A comparative study of anticorrosive paints containing polyphosphates

Beatriz del Amo[1], G. Blustein[2], Marta Cecilia Deyá [2], Roberto Romagnoli[3]

Abstract

The objective of this work was to study the efficiency of the anticorrosive pigments obtained by substituting the phosphate anion in zinc phosphate by other polyphosphate anions such as tripolyphosphate and pyrophosphate. A procedure to prepare the pigments is outlined and their anticorrosive properties were evaluated following the electrochemical behaviour of a steel electrode in pigments suspensions. Two resins were chosen as film forming materials: an alkyd and an epoxy one. The performance of the resulting anticorrosive paints was assessed by accelerated (salt spray and humidity chambers) and electrochemical tests (corrosion potential and ionic resistance). It was demonstrated that the substitution of the phosphate anion by tripolyphosphate or pyrophosphate anions, may give rise to effective anticorrosive pigments for paints. The essays performed with pigments suspensions suggested that zinc pyrophosphate has the best anticorrosive properties. The anticorrosive behaviour of the pigments in paints were found to depend on the resin employed to formulate the coatings.

Key words: corrosion, coatings, phosphate , tripolyphosphate, pyrophosphate, accelerated tests, electrochemical tests.

Introduction

Organic coatings are an inexpensive and effective way to protect metals against corrosion. However, current anticorrosive formulations contain lead or hexavalent chromium compounds which contaminate the environment. Regulations in different countries, against the employment of toxic compounds, produced an intense research to find out alternative non - toxic anticorrosive pigments.

The use of inorganic phosphates in anticorrosive paints formulations is widespread [1]. The protection afforded by zinc phosphate rests upon the formation of an iron oxyhydroxides film on the steel surface [2, 3]. The mechanism of protection would also involve the polarisation of the cathodic areas due to the precipitation of basic insoluble salts on the steel surface. These salts may contain the phosphate anion in their composition [2, 4, 5]. Zinc phosphate has been used with different binders [2, 4, 5] but with contradictory results [6-8]. Its performance in accelerated tests was not so good as in long-term ones (outdoor exposure) [7-11]. Many attempts have been made to modify zinc phosphate because it seems that it can not

[1] Doctor en Química – CIDEPINT-CONICET
[2] Doctor en Química – CIDEPINT-CONICET-UNLP
[3] Doctor en Química – CIDEPINT-CONICET-UNLP-UNNOBA

replace completely to zinc chromate [12]. These modifications are concerned with particle size reduction, the incorporation of aluminium or molybdenum, the application of organic treatments on the pigment particle surface, etc. [13, 14]. Other phosphate pigments were also developed such as calcium acid phosphate, aluminium tripolyphosphate, etc. [15-21].

The aim of this paper is to compare the anticorrosive behaviour of the pigments obtained by substituting the phosphate anion in zinc phosphate, by other anions such as tripolyphosphate and pyrophosphate. Pigments inhibitive properties were assessed by electrochemical techniques, employing pigment suspensions. The characteristics of the protective layer were established by scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDXA). Their anticorrosive behaviour was also evaluated in alkyd and epoxy paints, in comparison with that of zinc phosphate, by accelerated and electrochemical tests.

EXPERIMENTAL AND RESULTS

Experimental

Pigments preparation

Zinc phosphate was prepared by mixing 1.3 M zinc nitrate solution and 1.4 M phosphoric acid. Both solutions were dropped simultaneously into a glass beaker containing 500 mL of water. Finally, the pH of the solution was adjusted to 5.7.

Zinc tripolyphosphate was also obtained in the laboratory by precipitation from 0.1 N sodium tripolyphosphate (pH = 4) and 0.1 N zinc nitrate solutions at the same pH. Both solutions were dropped simultaneously, into a glass container and the final pH of the solution was adjusted at 5.1 by adding a 20% sodium hydroxide solution.

Zinc pyrophosphate was precipitated from 0.7 M sodium pyrophosphate solution, acidified with 65% by weight nitric acid (density 1.4 g/ml), till a pH value of 4.5 was attained. Then, this solution was dropped simultaneously with the 0.7 M zinc nitrate solution in a glass beaker containing 500 ml of water. Finally, the pH of the medium was adjusted with 20% sodium hydroxide to a pH value corresponding to the change in colour of the bromcresol green acid – base indicator (3.8 - 5.6).

