



## Corrosion aspects of alkyd paints modified with linseed and soy oils

W.S. Araujo<sup>a</sup>, I.C.P. Margarit<sup>b,c,1</sup>, O.R. Mattos<sup>b,\*,1</sup>, F.L. Fragata<sup>d</sup>, P. de Lima-Neto<sup>e</sup>

<sup>a</sup> Federal University of Ceará, CT/DEMM, CE, Brazil

<sup>b</sup> Federal University of Rio de Janeiro, EP/COPPE/PEMM, RJ, Brazil

<sup>c</sup> Federal University of Rio de Janeiro, EQ/DPI, RJ, Brazil

<sup>d</sup> Electrical Energy Research Center, RJ, Brazil

<sup>e</sup> University Fed. do Ceará, CC/Dep. de Quím. Anal. e Físico-Química, CE, Brazil

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### ABSTRACT

The anticorrosive performance of medium-long (54–59%) alkyd paints modified with linseed and soy oils was compared by accelerated tests (Prohesion Cycle) and natural exposition in marine and industrial atmospheres. Differences on the protection mechanism of anticorrosive pigments due to substitution of linseed oil by soy oil were investigated by polarization curves and electrochemical impedance spectroscopy (EIS). Complementary tests such as water vapor and ions permeability in freestanding films were also performed. Results suggested that the type of oil influenced the barrier properties of the paint pigmented with zinc phosphate. The same tendency was verified by resistance values obtained from impedance diagrams. Polarization curves suggest that the action of the pigments in the alkyd paintings is practically the same for both oils. The substitution of linseed oil by soy oil did not impair the anticorrosive performance of alkyd paints and from the economic point of view this substitution could be very interesting.

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### 1. Introduction

Alkyd resins are commercially used to produce anticorrosive organic coatings since 1940 decade [1]. These resins are polyesters modified with vegetable oils. Linseed oil is the most used one due to its faster drying process which is related to higher insaturation degree, leading to reactions between oxygen and the resin to form a polymeric network [2,3]. Some studies about the properties of linseed oil based coatings were previously reported in the literature [4–7].

In Brazil, the industrial production of alkyd paints using linseed oil is limited, from the economic point of view, because this oil is imported, increasing the cost of the final product. In this context, soy oil appears as a suitable alternative because Brazil is one of the biggest producers of soybeans in the world. Alkyd paints formulated with soy oil are around 30% cheaper than alkyd paints containing linseed oil.

Actually alkyd paints modified with soy oil have been used for a long time, but data about the effects of such substitution on alkyd paints anticorrosive properties are not easily available. Specifically, it is not known whether soy oil changes adhesion, barrier properties, or the action of anticorrosive pigments. Therefore the main

purpose of this paper is to evaluate some anticorrosive properties of alkyd coatings formulated with soy oil compared with that of alkyd coatings formulated with linseed oil. Three different pigments were added to the alkyd paints for investigating differences on their protection mechanism due to substitution of the oil.

The barrier properties of the paints are evaluated by electrochemical impedance, water vapor permeation tests and permeability to ions. The inhibiting properties of the pigments are investigated by polarization curves in aqueous extracts of dry painting films and aqueous extracts of the pigments. The performance of the paints is evidenced in corrosion tests.

### 2. Experimental

#### 2.1. Samples preparation

Medium-long alkyd resins with the same oil content in their compositions (54–59%) were especially formulated for this work: one with soy oil (S) and other with linseed oil (L). The codes S and L refer to paints with soy and linseed oils, respectively. These resins were used for the preparation of two groups of paints. Each group consisted of three paints containing one of the following pigments: red lead (S1 and L1), zinc phosphate (S2 and L2) and red iron oxide (S3 and L3). Another formula was also made up only with filler (S4 and L4). The formulation criterion consisted of employing the same raw material maintaining constant the PVC/CPVC ratio (around 0.7)

\* Corresponding author. Tel.: +55 21 25628534x214; fax: +55 21 22906626.

E-mail address: [omattos@metalmat.ufrj.br](mailto:omattos@metalmat.ufrj.br) (O.R. Mattos).

<sup>1</sup> ISE member.

**Table 1**  
Basic description of tested paints (w/w).

Raw materials	S1	S2	S3	S4	L1	L2	L3	L4
Resin (soy)	17.5	37.9	38.4	41.6				
Resin (linseed)					17.6	37.6	38.9	40.9
Red lead	67.9				67.9			
Zinc phosphate		15.1				15.1		
Red iron oxide		3.0	10.0			3.00	10.1	
Agalmatolite		22.0	30.4	37.0		22.0	30.5	36.4
Additive	2.9	2.6	2.6	2.7	2.9	2.5	2.6	2.7
Solvent	11.7	19.4	18.6	18.7	11.6	19.8	17.9	20.0
PVC/CPVC	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7

for all paints. Basic formulation characteristics are presented in Table 1.

AISI 1010 steel with 15 cm × 10 cm was used as substrate. Before painting, the samples were degreased with toluene and blasted to white metal according to grade Sa3 of ISO 8501-1 [8]. Paints were applied by air-spraying in two layers, reaching a total dry thickness of 100 μm. The samples were intentionally scribed aiming at evaluating the undercoating corrosion progress. The scratch (60 mm) was made parallel to the smaller dimension at the bottom part of the samples.

