



Anti-Corrosion Methods and Materials

Study of the pigment type effect on the corrosion behaviour of epoxy painted steel/sea water systems

D.M. Santágata P.R. Seré S. Hornus Sackl. Elsner G. Mendivil A.R. Di Sarli

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Contributed papers

Study of the pigment type effect on the corrosion behaviour of epoxy painted steel/sea water systems

D.M. Santágata

P.R. Seré

S. Hornus Sack

C.I. Elsner

G. Mendivil and

A.R. Di Sarli

The authors

D.M. Santágata is CIC Fellow, **P.R. Seré** is CIC Fellow, **S. Hornus Sack** is Ad-Honorem Assistant, **C.I. Elsner** is CONICET Researcher, UNLP, **G. Mendivil** is CONICET Technician and **A.R. Di Sarli** is CIC Researcher, all at CIDEPINT, Centro de Investigación y Desarrollo en Tecnología de Pinturas. (CIC-CONICET), La Plata, Argentina.

Keywords

Epoxy coatings, Corrosion prevention, Pigments, Sea water, Steel

Abstract

The effect of different kind of pigments on the corrosion resistance properties of an epoxy paint has been investigated using electrochemical impedance spectroscopy and open circuit potential measurements. Painted naval steel samples were studied during the exposure to artificial sea water. The epoxy paint coatings were prepared employing red lead, zinc, red iron oxide or titanium dioxide as pigment with 0.8 of PVC/CPVC ratio. From the EIS and corrosion potential data analysis was found that the anticorrosive protection given by an anticorrosive (pigmented with red lead) and a top coat (pigmented with red iron oxide) epoxy painting scheme was both more lasting and more effective. This was attributed to the fact that both the effective adhesion provided by the chemical bonding between the steel surface and the epoxy paint groups and the constant high barrier effect afforded by this type of top coat were a perfect complement for the high corrosion inhibitive capacity of the red lead pigment.

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An organic coating, usually supplied in liquid form, is a complex mixture. When applied to a surface, the liquid is changed by a film forming process to a solid composite film. In the final coating, pigments and fillers are dispersed in an amorphous glass-like polymer matrix. Coatings may be described as solvent solutions or dispersions of some sort of binding medium, usually called the resin, in which is dispersed the solid inorganic (sometimes organic) discrete phase known as the pigment. During and after application, the solvent evaporates and the dried film consists of the non-continuous phase of solidified binder. Pigments are one of the three basic components of a coating. It can be said about them that they are inorganic compounds such as oxides and silicates, although many of the brighter colors are complex organic materials having little or no solubility in the coating solvent (Hare, 1994; 1996).

The anticorrosive pigments used in paints applied on steel are capable, under certain conditions, of delaying the action of localized corrosion cells due to the formation of insoluble iron oxides directly on them; this process only takes place through the action of the pigment dissolved fraction. On the other hand, the binder must also fulfil certain important requirements as, for example, the capacity of swelling in contact with an aqueous electrolyte, allowing in consequence the diffusion of the dissolved pigment up to the metallic surface. This condition occurs when the pigment is close to the CPVC, thus justifying the fact that the anticorrosive primers normally have a high PVC. In some cases, the anticorrosive pigment cannot develop its protective action by means of the above mechanism, but it is observed that the coating adequately protects steel against corrosion; then, this is due to the barrier effect. This type of protection is particularly important in presence of Cl^- and SO_4^{2-} ions, since the passivating action corresponding to anticorrosive pigments, corrosion inhibitors or alkaline media is strongly damaged or completely suppressed by formation of complex soluble iron ions, which can diffuse and be transformed into insoluble oxides far from

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their original position, preventing the passive layer repairment.

The possibilities of improving the barrier effect of organic coatings are aimed mainly at achieving a lower permeability to water, oxygen and ionic species, and at obtaining a good and lasting adhesion to the metallic support, especially in the case of long exposures to aqueous electrolytes or high environmental humidity. Such improvements in this kind of protection can be obtained by using electrochemically-inert barrier pigments which physically reinforce the barrier properties of the paint film, i.e. they reduce the permeability of the paint film to the agents supporting corrosion (Di Sarli and Giudice, 1988).

The objective of the present work is to gain deeper insight on the corrosion protection of naval steel in sea water by a pigmented epoxy paint. Coating performance was studied from the changes of impedance spectrum and open circuit potential with exposure time in artificial sea water. The results illustrate the influence of the pigment (red lead, zinc, red iron oxide and titanium dioxide) on the protective properties of an epoxy-polyamide paint employed on steel.