Once the pigments precipitated, they were filtrated, washed three times thoroughly with distilled water, dried in air, and washed again twice by suspending them in distilled water. Zinc tripolyphosphate and zinc pyrophosphate must not be dried in stove because the anions undergo self-hydrolysis. The hydrolysis rate of zinc tripolyphosphate dependes on the temperature and the pH of the medium [22].

Pigmentent characterisation and the evaluation of their inhibitive properties

The composition of the pigment was determined by current analytical techniques (Table I). The composition of the saturated solution was also determined by the same way.

The inhibitive properties of the pigments were assessed by measuring the corrosion potential of a SAE 1010 steel electrode in the pigments suspensions during 24 hours. The saturated calomel electrode (SCE) was used as reference and the electrolyte was a 0.025 M sodium perchlorate solution. Steel polarisation resistance (with IR-drop compensation) was determined, in the same pigments suspensions in 0.025 M sodium perchlorate as supporting electrolyte. The reference electrode was SCE and the counterelectrode a platinum grid. The sweep amplitude was ±20 mV and the scan rate 0.166 mV/s. Measurements were taken employing a Model 273A EG&G PAR Potentiostat/Galvanostat plus SOFTCORR 352 software. The protective layer formed after 24 hours of exposure to the pigment suspension was examined by SEM and EDXA.

Anodic and cathodic polarisation curves of the SAE 1010 steel electrode in pigments suspensions in 0.5 M sodium perchlorate solution were obtained at different exposure times (2, 6 and 24 hours). The cell employed to carry out these measurements was similar to that employed to determine the polarisation resistance. The swept began in the vicinity of the corrosion potential, at a scan rate of 3 $mV.s^{-1}$.

In all cases the electrode surface was abraded with nº 600 emery paper and experiments were carried out at room temperature (20 $\pm$ 2 ºC) in normally aerated solutions.

Paints composition, manufacture and application

The film forming materials were: medium oil alkyd (50 % linseed oils, 30 % o-phtalic anhydride, 8 % pentaerythritol and glicerol and 12 % pentaerythritol resinate) and an epoxy-polyamide resin. The solvent employed in the former case was white spirit while the mixture xilene/methyl isobutyl ketone/ butyl cellosolve (13/45/42 % w/w) was used for the epoxy paint. The pigment content employed to formulate the paints was 30% by volume (v/v), with respect to the total pigment concentration. Titanium dioxide, 11.6 v/v; barium sulphate (barite), 29.2% v/v and talc, 29,2 % v/v, were incorporated to complete the pigment formula. Pigments were dispersed in the vehicle, employing a ball mill with a 3.3 litres jar, for 24 hours, to achieve an acceptable dispersion degree [23]. The PVC/CPVC relationship was 0.8.

SAE 1010 steel panels (15.0 x 7.5 x 0.2 cm) were sandblasted to Sa 2 1/2, degreased with toluene and then brushed up to a thickness of 75 $\pm$ 5 μm, the panels were kept indoors for 7 days before being tested.

The performance of anticorrosive paints in accelerated and electrochemical tests

A set of 3 panels was placed in the salt spray chamber (ASTM B 117). Rusting (ASTM D 610) and blistering (ASTM D 714) degrees and the failure at the scratch mark (ASTM D 1654) were evaluated periodically. The adhesion of the coatings to the steel substrate was measured by the cross-cut tape test (ASTM D 3359) at the beginning of the test period and after 48, 170 and 1440 hours of exposure to the salt spray chamber.

Another set of panels was placed in the humidity chamber at 38 ± 1°C (ASTM D 2247). Rusting and blistering degrees were assessed as described above. The panels exposed to the humidity chamber (ASTM D 2247) were evaluated after 144 and 500 and 2000 in the case of the alkyd paints and after 500 and 2000 hours in the case of the epoxy paints.

The cells to perform electrochemical measurements were constructed by delimiting 3 cm^2 circular zones on the painted surface by placing an acrylic tube, 7.0 cm high, on the painted specimen and filling it with the supporting electrolyte (0.5M sodium perchlorate solution). The ionic resistance between the coated steel substrate and a platinum electrode was measured employing an ATI Orion, model 170, conductivity meter at a 1000 Hz frequency. The corrosion potential was monitored against a reference electrode (SCE) with a high impedance voltmeter.