Free films of the paints were prepared using an extender on Teflon sheets. After drying, the films were detached. The dry thicknesses of the films are indicated in the corresponding figures.

## 2.2. Corrosion tests

All tests were conducted at least with triplicate samples. The samples were tested in laboratory by Prohesion Cycle according to ASTM G85 [9] and by exposure to industrial and marine atmospheres. Samples were withdrawn from their corrosive environment for EIS measurements at regular intervals (4 months for the natural exposure tests and 250 h for the Prohesion Cycle).

Electrochemical experiments were performed using a potentiostat/galvanostat AUTOLAB PGSTAT 30, linked with a PC computer and controlled by the GPES 4.9 and by FRA software for data acquisition.

EIS measurements of painted steel were carried out in three electrodes cells after 3 h of immersion. This immersion time was necessary to obtain stationary diagrams. The area of each sample exposed to the electrolyte was 19 cm<sup>2</sup> chosen in the non-scratched part of the sample. A Pt grid and saturated calomel electrode (SCE) were used as auxiliary and reference electrodes, respectively. These impedance measurements were accomplished by potentiostatic control at the open circuit potential, in the 40 kHz–2.5 mHz frequency range, seven points per decade, and with 10–15 mV ac signal perturbation. The working electrolytes were 0.01 M Na<sub>2</sub>SO<sub>4</sub> solution for the samples exposed to industrial atmosphere, 0.01 M NaCl solution for those exposed to marine atmosphere and 0.008 M NaCl + 0.026 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution for samples tested in the Prohesion Cycle.

## 2.3. Complementary tests

In order to assess the corrosion inhibiting properties of the pigments, polarization curves were performed with the metal substrate in filtrates of aqueous extracts of each pigment and in filtrates of aqueous extracts of free films. Comparison between the two kinds of polarization curves should allow detecting when interactions with the resin have influence on the pigments anticorrosion action.

The extractions of the pigments were carried out by shaking 2 g of the pigment in 1 L of the 0.01 M NaCl or 0.01 M Na<sub>2</sub>SO<sub>4</sub> solutions during 4 h. Extracts were then filtered to remove the undissolved

matter. A similar method was previously employed by Amirudin et al. [10].

The extracts of the films were prepared according to an adaptation of the procedure used by Bethencourt et al. [11]. The extractions were carried out by shaking 15 g of the film triturated in 300 mL of the 0.01 M NaCl or 0.01 M Na<sub>2</sub>SO<sub>4</sub> solutions during 30 min at 60 °C. Extracts were then filtered to remove the undissolved matter. After the filtration, the solutions were utilized as electrolytes in the polarization cells.

Cathodic and anodic curves were obtained with each working solution by potentiodynamic polarization at 1 mV s<sup>-1</sup>, at room temperature. The working electrode was a 0.785 cm<sup>2</sup> disc of AISI 1010 steel embedded in epoxy resin. Prior to each branch of the polarization curves, the working electrode was polished with grid 400 emery paper, degreased with ethylic alcohol and washed in distilled water. Platinum foil was employed as auxiliary electrode. The ohmic drops in the polarization curves were corrected using the electrolyte resistance measured as the limit at high frequency of impedance.

The permeability of free films to water vapor was evaluated following ASTM D1653 [12], while permeability of the free films to Cl<sup>-</sup> was carried out in cells with two compartments separated by the films. In one compartment there was distilled water and in the other there was 0.1 M NaCl solution. The ion permeability was characterized by the crossing of the ions through the film, increasing the conductivity on the water side which was monitored by conductivity measurements with a Radiometer CDM83 Conductivity Meter. The dry thicknesses of the free films are indicated in the corresponding figures.

