



Influence of zinc molybdenum phosphate pigment on coatings performance studied by electrochemical methods



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ABSTRACT

The effect of the addition of the zinc molybdenum phosphate pigment, on the corrosion resistance properties of powder coating on steel, has been investigated by means of different electrochemical methods: electrochemical impedance spectroscopy (EIS) and the accelerated cyclic electrochemical technique (ACET). Anticorrosive properties obtained from both techniques showed similar results. Powder coatings incorporating this pigment have shown better performance which is attributed to both barrier properties enhancement and inhibition action of the pigment. This behavior can be correlated to the results obtained in the pigment extract study (polarization test and EIS). Finally, salt fog spray test was performed to confirm the electrochemical tests results.

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1. Introduction

Using coatings and paints is the most widely used method to protect metals from corrosion. The protection mechanism of each coating is determined by several factors but can be summarized in three effects that act combined: physical barrier (hindering diffusion pathways of water and oxygen to the substrate), chemical inhibition and electric resistance [1,2].

Pigments are one of the principal components of a coating formulation and part of their function is to strengthen the physical and chemical properties of the coating acting as corrosion inhibitors [3]. Zinc molybdenum phosphate seems to have become into an excellent alternative to toxic inhibiting pigments due to its similar anticorrosive behaviour to chromates and better than non-modified zinc phosphates. This pigment belongs to the second generation of zinc phosphates that are obtained by applying an organic surface treatment to the particles, designed to enhance the continuity between the inorganic pigment and the surrounding organic binder [4,5].

Zinc molybdenum phosphate pigment produces a molybdate anion (MoO_4^{2-}) that acts as an effective anodic inhibitor [6,7] whose capacity to passivate is only slightly lower than the one of the chromate anion [8]. Among all the molybdate-based pigments, zinc molybdenum phosphate is one of the highest reported due

to a synergistic effect between the phosphate and the molybdate ions [9,10]. According to the literature [11], this dual metal pigment provides higher degree of passivation than zinc chromate under acidic conditions but inferior under alkaline conditions. Although there have been several studies to investigate the anti-corrosion efficiency of pigments, most of them are comparative studies of extracts for different types of pigments [12–14] or comparative studies for coatings formulated with such inhibitors [15]. Only few studies are focused on a complete electrochemical investigation to establish the mechanism of action of a specific pigment [16].

In despite the fact that the application of electrochemical impedance spectroscopy (EIS) to coated metals has been shown to be a useful technique in the study of the performance of pigments and coatings [17–23], there is still a great interest in creating rapid assessment methods for practical applications in order to provide faster indications of corrosion processes in the surface and the interface of coated metallic substrates. Hollaender et al. [24–26] developed a rapid method for testing coated metals in food packaging which consists on a combination of cathodic polarization (DC) and EIS measurements (AC). The accelerated cyclic electrochemical technique (ACET) [27–32] is based on the Hollaender method but uses a new potential relaxation step following each cathodic polarization, which is used to evaluate the adhesion of the coating to the substrate. This technique has been used in previous works to study the effect of corrosion inhibitors and other additives that promotes adherence in epoxy, alkyd and polyester powder coatings.

The aim of this paper is to investigate the inhibition mechanism of zinc molybdenum phosphate using electrochemical tests

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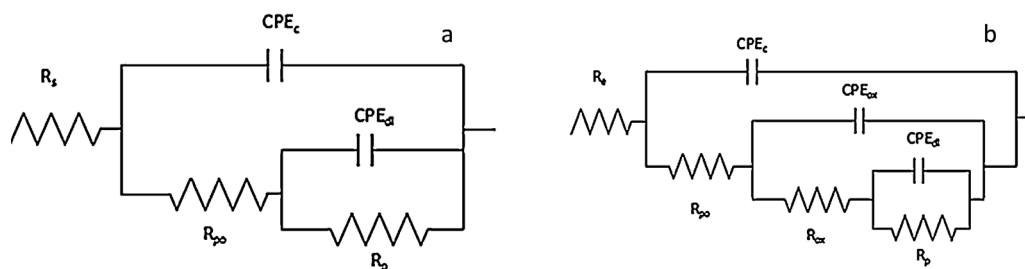


Fig. 1. Equivalent circuits used to model EIS and ACET impedance data where passive parameters (R_s = electrolyte resistance, R_{po} = pore resistance, CPE_c = constant phase element of the coating capacitance, R_p = polarization resistance, CPE_{dl} = constant phase element of the double layer capacitance, R_{ox} = resistance of corrosion products, CPE_{ox} = constant phase element of corrosion layer) can be defined.