Results and discussion

The pH of the saturated solution of zinc phosphate and zinc pyrophosphate were close to neutrality while that of zinc tripolyphosphate was noticeable lower. Zinc phosphate and, in second term, zinc tripolyphosphate exhibited the highest concentration of soluble phosphate in the satured solution (Table II). Higher phosphate concentration in the saturated solution may be useful to improve anticorrosive protection of steel. As it would be seen later, the higher concentration of phosphate anion generated by zinc phosphate, in combination with the more impervious film of epoxy paints, would result in an improved protection of the steel substrate.

According to corrosion potential measurements, all pigments inhibited steel corrosion. In this sense, zinc phosphate shifted the corrosion potential towards more positive values with respect to other pigments. Zinc tripolyphosphate behaved as classical anticorrosive pigments (i.e.: chromates) did [24]. It exhibited a broad peak pointing to positive values. The corrosion potential began to decrease steadily beyond 150 minutes of exposure. Zinc pyrophosphate, as it occurred with zinc phosphate, maintained the corrosion potential almost constant during the whole test. After 24 hours of exposure the corrosion potential of the steel panel in contact with zinc phosphate was measured and found to be -690 mV while that of zinc tripolyphosphate was -622 mV and that of zinc pyrophosphate was –531 mV. However, no signs of corrosion were detected in any case. This led to think that the corrosion protection was achieved through an iron oxide layer. This statement was lately confirmed by SEM observations. After 24 hours of exposure to the pigment suspension, the most effective pigment, to restrain corrosion, seemed to be zinc pyrophosphate.

The polarisation resistance of SAE 1010 steel in pigment suspensions resulted higher than the polarisation resistance of SAE 1010 steel in the supporting electrolyte, which was found to be equal to 0.2 $k\Omega cm^2$. Zinc pyrophosphate exhibited the highest value (19.0 $k\Omega cm^2$) which indicated a better protection of the steel substrate. Zinc tripolyphosphate had an intermediate value (2.1 $k\Omega cm^2$) while zinc phosphate exhibited the lowest value (1.1 $k\Omega cm^2$). From these values, steel corrosion rate was calculated and found to be equal to 2.00 x 10^{-3} $\mu A.cm^{-2}$ in the case of zinc phosphate, 1.05 x 10^{-3} $\mu A.cm^{-2}$ for zinc tripolyphosphate and 1.16 x 10^{-4} $\mu A.cm^{-2}$ when zinc pyrophosphate was the anticorrosive pigment. The higher values of steel polarisation resistance encountered for anticorrosive pigments, with respect to that of steel in the supporting electrolyte, were attributed to the nature of the protective film formed on steel.

As it was said previously, the protective film formed by the reaction between steel and the anticorrosive pigment was examined by SEM and its composition determined by EDXA.In the case of zinc phosphate, the protective layer was mainly composed of non expansive iron oxide and, in much less proportion, zinc hydroxide. As an example, the protective layer formed by the action of zinc phosphate is shown in Fig. 2. A light grey film was formed due to the reaction between steel and zinc tripolyphosphate after 24 hours exposure. It was constituted by a rather compact layer in which the superficial amount of zinc was found to be equal to 1.75%, the phosphorous amount was 2.92%, being the rest iron. No corrosion signs were detected. The protective layer generated by zinc pyrophosphate was also constituted by a rather compact oxide layer with some globular formations. These formations appeared to be constituted principally by iron oxide (90,70 %) stabilised with phosphorous pentoxide (7.45%)

and zinc oxide (1.84%). It was thought that the presence of these films increased polarisation resistance which, in turn, resulted in a lower corrosion rate.

From the analysis of the anodic polarisation curves, obtained after 2 hours of exposure, it must be concluded that the most effective pigments to restrain steel corrosion were zinc tripolyphosphate and zinc pyrophosphate. These pigments not only exhibited the lower critical current density to achieve passivation but they also show the lowest current density in the passive region. It is interesting to point out that steel oxidation became very much polarised in the presence of zinc pyrophosphate resulting this fact in a better protection of the metallic substrate. As time went on, steel became a bit more active in the case of zinc phosphate and zinc tripolyphosphate. The peak current of the curve for zinc pyrophosphate, after 1 day, appeared displaced to very high positive potentials.

