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Corrosion inhibition mechanism of phosphates for early-age reinforced mortar in the presence of chlorides

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ABSTRACT

A study was performed on the corrosion-inhibition mechanism of three phosphate compounds (sodium monofluorophosphate, disodium hydrogen phosphate and trisodium phosphate) for steel rebars embedded in ordinary Portland cement (OPC) mortar exposed to 3% NaCl solutions. The corrosion inhibitors were deployed in two different ways: by migration after the curing period, through the immersion of mortar specimens in aqueous solutions containing the soluble phosphates; and by initial admixture of the phosphate powders in the OPC mortar. The tested specimens were studied using electrochemical corrosion potential and linear polarisation resistance for 70 days of experimentation. A correlation was found between the phosphate content of the specimens (by migration or admixture) and the steel corrosion rate. The results indicate that all the phosphates have the ability to inhibit steel corrosion in mortar.
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1. Introduction

Chloride-induced corrosion of reinforcing steel is one of the most important factors governing the durability of reinforced concrete structures (RCS), particularly in coastal marine environments [1,2]. Many different methods have been employed to mitigate the corrosion risk in RCS, such as barrier layers [3], cathodic protection [4], galvanized or stainless steel rebars [5], and corrosion inhibitors. The latter may be a good way to prevent and/or control reinforcing steel corrosion because inhibitors are easy to apply and less costly than other prevention methods [6–8]. They can be deployed by addition to the mortar or by application on the hardened surface [9].

The use of phosphate compounds as corrosion inhibitors has been widely studied, but there continues to be a lack of consensus about the inhibition mechanism [10,11]. Phosphates offer an environmentally-friendly alternative to traditional and possibly toxic sodium nitrite ($NaNO_2$) inhibitor [12,13], which has been forbidden in many countries [8]. Soluble phosphates, traditionally sodium monofluorophosphate (Na_2PO_3F) (MFP) or disodium hydrogen phosphate (Na_2HPO_4) (DHP), can be deployed in concrete in two ways: by immersion of the concrete in a phosphate solution or surface application of a phosphate solution on the hardened concrete by brushing [14,15], or by addition to the cement, sand and water mixture [16]. In the first case the phosphates penetrate the concrete pore network by capillarity, and the efficiency of these methods generally depends on the permeability of the concrete. In all cases, the soluble phosphates react with calcium hydroxide (portlandite) ($Ca(OH)_2$) to trigger the precipitation of a calcium phosphate, reducing their capacity to act as corrosion inhibitors [17–21], and delaying the setting of cement [2].

In a recent work by the authors [22], a comparative study was performed on the inhibition of steel reinforcement corrosion using MFP, DHP and trisodium phosphate 1-hydrate ($Na_3PO_4{\cdot}H_2O$) (TSP) compounds in the absence of chlorides. The three corrosion inhibitors (MFP, DHP and TSP) showed good inhibition behaviour. The order of efficiency for phosphates acting as migrating corrosion inhibitor (MCI) was MFP > DHP > TSP, and as admixture corrosion inhibitor (ACI) the best corrosion inhibitor was the DHP compound. Thus it is interesting to investigate the efficiency of these phosphate-based corrosion inhibitors for exposure to chloride containing solutions, as an alternative to traditional nitrite-based compounds.

The aim of this paper is to study the corrosion inhibition mechanism of three soluble phosphates (MFP, DHP and TSP) for reinforced mortar immersed in 3% sodium chloride (NaCl) solutions and to evaluate the effectiveness of the corrosion inhibitors

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application method by migration, through the immersion of mortar specimens in an aqueous phosphate solution, or by initial addition to cement as an admixture.

2. Experimental

Type I 52.5N/SR ordinary Portland cement (OPC) was used with the chemical composition given in Table 1, as determined by X-ray fluorescence. Its particle size distribution (Fig. 1) was determined by laser diffraction, with 10% of the particles being smaller than 1.5 μm, 50% smaller than 10.5 μm, and 90% smaller than 29.2 μm. Carbon steel bars of 8 mm in diameter and a chemical composition (wt.%) of 0.45 C, 0.22 Si, 0.72 Mn, <0.010 P, 0.022 S, 0.13 Cr, 0.13 Ni, 0.18 Cu, and balance Fe were used as reinforcements.

Three soluble phosphates were evaluated: MFP (Na_2PO_3F) 95% pure Aldrich, DHP (Na_2HPO_4) 99% pure Panreac, and TSP ($Na_3PO_4{\cdot}H_2O$) 97% pure Panreac.

Two types of samples were tested: MCI and ACI specimens. Both had dimensions of 20 mm × 55 mm × 80 mm and incorporated embedded steel reinforcement bars.