Experimental details

Each sample substrate was a $15 \times 8 \times 0.2$ cm SAE 1020 test panel, whose surface was sandblasted up to Sa 2–3 degree (SIS Standard 05 59 00/67). The average value of the surface roughness profile was $2.5 \pm 0.25 \mu\text{m}$. After this procedure, steel panels were cleaned with toluene, to ensure surface uniformity, and later coated with a commercially available epoxy-polyamide paint pigmented with anticorrosive (active zinc, alkaline red lead) or inert (titanium dioxide or red iron oxide) particles. Comparatively, an unpigmented epoxy-polyamide paint was also employed. The curing period in a dessicator kept at room temperature was two weeks. The dried film thickness was measured with an electromagnetic instrument, Elcometer 300, using a sandblasted bare sheet and patterns of known thickness as reference. The average coating thickness values are summarized in Table I.

On each plate two cylindrical tubes of transparent acrylic were fixed by using an epoxy adhesive to get good adhesion to the

coated substrate. The geometrical area for impedance measurements at the corrosion potential was 15.9cm^2 . The electrochemical tests were carried out in a quiescent under ambient aeration artificial sea water, prepared in according with the ASTM Standard D-1141/90. The electrolyte level and the pH (8.2) were kept constant. A three electrodes electrochemical cell was formed with a Pt-Rh mesh counterelectrode oriented parallel to the working electrode (painted metal surface) while the potentials were measured and referred to in the text against a saturated calomel electrode (SCE). In order to minimize external interference to the studied system, all the impedance measurements were performed in a Faraday cage using a Solartron 1255 frequency response analyzer, Solartron 1286 potentiostat and a PC. Impedance data were collected at frequencies range 100kHz to 1mHz. The experimental impedance spectra were interpreted on the basis of equivalent electrical circuits using a suitable fitting procedure developed by Solartron.

The water diffusion, solubility and permeability coefficients for each one of the applied organic coating were determined just after immersion in the electrolyte. For this, the electrochemical impedance and phase angle evolution at a fixed frequency (20kHz) were monitored as a function of the immersion time, from the exposure to the aqueous electrolyte up to obtaining constant values (Schwiderke and Di Sarli, 1986). All the tests were accomplished at room temperature ($20 \pm 2^\circ\text{C}$).

Experimental results

Water permeation

The water permeation resistance is one of the most important features of protective coatings. Therefore, some comparative data related with this transport property are shown in Table I. The main finding here is the great difference among the permeation of the different paint films, which is substantially greater for samples C, F and H than for samples A, B, D, G, I and J.

In the case of the thinner primer paints (A, B, C, D and E), only the red lead pigment (sample B) caused a further reduction of water permeability with respect to the unpigmented film (sample A). On the contrary, red iron oxide (sample C) or titanium dioxide

Table I Type, thickness and water diffusion, solubility and permeability coefficients of the tested samples and painting systems

Sample	Thickness (μm)	Diffusion $\text{D} \cdot 10^9 \text{cm}^2 \text{s}^{-1}$	Solubility $\text{S} \cdot 10^2$	Permeability $\text{P} \cdot 10^{10} \text{cm}^2 \text{s}^{-1}$
One coat of epoxy paint pigmented with				
Unpigmented	A	30 ± 3	0.39	7.67
Red lead	B	29 ± 3	0.12	16.60
RIO	C	30 ± 3	9.12	8.04
TiO ₂	D	28 ± 3	0.17	39.31
Zinc	E	30 ± 3	–	–
Zinc	F	95 ± 3	4.04	5.96
RIO	G	67 ± 3	1.53	3.49
Painting scheme pigmented with				
AC (red lead) + TC (RIO)	H	93 ± 3	2.35	4.32
TC (RIO + TiO ₂)	I	80 ± 3	0.63	7.99
AC (red lead) + TC (TiO ₂)	J	81 ± 3	0.12	17.70