The examination of the cathodic polarisation curve, obtained after 2 hours of testing, revealed that the oxygen reduction current resulted diminished in the presence of inorganic phosphate, as it was stated in the literature [2, 5]. The oxygen reduction current in the supporting electrolyte was, as an average, after 6 hours of exposure, 3.0 $mA.cm^{-2}$ while in the presence of pigments it was, in the worst case, half of the value observed for the supporting electrolyte. These rather high cuurrent density values were originated in oxides reduction coupled with the oxygen reaction. The most important reduction in the oxygen current was detected when zinc tripolyphosphate was the anticorrosive pigment. The reduction of oxides formed as a consequence of the corrosion process may be clearly appreciated before hydrogen evolution. As time elapsed the oxygen reaction became slightly polarised and the oxide layer seemed to increase in thickness because oxide reduction current also increased. This behaviour was more striking for zinc pyrophosphate which seemed to develop a thicker protective layer.

Alkyd paints developed blisters after 650 hours in the salt spray cabinet (Table III). Severe blistering was observed in the case of the paint pigmented with zinc phosphate which also showed the lower corrosion resistance. The best anticorrosive performance was observed for the alkyd paint containing zinc pyrophosphate. Only the paint pigmented with zinc pyrophosphate showed an acceptable anticorrosive behaviour at the end of the test period. Zinc pyrophosphate also showed an excellent behaviour at the scribe (Table III).

Epoxy paints did not blister during the whole test period (Table IV). The anticorrosive behaviour of all paints was very good up to 1100 hours of testing. After 2300 hours of exposure zinc phosphate and zinc tripolyphosphate still showed an acceptable behaviour while zinc pyrophosphate had a lower performance.

The behaviour at the scribe showed the same tendency observed for rusting. No important changes were observed up to 1100 hours. The best performance at the end of the test period was observed for zinc pyrophosphate (Table IV).

The adhesion of the alkyd paints to the steel substrate (Table V) was lost after the first week of exposure to the salt spay cabinet. The paint with zinc tripolyphosphate lost all the adhesion after two days. On the contrary, no loss of adhesion was detected in the case of the epoxy paints, after two month of exposure. The anticorrosive action of the pigments is clearly seen in the case of alkyd paints. As it was said previously, these paints lost their adhesion to the substrate after 170 hours of exposure but they continued protecting steel beyond 650 hours.

All the alkyd paints blistered strikingly after a week of exposure to the humidity chamber (Table VI). Only the alkyd paint pigmented with zinc phosphate exhibited rust spots after 500 hours, the others remained oxides free even after 2000 hours in the cabinet. The most resistant coating was that pigmented with zinc tripolyphosphate.

The epoxy paints formulated with zinc phosphate and zinc tripolyphosphate showed neither rust spots nor blisters after 2000 hours in the humidity chamber (Table VII). The paint containing zinc pyrophosphate attained a rust degree of 7, but no blisters appeared on the surface.

The alkyd paint containing zinc pyrophosphate showed the best performance while that containing zinc phosphate exhibited the lowest anticorrosive properties. In change, with the epoxy binder, the best performance was achieved when zinc phosphate and zinc tripolyphosphate were the active pigments; however, the differences are not so big and became perceivable after 2300 hours of exposure.

The morphology and composition of the protective layer developed during the exposure to the salt spray chamber was studied by SEM and EDXA after removing the coating. In all cases, as general rule, it was observed the growth of the amorphous oxide layer reported in the case of the reaction among pigments suspensions and the steel substrate. Sometimes the growth of this layer was accompanied by the formation of other differential structures which may contain phosphorous and zinc together with large amounts of iron oxides.

The corrosion potential of alkyd paints varied jointly with the ionic resistance (Fig.5). As the ionic resistance had high values the corrosion potential remained displaced towards more positive values. In this sense, the alkyd paint containing zinc pyrophosphate showed the best performance indicating that the protection afforded by the paint was principally due to its barrier properties rather than to the anticorrosive properties of the pigment itself.

A similar tendency was observed with the epoxy paints (Fig. 5). In this case, the best behaviour was observed for the paint containing zinc phosphate, while the lower performance was attributed to zinc pyrophosphate paint.

