

Effectiveness conditions of sodium monofluorophosphate as a corrosion inhibitor for concrete reinforcements

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Abstract

Among the array of protection and repair methods for reinforced concrete structures when steel reinforcements undergo corrosion, the use of migrating corrosion inhibitors could prove to be an attractive alternative. These products, applied to the concrete surface, are intended to diffuse into the concrete in order to reach the reinforcement and stop or delay corrosion.

Some key aspects on the use of monofluorophosphate (MFP) as an inhibitor for concrete reinforcements are presented first. The experimental work has focused on developing a method to determine the amount of MFP in concrete and on studying the chemical interactions taking place between MFP and certain concrete components. These data are then used to explain MFP penetration profiles in concretes as well as the correlation with electrochemical measurements.

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1. Introduction

Monofluorophosphate (MFP) has been used as a corrosion inhibitor for concrete reinforcements over the last 20 years. It is applied at the concrete surface in the form of an aqueous solution with a mass percentage ranging between 10% and 20%. The effectiveness of MFP, applied to the surface of a concrete structure, is based on both its diffusion into the concrete pore network and its action on steel reinforcement surface. Accordingly, the fundamental stage herein is the ease of access of MFP to the reinforcements.

It has often been mentioned in the literature [1–3] that in early-age concrete, the penetration of an aqueous MFP solution is insufficient or impossible and, as a consequence, the PO_3F^{2-} ions, which actually perform the inhibiting action, are not present in sufficient quantities within the interstitial solution to successfully inhibit corrosion. By forming apatites with Ca-

containing compounds when the concrete has not been carbonated, PO_3F^{2-} ions are trapped but reaction products may accumulate in some parts of the porous network, thus blocking the penetration of aggressive species. In a carbonated concrete, aqueous MFP solutions are better able to penetrate [2–4]. MFP penetration should occur by means of a pure diffusion process whenever the concrete is saturated. Other types of penetrations could also arise depending on the amount of water in the concrete pore network [4].

The work presented in this paper lies within the scope of a research programme [5] conducted on the effectiveness of MFP and focuses on the knowledge of MFP, in particular in terms of its potential penetration into concrete materials.

2. Materials and methods

2.1. Materials

Cylindrical mortar specimens (35 mm diameter and 40 mm long) containing a centred mild steel bar ($\text{C}\approx 0.16\%$ – $\text{Si}\approx 0.12\%$ and $\text{Mn}\approx 0.4\%$) (6 mm diameter) were cast with

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Table 1
Chemical composition of the cement and mix design of mortar specimens

Cement composition	Mass %	Mortar composition	Kg/m ³
SiO ₂	20.6	Cement	516
Al ₂ O ₃	4.9	Sand	1504
Fe ₂ O ₃	2.9	Water	258 (W/C=0.5)
CaO	61.3		
MgO	4.5		
K ₂ O	1.16		
Na ₂ O	0.24		
SO ₃	3.1		
Free CaO	1.1		
LOI	0.7		

ordinary Portland cement. The compositions of both the cement and the mortar are listed in Table 1.

Cement paste specimens were also produced using Portland cement and a W/C ratio of 0.5. All specimens were wrapped in an adhesive aluminium sheet, in order to reduce moisture loss, and then conserved one year at 20 °C before the experimental campaign.

The purity of the MFP used, as determined by quantifying the phosphorus, was 96±3%. Prior to applications on cementitious specimen surfaces, an X-ray diffraction diagram of MFP was carried out to verify it had not been hydrolysed.

2.2. Methods

The method used for determining the MFP content in concrete has been previously presented in [6]. This method consists of the total dissolution of concrete in a 4% nitric acid solution, followed by determination of the MFP amount in the solution by means of ion chromatography (Dionex DX120 chromatograph).

As regards the electrochemical tests performed on an EG and G apparatus (Model 263A Potentiostat and Model 1025 Frequency Analyzer), the study focused on reinforced mortar specimens submitted or not to degradation (chloride ions, carbonation). Two electrochemical techniques were selected herein, i.e.:

- monitoring of steel corrosion potential over time; this technique, mainly used in situ to produce maps of the corrosion state, makes it possible to detect electrochemical “events” (more or less distinct variations in potential); and
- determination of electrochemical impedance spectroscopy (EIS) diagrams, a technique that has been used quite often [7–9] over the past few years yet one in which implementation for metal embedded in concrete, as well as in results interpretation, is not straightforward.

3. Experimental program and results

3.1. Interactions between MFP and concrete constituents

This first part of the study was devoted to the analysis of chemical interactions between MFP and some of the major

concrete constituents in order to define the penetration conditions of this inhibitor within concrete structures. As indicated previously, the main limiting factor is apatite formation. It thus appeared necessary to determine the apatite formation conditions. In pursuit of this objective, the study has been carried out to determine the following:

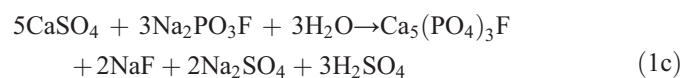
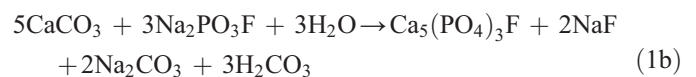
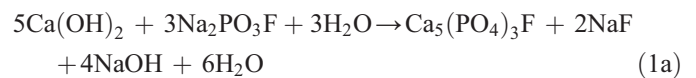
- Influence of high alkalinity (pH≈13) in the pore solution: OH⁻ ions and Na⁺ and K⁺ counterions present in the concrete pore network;
- Influence of portlandite Ca(OH)₂, which is another compound influencing concrete alkalinity;
- Influence of CaCO₃ resulting from Ca(OH)₂ carbonation; and
- Influence of the other compounds containing calcium as gypsum (CaSO₄·2H₂O).

For compounds with low solubility (i.e., portlandite (pK_s=5.3), calcite (pK_s=8.4) and gypsum (pK_s=4.6)), the tests consisted of placing these compounds for 24 h in contact with an aqueous solution of 10% MFP by mass. The solid phases were then dried at either 50 or 105 °C (depending on their thermal stabilities) and analysed by means of X-ray diffraction. The filtered solutions were analysed by ion chromatography and atomic spectrometry.

For soluble compounds (KOH and NaOH), the tests consisted in observing the possible formation of a precipitate and analysing the solution by ion chromatography.

Ion chromatograms from the NaOH or KOH solutions showed no interaction between MFP and the alkaline solutions: only the PO₃F²⁻ ion, resulting from MFP dissolution, was observed in the chromatograms.

Concerning Ca(OH)₂, CaCO₃, and CaSO₄ in contact with MFP solutions, it may be stated that if a chemical reaction occurs with apatite formation (Ca₅(PO₄)₃F), the reactions for each of the considered compounds would be:



Consequently, PO₃F²⁻ ions would disappear (due to low apatite solubility) and F⁻ ions appear (soluble NaF) within the liquid phase. Under stoichiometric conditions and after a 24-h contact period, the ion chromatograms derived on the solutions (see Fig. 1a, b and c) indicate that just the portlandite had reacted.

This result was confirmed by X-ray diffraction: a new solid phase appeared only with portlandite, which was identified as apatite (Fig. 2).

Taking into account the similarity of X-ray diffraction spectra for fluoroapatite (card ICDD 15-876) and hydroxy-

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