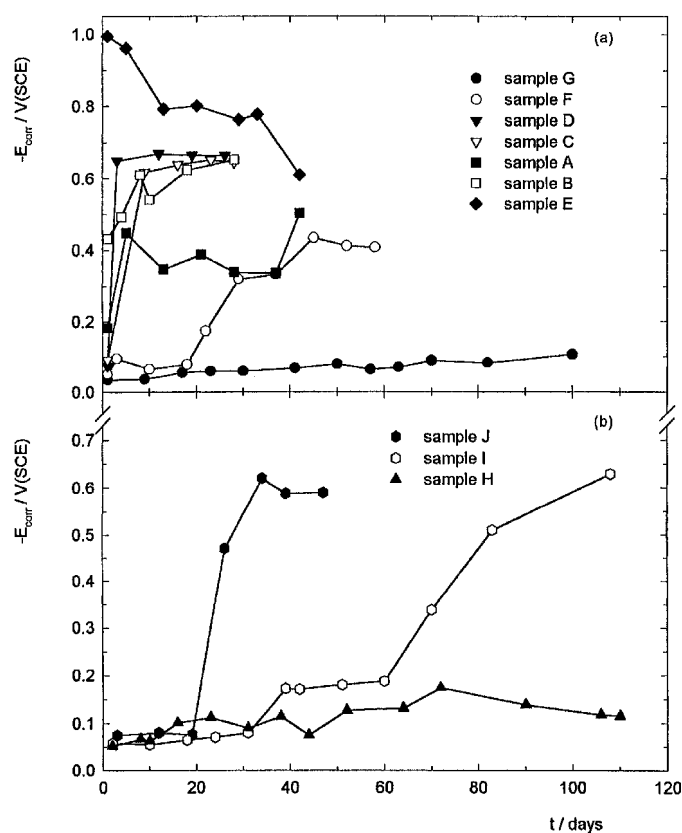
Notes: AC: anticorrosive; TC: topcoat; RIO: red iron oxide

(sample D) pigmented epoxy paints showed an enhanced water permeation, while the corresponding to those using zinc powder (samples E) could not be measured. This occurred because the water permeation rate was high enough as the paint film became saturated just after a short exposure time, and it causes a too significant experimental error (Schwiderke and Di Sarli, 1988). Such a performance may be attributed to the creation of additional ways for water permeation at the pigment/binder interface in the coating. On the other hand, when the thickness of two pigmented applied paint films showing the highest water permeability was increased, data presented in Table I for samples F and G prove that a substantial decrease of their diffusion coefficient was found. The same relative trend was observed when the water permeability of the samples H, I and J was evaluated.

In principle, therefore, the water permeability measurement provides a sensitive enough evaluation of the coating barrier properties.

Electrochemical measurements

Figure 1a shows the variation of the corrosion potential (E_{corr}) as a function of the exposure time. During the first week immersion, for steel coated with an unpigmented (sample A) or pigmented with either red lead (sample B),

Figure 1 Corrosion potential (E_{corr}) vs immersion time (t) of all the painted steel/artificial sea water systems

red iron oxide (sample C) or titanium dioxide (sample D) epoxy paint, a continuous displacement of E_{corr} towards more negative (i.e. electrochemically active) values was observed.

After this period, E_{corr} changed slightly up to ending the test. On the contrary, the E_{corr} corresponding to sample E started at -1V but then it also moved towards more positive values up to reaching -0.6V , i.e. a potential value very similar to the attained by the other four samples.

Such behaviour could however be improved when a thicker film pigmented with active (Zn, sample F) or inert (red iron oxide, sample G) particles was used, Figure 1a. This last shows that even better results can be found if instead of one paint layer, an organic coating system properly chosen is employed. Thus, under the present exposure conditions three different painting systems with strongly different electrochemical responses were tested. Figure 1b, illustrating the E_{corr} evolution for systems H, I and J, proves that the potential of system H stayed almost stabilized while that corresponding to systems I and J showed a clear although kinetically different trend to move towards more negative values throughout the test.

Data concerning the ionic (R_f) and charge transfer (R_{ct}) resistances for all the tested samples exposed to artificial sea water are summarized in Figures 2a, 2c and 2e, respectively.

As a consequence of the water and ionic species uptake and the diffusion process through the epoxy paint coating to the steel substrate, the R_f values for the thinner (samples A-E, Figure 2a) and thicker (system J, Figure 2c) films become substantially smaller. This action was however either less significant in the case of samples F (Figure 2a) and I (Figure 2c) or not observed as for samples G (Figure 2a) and H (Figure 2c).