All tested paints showed a certain barrier effect after 1 day of exposure, $Ri > 10^6\ \Omega.cm^2$ [24]; full barrier effect is attained when $Ri > 10^8 \Omega.cm^2$ [24]. The ionic resistance of the alkyd paints pigmented with zinc phosphate diminished drastically after one day of exposure to the electrolyte solution (Fig. 3). In this sense, the alkyd paint containing zinc polyphosphate did not show important changes before ten days of exposure. On the other hand, the paint containing zinc pyrophosphate maintained a certain barrier effect beyond three weeks of immersion.

All the paints had the same PVC/CPVC ratio and the same pigment content; however, the lowest permeability was achieved with zinc pyrophosphate. This lower permeability may be due to some kind of pigment-binder interaction. This property was reflected in the better corrosion resistance of this paint in the salt spray test.

The epoxy paints formulated with zinc phosphate and zinc polyphosphate developed and maintained an acceptable barrier effect ($Ri > 10^7 \Omega.cm^2$) during the whole test period. On the contrary, the paint pigmented with zinc pyrophosphate lost its barrier properties after the first day of immersion (Fig. 5).

CONCLUSIONS AND ACKNOWLEDGEMENTS

Conclusions

1. The substitution of the phosphate anion by other anions such as tripolyphosphate or pyrophosphate anions, may give rise to effective anticorrosive pigments for paints.

2. The essays performed with pigments suspensions suggested that zinc pyrophosphate has the best anticorrosive properties. The lowest anticorrosive performance corresponds to zinc phosphate.
3. All the pigments inhibited steel corrosion by generating a protective layer mainly composed of non expansive iron oxides.
4. The best anticorrosive behaviour in alkyd paints was obtained with zinc pyrophosphate, while in the case of epoxies the best anticorrosive performance was attained with zinc phosphate and zinc tripolyphosphate.

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Table I. Pigments Compositions as % by weight

Ionic components of the pigments	Pigments		
	Zinc phosphate	Zinc pyrophosphate	Zinc tripolyphosphate
Phosphate (PO_4^{3-})	52.5	49.4	49.3
Zinc (Zn^{+2})	36.3	31.7	25.2
Potassium (K^+)	8.7	-----	-----
Sodium (Na^+)	-----	0.7	2.1
Loss at 100°C	2.5	18.2	23.4

Table II. Chemical features of the pigments suspensions

Pigment	pH	Conductivity (µS)	Ionic concentration of the Saturated solution (mg/l)	
			Phosphate (PO_4^{3-})	Zinc (Zn^{+2})
Zinc phosphate	6.72	3610	1990	1.7
Zinc tripolyphosphate	5.75	174.5	547	89.2
Zinc pyrophosphate	6.97	112.1	24.1	4.6

Conductivity of 0.1 M KCl: 14630 µS

Table III. Rusting, blistering and failure for the alkyd paints in the salt spray cabinet

Paints	Hours								
	650			840			1100		
	R	B	S	R	B	S	R	B	S
Zinc phosphate	7	2D	5	---	---	---	---	---	---
Zinc tripolyphosphate	8	8D	5	7	6D	4	---	---	---
Zinc pyrophosphate	10	8F	10	10	8F	10	7	8F	10

Note: R: rusting; B: blistering; S: failure at scribe. Panels were taken out from the salt spray cabinet when the corrosion the qualification was lower than 7.

Table IV. Rusting, blistering and failure for the epoxy paints in the salt spray cabinet

Paints	Hours								
	650			1100			2300		
	R	B	S	R	B	S	R	B	S
Zinc phosphate	10	10	10	10	10	5	8	10	4
Zinc tripolyphosphate	10	10	7	10	10	7	7	10	6
Zinc pyrophosphate	9	10	10	9	10	10	6	10	8

NOTE: R: rusting; B: blistering; S: failure at scribe.