Table 1
Composition for Nubirox 106.

Compound	% (Weight)
% Trizinc bis(orthophosphate)	87–97
Zinc Oxide	1–10
Organic titanate	1–5
Molybdenum and zinc tetraoxide	0.5–5

for both bare steel samples in the pigment extracts in 3.5 wt.% NaCl solution and for pigmented polyester powder coatings.

2. Experimental

2.1. Anticorrosive pigment

Pigment was obtained from Nubiola® (Nubirox 106) and it is based on a mixture of zinc phosphate and zinc molybdate conditioned with an organic titanate in the surface. The chemical composition of the pigment is described in Table 1.

2.2. Pigment extract preparation

5 g of zinc molybdenum phosphate pigment in 250 g of distilled water were used. The solution was stirred for 24 h at 800 rpm and then filtered to obtain the pigment extract. The solubility of the pigment was 32 mg/100 mL, determined from the measurement of the weight of the dried residue of the pigment extract. Finally, 3.5 wt.% NaCl was added to the total pigment extract and then used as a test solution.

2.3. Powder coating formulations/Sample preparation

The coatings were developed from a saturated carboxylate polyester resin of low molecular weight (Reafree 8585 from Cray Valley Iberica, S.A.) to combine with a hydroxyalkylamide crosslinker (Primid-XL 552 from EMS-GRILTECH). Other inorganic fillers and additives used were titanium dioxide (Kronos 1171), barite (R-2 from Miber Minerales Roset), a flow agent (Additol from Cytec-Liquid Coatings Resins & Additives), a levelling agent (Cray-Vallac-PC from Cray-Valley Ibérica), a degassing agent (benzoin from DSM Special Products), a surface hardener (Licowax PE520) and a Teflon wax (Ceridust 9610F from Clariant).

The components of coatings A and B (Table 2) were pre-mixed and shaken by hand until a good premixing was obtained. Subsequently, the mixture was extruded in a double screw extruder (Werner & Pfleiderer ZSK25) and the temperature profile was set at 40, 120, 120 and 70 °C (rear to front along the extruder). The material produced was ground in an ultracentrifugal mill ZM 100 and sieved at 140 nm; thus, obtaining the different powder coatings ready for study. Coating A is used as a control sample and Coating B replaces barium sulphate with the anticorrosive pigment.

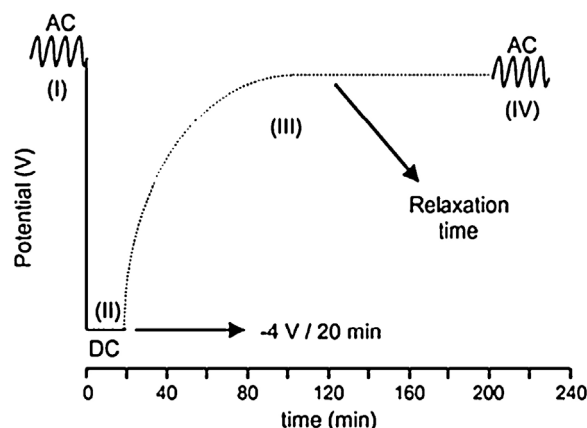


Fig. 2. Scheme of the ACET test versus time.

Table 2
Coatings formulation.

Material	A	B
Resin	46.2	46.2
Crosslinker	2.6	2.6
TiO ₂	30	30
Barium sulphate	16.9	1.9
Degassing agent	3.0	3.0
Levelling agent	0.5	0.5
Benzoin	0.3	0.3
Polyamide wax	0.3	0.3
TFPE wax	0.2	0.2
Zinc molybdenum phosphate	–	15.0
PVC	19	20
PVC/CPVC	50	47
λ	0.38	0.43

The carbon steel panels (100 mm × 150 mm × 1 mm) were provided by Espancolor S.L. having an average surface roughness below 0.50 microns according to the supplier. The formulations were deposited on the metallic substrates, previously degreased with acetone, by means of an electrostatic gun. All the coated samples were cured at 180 °C for 15 min and the thicknesses obtained were 90 ± 10 μm.

2.4. Testing methods and equipment

2.4.1. Linear polarization

Potentiostatic polarization measurements were performed on an Autolab PGSTAT30 potentiostat with a three electrode system. The electrochemical cell was obtained by sticking a glass cylinder on the sample sheet and filling it with the test solution. The exposed surface area was 9.62 cm². A three-electrode system was used, in which the sample without coating acts as working electrode, a car-

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