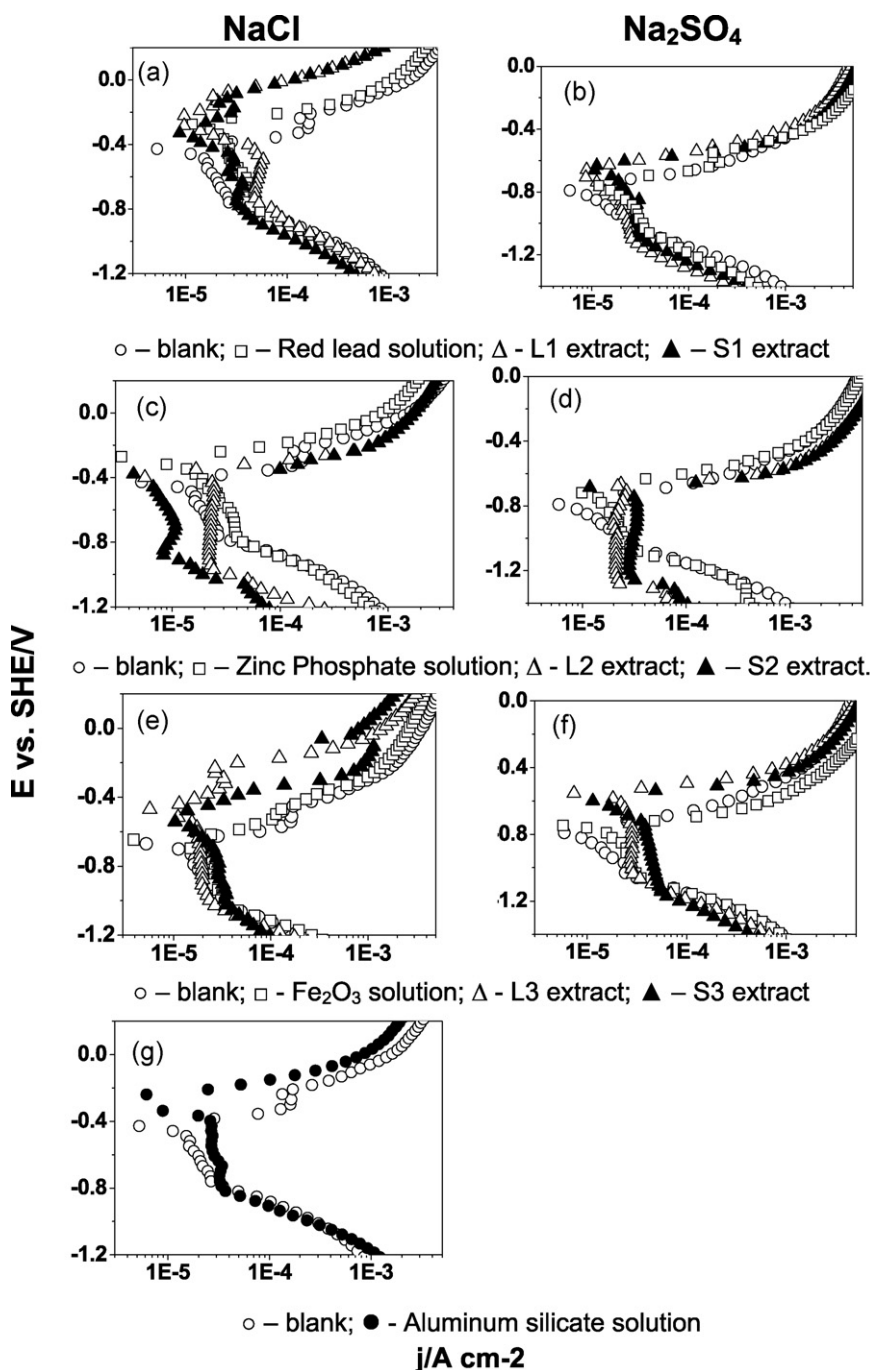
Impedance measurements of the free films were carried out in a two electrode configuration. The cell with two compartments separated by the free painting film was filled with 0.01 M NaCl solution. A platinum grid was placed in each compartment and the impedance measurements were made between these two electrodes with galvanostatic control at zero current.

## 2.4. Adhesion measurements

The adhesion of the dry paintings to the substrate after corrosion tests was measured according to ASTM D3359 B [13].

## 3. Results and discussion

The anticorrosive performance of any painting system depends on its barrier properties, inhibiting action of pigments and adherence. Concerning the pigments, the chosen ones were red lead, zinc phosphate and red iron oxide. Although red lead is not used anymore in paints industry [14], its inhibition mechanism is well known and it was included in this work to validate the adopted methodology. Red lead reacts with vegetable oils of the alkyd resin, forming lead soaps, which in contact with water and oxygen release inhibitors passivating the undercoating metal [15]. Some authors point out that zinc phosphate increases the barrier proper-



**Fig. 1.** Polarization curves of steel in 0.01 M NaCl or 0.01 M Na<sub>2</sub>SO<sub>4</sub> solutions (blanks), filtrates of aqueous extracts of the pigments and filler (pigment or filler solution) and aqueous extracts of the free films (Lx and Sx extract). Extract of the filler was obtained only in 0.01 M NaCl solution.

ties of organic coatings [16], while others state that zinc phosphate enhances adhesion, phosphating the base metal [17]. If so, the inhibition by zinc phosphate depends on its arrival at the metal coating interface by leaching. But as the solubility of zinc phosphate is very low, this limits its action as an inhibiting pigment [15,18,19] and there is controversy in literature about its efficiency [20]. Red iron oxide ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) is a non-toxic pigment without corrosion inhibiting properties [21]. The addition of this pigment to anticorrosive paintings aims to improve the throwing power. From paintings pigmented with red iron oxide only barrier properties are expected.

Kinetic effects of each pigment on the steel corrosion are characterized by polarization curves. Evidences of inhibiting action and dependence on interactions with the modified alkyd resin are obtained by comparison of the curves of the bare substrate plotted

in extracts of the dry painting films and extracts of the pigments. If the oil has any influence on such interactions, it can be evaluated by comparison of curves Sx (extracts of paintings with soy oil) and Lx (extracts of paintings with linseed oil).

The final conclusion about the influence of the oil is taken with corrosion tests where all aspects, including adherence to the substrate, are considered.