Table V

Adhesion (ASTM D 3359) of the alkyd paints in the salt spray cabinet (ASTM B 117)

Paints	Time (hours)					
	0		48		170	1440
	Alkyd	Epoxy	Alkyd	Epoxy	Alkyd	Epoxy
Zinc phosphate	5B	5B	5B	5B	2B	5B
Zinc tripolyphosphate	5B	5B	0B	5B	---	5B
Zinc pyrophosphate	5B	5B	5B	5B	1B	5B

Table VI. Rusting and blistering degree of the alkyd paints in the humidity chamber

Paint	Time (hours)					
	144		500		2000	
	Rusting	Blistering	Rusting	Blistering	Rusting	Blistering
Zinc phosphate	10	6MD	6	6MD	-----	-----
Zinc tripolyphosphate	10	8M	10	8M	10	8M
Zinc pyrophosphate	10	8MD	10	8D	10	6D

Table VII. Rusting and blistering degree of the epoxy paints in the humidity chamber

Paint	Time (hours)			
	500		2000	
	Rusting	Blistering	Rusting	Blistering
Zinc phosphate	10	10	10	10
Zinc tripolyphosphate	10	10	10	10
Zinc pyrophosphate	10	10	7	10

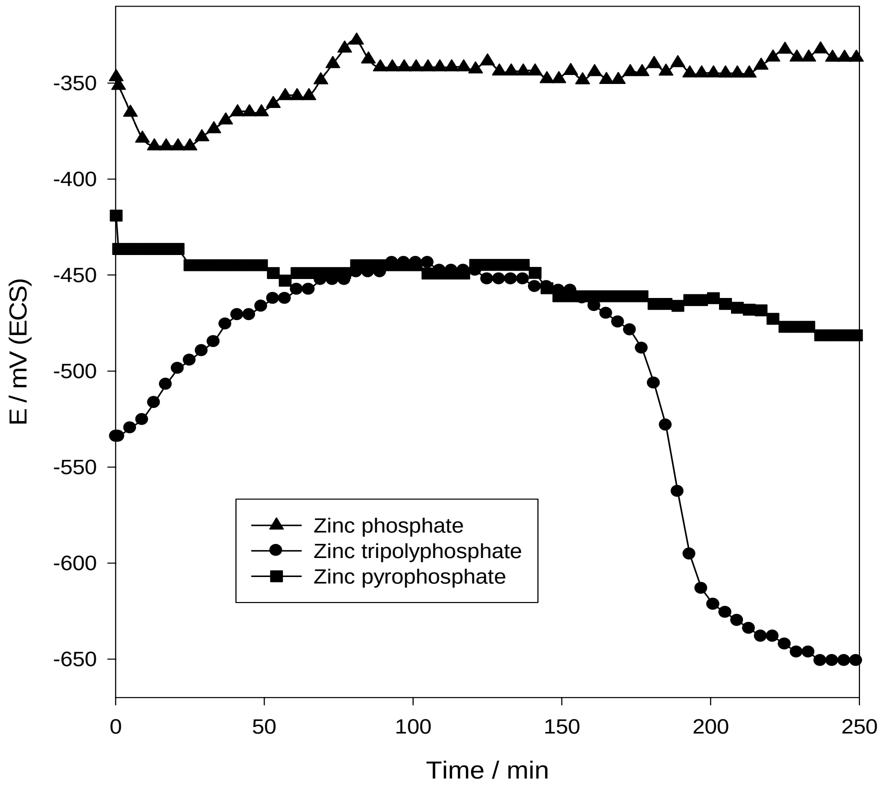

Fig. 1. Corrosion potential of SAE 1010 steel in pigment suspensions

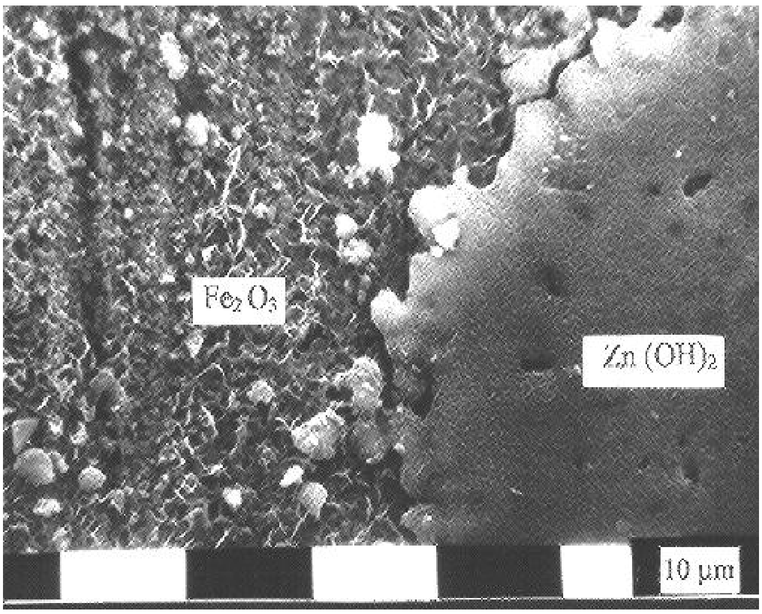

Fig. 2. SEM micrograph of the protective layer formed on SAE 1010 steel in zinc phosphate suspension

