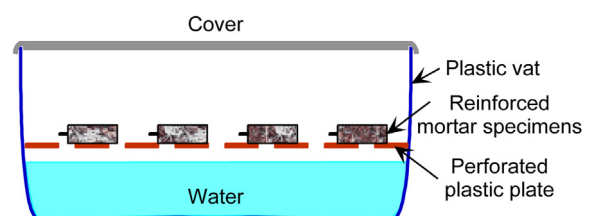


Fig. 2. Humidity box in plastic vat for ageing of pre-chlorinated mortar specimens.

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