### 3.1. Influence of the oil on the action of anticorrosive pigments

In Fig. 1(a)–(f) anodic and cathodic polarization curves are shown for blanks, pigments aqueous extracts and respective free films aqueous extracts in chloride and sulphate media. The curves obtained with the blanks are identified by the electrolytes names:

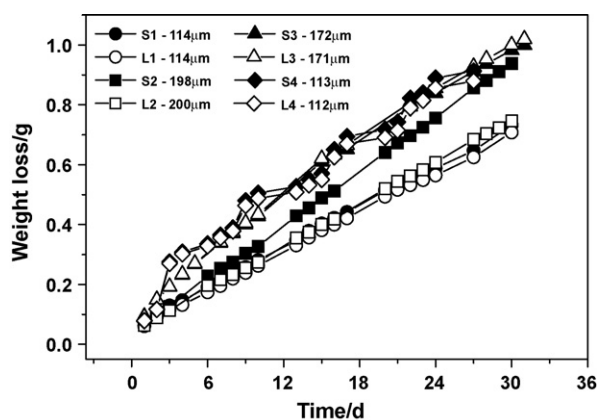


Fig. 2. Water vapor permeability according to ASTM D1653.

chloride or sulphate. The curves obtained with the pigments aqueous extracts are identified with the names of the pigments: red lead, zinc phosphate or iron oxide. Finally, the curves obtained with the films aqueous extracts are identified with the paints code (see Table 1). The main features obtained from these polarization curves are:

(i) Red lead, L1 and S1 (curves a and b): In both electrolytes (chloride and sulphate) cathodic inhibiting effect was not seen and the cathodic plateaus for L1, S1 and red lead solution in the chloride medium are even clearly depolarized. In the sulphate medium such effect is more subtle. This aspect has already been mentioned in literature and attributed to small quantities of metallic lead deposited on the surface of the steel, creating points at which the cathodic reduction of oxygen can proceed more easily [15]. In the anodic branch there is evidence of a slight inhibiting effect for L1 and S1, more pronounced in chloride than in sulphate solution. The important point to remark is that the curves for L1 and S1 are very similar, indicating an equivalent kinetic behavior of red lead in alkyd resins modified either with soy or linseed oil. Still in the anodic branch, red lead solution, in both electrolytes, the pigment is not playing any role. These results are coherent with the well known inhi-

bition associated with red lead. Indeed, this pigment to act as inhibitor needs to react with the fatty acids of the alkyd resins and there is no evidence that substitution of linseed oil by soy oil impaired this interaction.

(ii) Zinc phosphate, L2 and S2 (curves c and d): in the cathodic polarization curves there is better definition of the diffusion plateau whenever zinc phosphate is present. At low overpotentials there is depolarization of the cathodic reaction in comparison with the blank. At overpotentials higher than 400 mV the cathodic curves of zinc phosphate and the blank overlap. Meanwhile the curves for S2 and L2 become more polarized. Mayne [15] verified some kind of interaction between zinc phosphate and linseed oil, suggesting the formation of zinc soaps with very subtle inhibiting properties. Actually, the main responsible for corrosion inhibition was linseed oil itself. The author verified it by weight loss. Further research is desirable to explain depolarization of the cathodic reaction by zinc phosphate solution. In the case of L2 and S2 such effect may have also contribution of the agalmatolite in the painting film (see Table 1). As shown in Fig. 1(g), polarization of steel in extracts of this filler are characterized by lower anodic currents and better defined less polarized cathodic plateau. Therefore, results suggest some synergistic effect among zinc phosphate, agalmatolite and the oil. Nevertheless, this synergism does not seem to contribute positively for inhibition as both anodic and cathodic branches of the polarization curves are depolarized around the corrosion potential. Cathodic curve for S2 is less polarized in comparison to L2, but qualitatively the synergistic effect occurs equally for linseed and soy oils. The only signal of inhibition is in the anodic polarization curve for zinc phosphate solution, once there is a tendency for lower current densities in comparison with the blank along all the anodic overpotential range.

(iii) Iron oxide, L3 and S3 (curves e and f): all pigments (red lead, zinc phosphate and red iron oxide) have the same effect of the filler depolarizing the cathodic plateau. This aspect is better seen in the graph at the bottom part of Fig. 1(e). The anodic curves present a surprising inhibiting effect for L3 and S3. Moreover, in the cathodic curves, the  $O_2$  plateau is better defined for L3 and S3 as seen in the case of S2 and L2. As discussed before, this effect seems to be associated with the filler