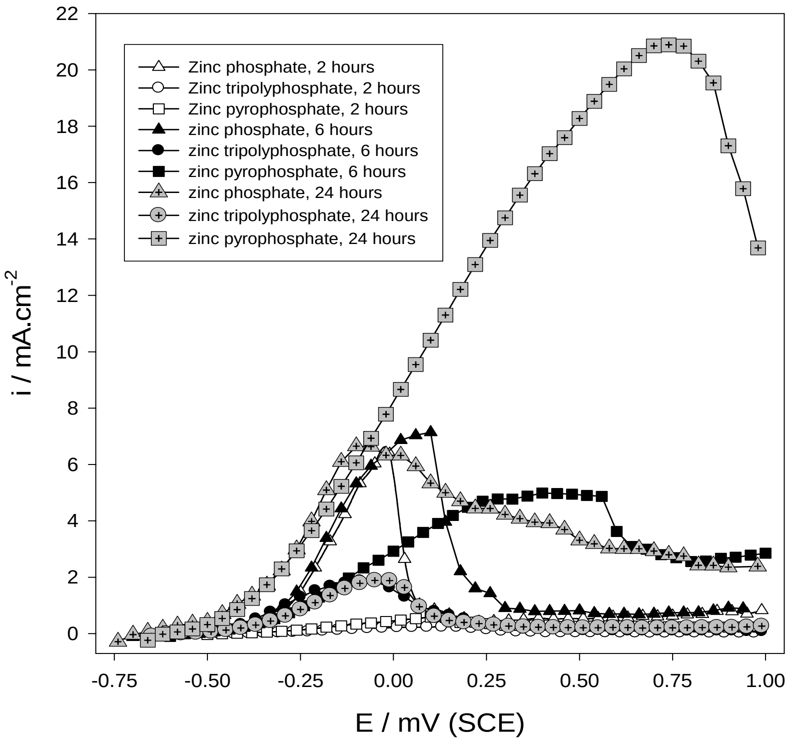

Fig. 3. Anodic polarization curves of SAE 1010 steel in pigment suspensions

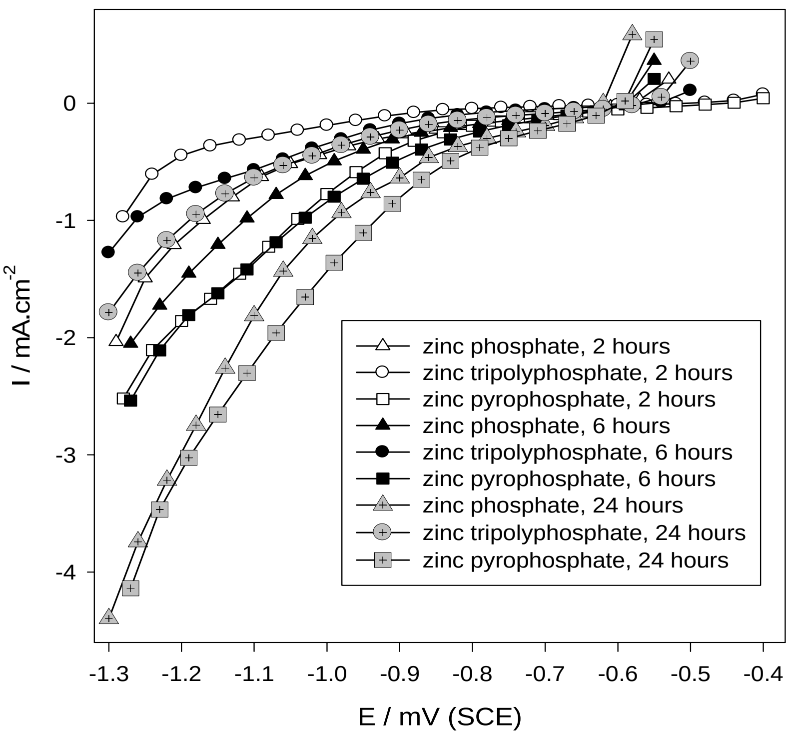