To assess MCI efficiency, mortar specimens were prepared using a blend of OPC, siliceous sand (SiO_2, 99% pure) and water with a water/sand/cement ratio of 0.5/3/1. Mortar specimens were used instead of concrete to avoid the formation of large pores by the gravel addition, which could lead to high water content within the pore network and also affect the pathways for chloride and phosphate transport. Two 8 mm diameter carbon steel bars were symmetrically embedded lengthwise in the specimens to act as working electrodes and an external stainless steel cylinder of 50 mm in diameter was placed on top of the specimens to act as counter electrode, with a central drill-hole that allowed the use of a saturated calomel electrode (SCE) as reference. An active surface area of 10 cm^2 was marked on the working electrodes with adhesive tape, thus isolating the triple mortar/steel/atmosphere interface to avoid possible localised corrosion attack due to differential aeration phenomena. The fresh mortar was poured immediately into moulds and cured in conditions of ~95% relative humidity (RH) for 28 days at room temperature. After curing, the MCI specimens were fully immersed for 28 days in 0.2 M aqueous solutions of each studied phosphate (MFP, DHP and TSP) or for 28 days in distilled water (blank experiment). It is worth to note that the specimens were fully moist prior to the immersion in the phosphates solution. Finally the specimens were immersed in a 3% NaCl aqueous solution and measurements were performed up to 70 days at room temperature.

The ACI mortar specimens were prepared in the same way as the MCI mortar specimens but adding 3% weight of each phosphate compound (MFP, DHP or TSP) to the OPC powder. A blank specimen without phosphate was also prepared. After curing all the specimens at room temperature for 28 days (~95% RH), they were immersed in a 3% NaCl aqueous solution.

Corrosion behaviour over time was monitored using the electrochemical corrosion potential (E_{corr}) and linear polarisation resistance (LPR) techniques. In LPR tests, the reinforcing steels were polarised to ±10 mV vs. E_{corr} with a scan rate of 0.166 mV s^{-1}. This allows the steel corrosion current density (i_{corr}) to be calculated using the formula: $i_{corr} = B/R_p$, where B is a constant and R_p is the value obtained by LPR. To calculate i_{corr}, B values of 26 and 52 mV were used for specimens with E_{corr} in the active and passive zone, respectively [23]. An EG&G Parc potentiostat model 273A was utilised for electrochemical measurements.

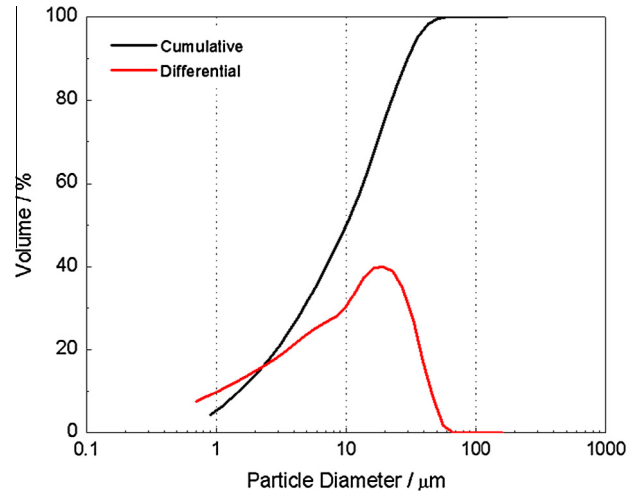

Fig. 1. Granulometry distribution of the tested ordinary Portland cement (OPC) obtained using the laser ray diffraction method.

3. Results

Fig. 2 shows the E_{corr} vs. time for steel bars embedded in MCI mortar specimens. The E_{corr} values are situated at levels of low, medium or high risk of corrosion. Even so, the E_{corr} parameter can be used to define the probability of corrosion in these specimens: for $E_{corr} < -0.27$ V vs. SCE the probability of active corrosion is high (~90%), for -0.27 V $< E_{corr} < -0.12$ V vs. SCE corrosion is uncertain, and for $E_{corr} > -0.12$ V vs. SCE there is a 10% probability of corrosion ASTM C 876 [24]. The E_{corr} values for all the MCI mortar specimens in the presence of inhibitor are less negative (noble) that for the blank specimen, except in the case of the specimens immersed in the aqueous TSP solution, which presented a high risk of active corrosion, at least from a qualitative point of view. The specimens immersed in the aqueous MFP solution present the most noble corrosion behaviour, with the least negative E_{corr} value. Accordingly, the E_{corr} values indicate that MFP or DHP may be considered anodic inhibitors that reduce the corrosion rate by increasing the E_{corr} value, for the 0.2 M inhibitor concentration tested.

Fig. 3 shows the i_{corr} vs. time for steel bars embedded in MCI mortar specimens. The i_{corr} values are situated at levels of low or medium risk of corrosion. The dotted line indicates the approximate limit for passive steel values, ⩽0.1 μA cm^{-2} [25]. The best corrosion inhibitor behaviour is shown by the MFP and DHP compounds, with i_{corr} values of <0.1 μA cm^{-2}, typical of the passive state. The blank specimen was actively corroding.

Fig. 4 displays E_{corr} vs. time for steel bars embedded in ACI mortar specimens. As with the MCI specimens (Fig. 2), the best corrosion inhibitor behaviour is shown by the MFP compound, with E_{corr} values at the medium and high risk of corrosion level. The DHP and TSP compounds show E_{corr} values at the high risk of corrosion level. E_{corr} values for the blank experiment were between −0.3 and −0.5 V vs. SCE, similar to those obtained with the DHP

Table 1
Chemical composition of tested ordinary Portland cement (OPC).

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	MnO	TiO_2	K_2O	Na_2O	SO_3	Ignition loss
Mass %	20.33	3.40	4.68	57.84	1.51	0.10	0.09	0.72	0.51	7.26	3.42

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