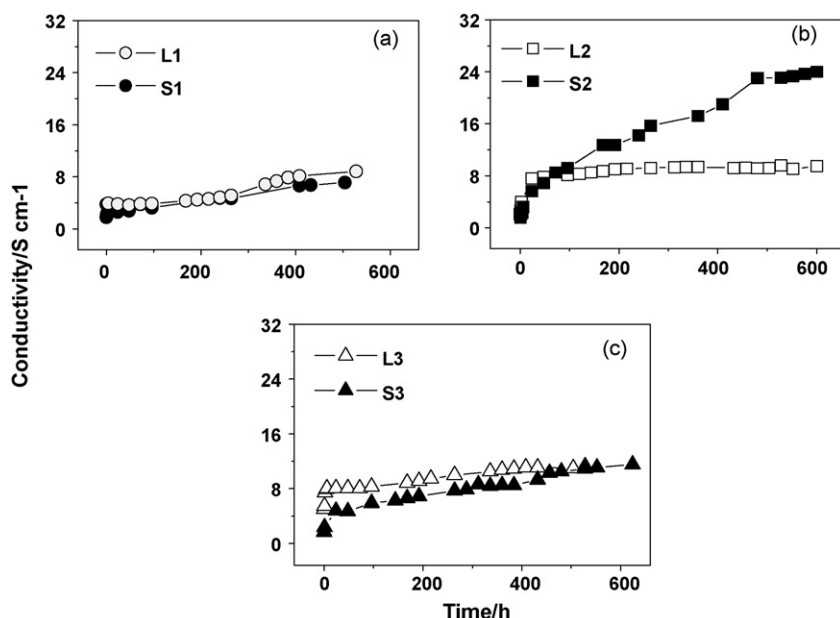
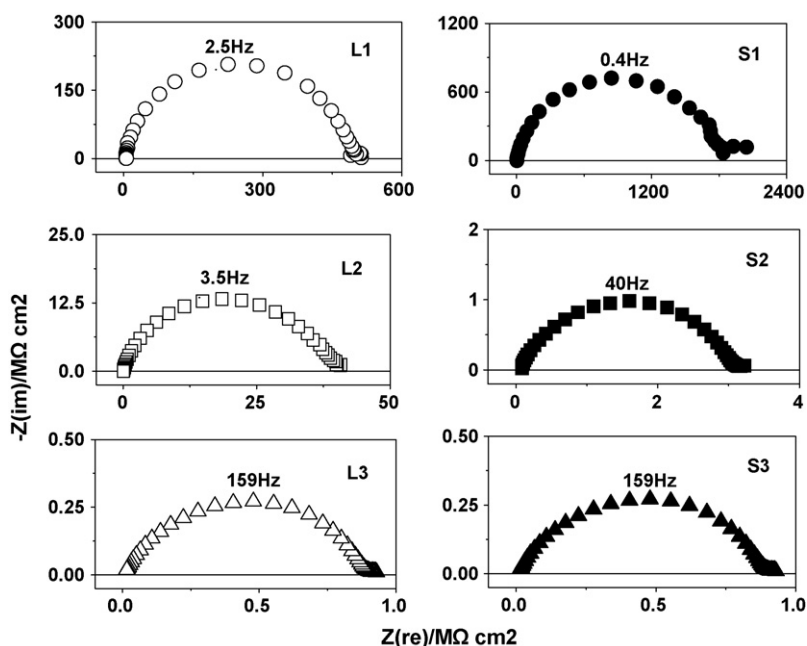


Fig. 3. Conductivity measurements during permeation of painting films by chloride ions. The dry thicknesses of the films were around 120  $\mu\text{m}$ .



**Fig. 4.** Impedance diagrams of free films of paints L1, S1, L2, S2, L3 and S3 after 800 h of immersion in 0.01 M NaCl solution. The dry thicknesses of the films were around 120  $\mu\text{m}$ .

(Fig. 1g). In this figure, not only the  $\text{O}_2$  plateau is better defined in the presence of the filler, but the anodic curve shows a slight inhibiting effect. Therefore, the present filler was not inert. The effects detected in the extracts of the painting films containing filler (see Table 1), particularly in the case of iron oxide (L3 and S3), must be attributed to the filler and not to the pigment used.

Summarizing, the effect of the oil on the action of anticorrosive pigments, Fig. 1(a–f) shows that the current values obtained for steel immersed in aqueous extracts of the dry painting films are not significantly influenced if the film is originated either from linseed oil or from soy oil modified paints.

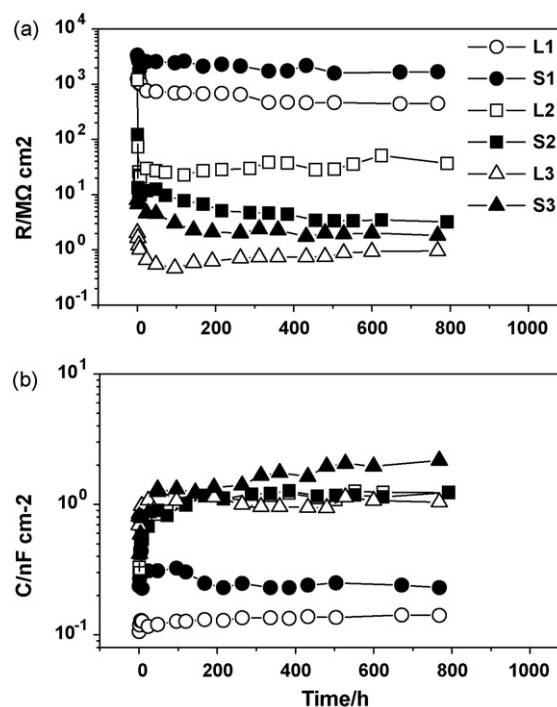
### 3.2. Influence of the oil on the barrier properties of the paintings

The permeability to water vapor tests was performed with three films of each paint. Representative results are presented in Fig. 2. According to paints formulation criteria, where all the paints have the same PVC/CPVC ratio, differences in the barrier properties suggest distinct interactions between pigment/resin. As the dry thicknesses of the films are not the same, the contribution of each pigment for the barrier properties cannot be evaluated in this experiment, and only the oil effect will be analyzed. According to Fig. 2, the permeability of paints pigmented with red lead (S1 and L1), iron oxide (S3 and L3) and filler (S4 and L4) does not change if the oil added to the resin is soy or linseed. On the other hand, in the case of zinc phosphate, for soy oil (S2) the permeability is higher than for linseed oil (L2).