Fig. 4. Cathodic polarization curves of SAE 1010 steel in pigment suspensions

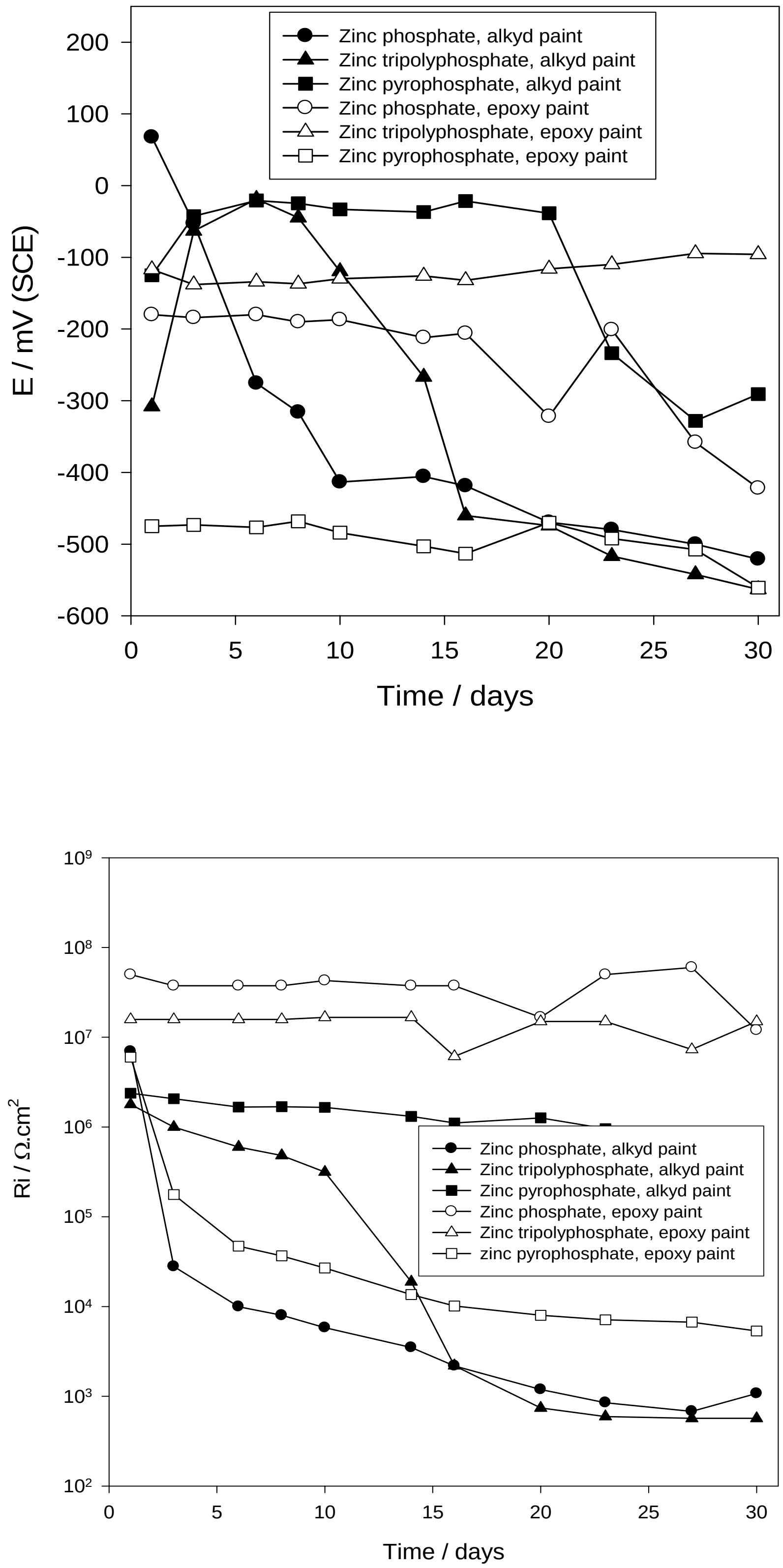

Fig. 5. Corrosion potential and ionic resistance of painted steel panels in NaClO4 0.5 M