The conductivity data related with chloride ions permeability are shown in Fig. 3. The free films with red lead and iron oxide did not present significant changes concerning the permeation of chloride ions. Only the films with zinc phosphate showed distinct behavior with the replacement of the oil. In this case, the conductivity increased more for the paint with soy oil. These results are coherent with the water vapor permeability presented in Fig. 2.

The composition of linseed and soy oils differs in the acids contents. Linseed oil has more unsaturated acids [3] which would enhance the drying process by oxidation and crosslinking. This

aspect alone does not explain why permeability is lower for alkyd paint modified with linseed oil only when it is pigmented with zinc phosphate. One possible explanation is that phosphates can react with acid groups to form esters which can act as plasticizers [22,23]. The work to better explain the interactions between zinc phosphate and alkyd modified paints is still in progress. However, there is no doubt that barrier properties are better when the modification is with linseed oil. This statement is confirmed with EIS.



**Fig. 5.** Resistances (a) and capacitances (b) of free films obtained from impedance diagrams.

Measurements of electrochemical impedance were carried out with the free films of the paints in 0.01 M NaCl solution until approximately 800 h of immersion. Without the influence of the substrate, the diagrams showed only one capacitive loop during all immersion times. Some representative results obtained at 800 h of immersion are shown in Fig. 4. Resistance values obtained at the low frequency limit of the diagrams are shown in Fig. 5(a). Capacitance values were calculated by  $C = (2\pi Rf)^{-1}$ , where  $f$  is the frequency of the maximum and  $R$  the diameter of the loop. Variation of capacitances with immersion times is shown in Fig. 5(b).

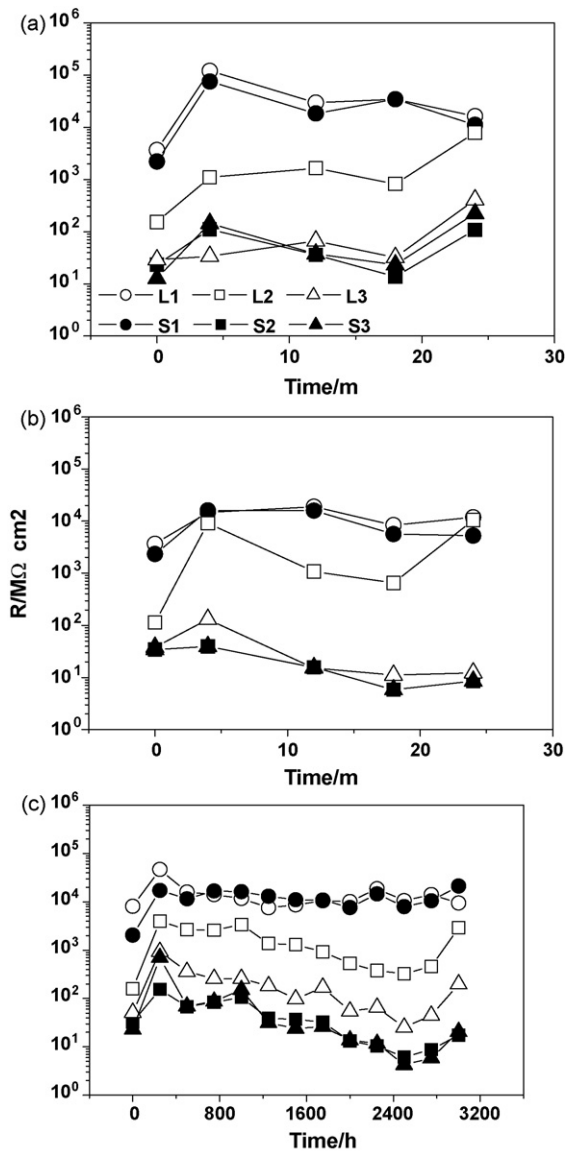
It can be seen in Fig. 5(a) that the paintings had similar behavior. A fast reduction in the resistance values occur during the first hours until reaching a minimum at about 100 h. Coherently, the capacitances reach a maximum at the same immersion time. Afterwards, both resistances and capacitances reach constant values until the end of the test.

Still in Fig. 5(a), the paintings with red lead presented the highest resistance values, while the paintings with iron oxide presented the lowest values. The paintings with zinc phosphate (L2 and S2) presented intermediate values of resistance. After about

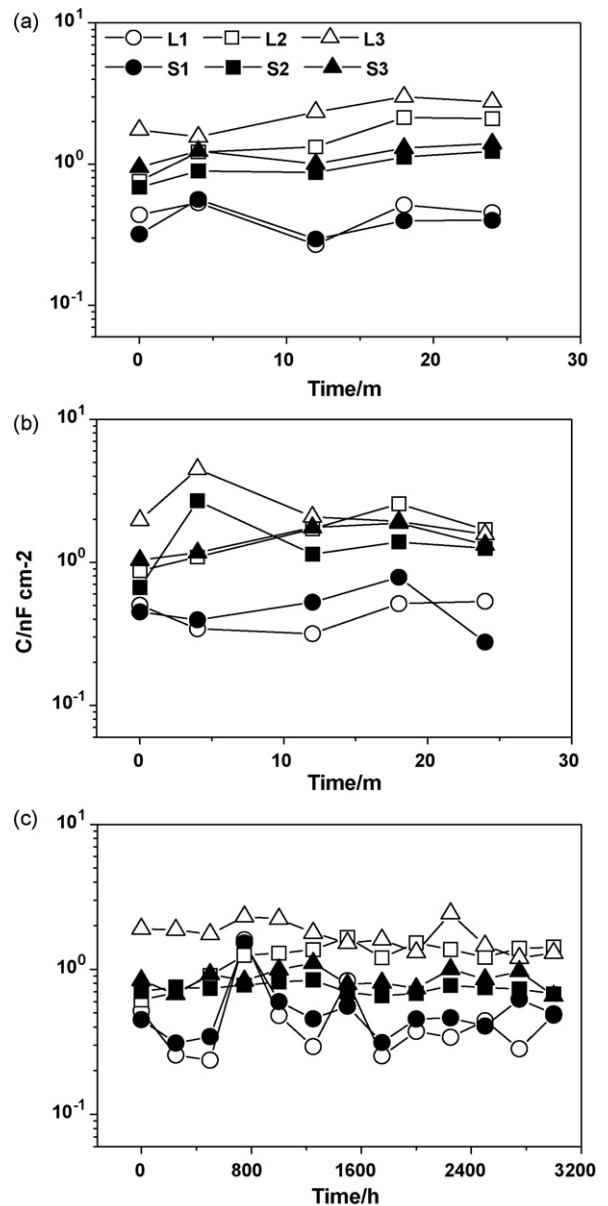
200 h, the difference between resistances measured for L2 and S2 is approximately one order of magnitude:  $10^1 \text{ M}\Omega \text{ cm}^{-2}$  for L2 and  $10^0 \text{ M}\Omega \text{ cm}^{-2}$  for S2. Thus painting L2 presented higher resistance than S2 suggesting better barrier properties. This aspect is coherent with results discussed in Figs. 2–3.

In Fig. 5(b) the capacitances of the paintings with red lead presented the lowest values. The paintings with zinc phosphate and iron oxide presented higher values and no significant difference between them was noticed.

Impedance results pointed out that the change of the oil is better noticed for paintings pigmented with zinc phosphate. Permeability to water vapor, permeability to chloride and electrochemical impedance of free films, all these results are coherent on the suggestion that barrier properties of alkyd paint pigmented with zinc phosphate are better when it is modified with linseed oil than with soy oil. Nevertheless, all these experiments were performed with free films. The interaction of the paintings with the metal substrate was not taken into account. This aspect is evaluated in corrosion tests in the following.



**Fig. 6.** Resistances obtained from impedance diagrams of painted samples during corrosion tests. (a) Industrial atmosphere, (b) marine atmosphere and (c) Prohesion Cycle.



**Fig. 7.** Capacitances obtained from impedance diagrams of painted samples during corrosion tests. (a) Industrial atmosphere, (b) marine atmosphere and (c) Prohesion Cycle.

**Table 2**  
Adherence test. ASTM D3359 B.

Initial condition				Industrial atmosphere (after 2 years of exposition)			
Paint	Adherence	Paint	Adherence	Paint	Adherence	Paint	Adherence
L1	5	S1	5	L1	5	S1	5
L2	5	S2	5	L2	5	S2	5
L3	5	S3	5	L3	5	S3	5
Marine atmosphere (after 2 years of exposition)				Prohesion Cycle (after 3000 h of exposition)			
Paint	Adherence	Paint	Adherence	Paint	Adherence	Paint	Adherence
L1	5	S1	5	L1	5	S1	5
L2	5	S2	5	L2	2	S2	5
L3	5	S3	5	L3	3	S3	5

### 3.3. Influence of the oil on the anticorrosive performance of the paints

Measurements of electrochemical impedance were also performed on painted samples during corrosion tests. The diagrams were characterized by only one capacitive loop until the end of the tests. Fig. 6(a)–(c) presents the resistance of the coatings obtained from impedance diagrams at the low frequency limit, in function of testing time, for samples in industrial atmospheres, marine atmosphere and Prohesion Cycle, respectively. Initially the paintings presented in all corrosion tests the same standard behavior, that is, an increase in the value of resistances characteristic of alkyd resins [24]. The paintings with red lead showed the highest values of resistance. Once again, changing the oil only influenced the performance of the paintings with zinc phosphate; L2 had higher resistances in comparison with S2 during all corrosion tests. Values obtained for S2 are almost the same to those obtained for the iron oxide painting. In effect, S2, L3 and S3 presented lower resistance values in all corrosion tests.

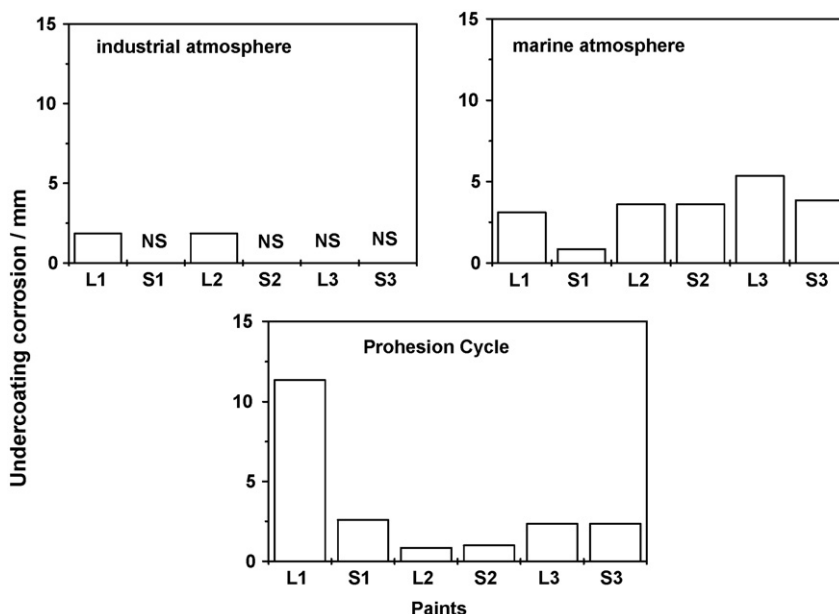
Fig. 7(a)–(c) presents the graphs of capacitance, in function of testing time, for samples in industrial atmospheres, marine atmosphere and Prohesion Cycle, respectively. The paintings L1 and S1 presented lower values of capacitance. This behavior is coherent with results discussed in Figs. 2–4. Red lead pigmented paintings not only have better barrier properties, but also, in Fig. 7, the passivation of the metal substrate may be contributing to the lowest

capacitance values. Among paintings S2, S3, L2 and L3 no significant tendency was noted in the corrosion tests which should be attributed to the oil or to the pigment. Until the end of the corrosion tests, neither blistering nor corrosion spots were detected on the samples.

After the tests in industrial exposure, marine exposure and Prohesion Cycle, the corrosion progress around the scribes was evaluated. The results are presented in Fig. 8. The industrial atmosphere was the less aggressive condition. In this case, only paintings L1 and L2 presented measurable corrosion advance around the scribe. In marine atmosphere and Prohesion Cycle, corrosion advance was more important. Nevertheless, it can be seen that substitution of linseed oil by soy oil did not impair this corrosion aspect. On the contrary, corrosion spread for paintings S are equal or lower than for paintings L in all corrosion tests.

The adherence of the paintings was evaluated after the corrosion tests according to ASTM D3359 B [13]. The results are presented in Table 2. In all corrosion tests, no adhesion loss was detected for the paintings modified with soy oil. On the other hand, paintings L2 and L3 modified with linseed oil were classified with grades 2 and 3, respectively, denoting significant adhesion loss after 3000 h of Prohesion Cycle.

It is interesting to note that painting L2, with better barrier properties than S2, presented more important corrosion spread and adhesion loss when compared with S2. Barrier properties, corrosion spread around a scribe and adhesion loss are complementary



**Fig. 8.** Undercoating corrosion in scribed painted samples after corrosion tests.

properties evaluated in corrosion tests. The differences on the barrier properties detected with free films for the paint with zinc phosphate were confirmed by impedance measurements of coated samples, predicting the occurrence of fails, such as underfilm corrosion or blistering, whether the corrosion tests lasted more. Permeability data suggested that interaction of zinc phosphate with the linseed oil modified paint is more important than with the soy oil one. One hypothesis is that such reaction decreases the polarity of the polymeric chain and adhesion forces are impaired.

It is known that polarity of the polymeric chain can contribute for adherence mechanisms [25]. In the case of alkyd paints, such contribution was identified by Reddy et al. [26] varying the oil content added to the resin.

Moreover, if the zinc phosphate is more attached to the polymeric chain in the case of linseed oil, it would not be so easily leached and corrosion spread around the scratch should not be inhibited by this pigment.

#### 4. Conclusions

The main conclusions about the modification of alkyd pigmented paints with soy oil instead of linseed oil are:

- (1) Red lead has inhibiting properties only in extracts of the painting film. On the other hand, light inhibiting properties of zinc phosphate were evidenced with extracts of the pigment.
- (2) The polarization curves suggested that agalmatolite added as filler in the paints formulation is not totally inert under the corrosion point of view.
- (3) The barrier properties, adhesion loss and corrosion spread of the paintings pigmented with red lead or iron oxide were not influenced by the oil.
- (4) There are evidences that zinc phosphate reacts with linseed oil modified paint enhancing its barrier properties. In effect, lower permeability to water vapor and chloride ions was detected, as well as impedances one order of magnitude higher in comparison with the soy oil modified paint. However, adhesion loss and corrosion spread around a scratch, which are also important properties, were better for the soy oil modified paint. Therefore, one cannot state that in long-term corrosion tests the modification with linseed oil should result in better performance.

- (5) Based on the adopted experimental approach, it is possible to state that substitution of linseed oil by soy oil does not impair the performance of medium-long alkyd paintings.

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