The magnitude of R_{ct} (Figure 2e) is inversely related to the rate of the corrosion processes, therefore, it can be used for monitoring the inhibitive efficiency of pigments added to the paint formulation. As is shown in Figure 2e, no data of R_{ct} were detectable for samples F and G and systems H, I and J, which is clearly indicative that an efficient protection was afforded by these pigmented paint films. On the other hand, and despite the poor barrier properties shown by samples A-E, the estimation of R_{ct} at a given exposure time was not always possible. Nevertheless, when it can be done their values were further oscillating and demonstrative of a relatively moderate corrosion protection, except for the unpigmented

sample A which exhibited good enough protective properties.

Referring to the dielectric capacitance (C_f) of the paint films, the results are shown in Figure 2b for the thinner films (samples B, C, D, E). Their values indicate that the expected behaviour was followed, i.e., the water permeation causing an initial fast increase of several orders of magnitude up to reaching saturation; then, C_f kept relatively stable or slightly oscillating. On the other hand, either for the unpigmented or the thicker paint films, samples A and F-G respectively, the capacitance values exhibited small changes since they ranged between 10^{-10} – 10^{-9}Fcm^{-2} throughout the exposure time. This performance was similar to that observed for the painting systems H, I, J, Figure 2d, whose dielectric capacitance remained oscillating within an interval of one or two orders of magnitude.

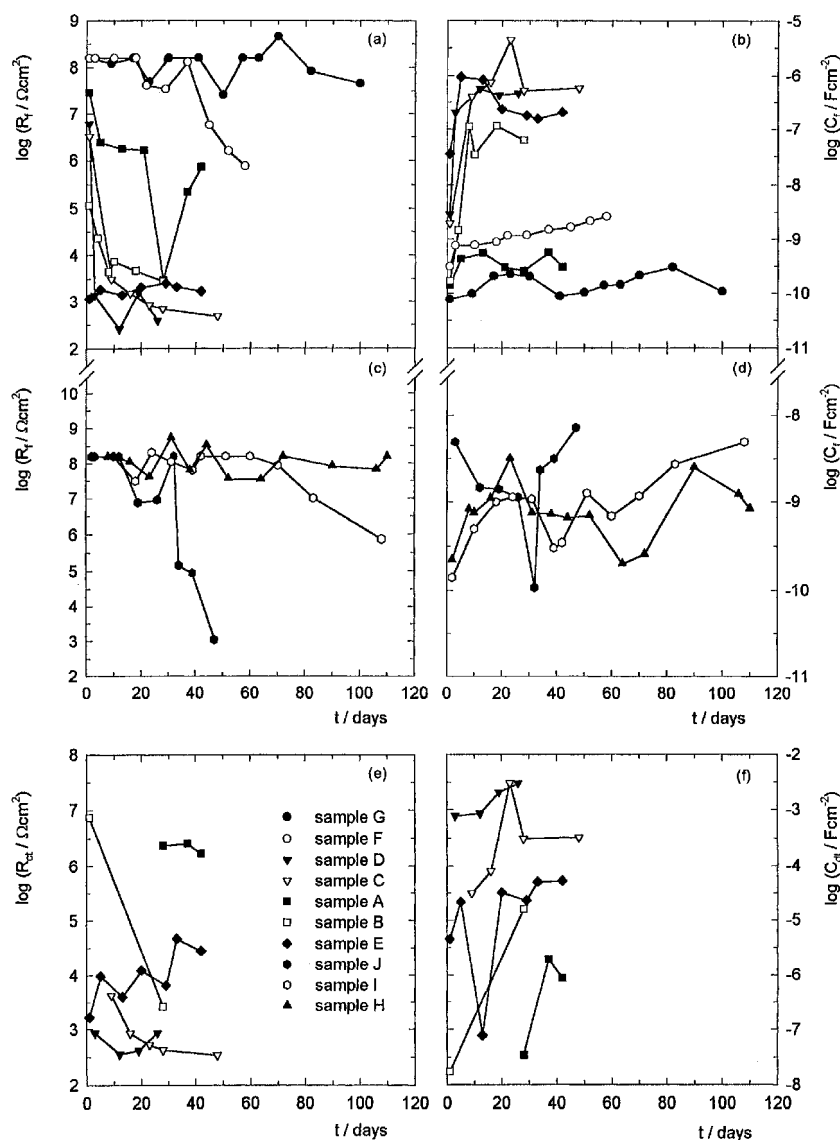
With regard to the electrochemical double layer (C_{dl}), Figure 2f, the values either increased continuously to attain the “normal” (or greater) value for an uncoated steel surface in sea water or else they kept oscillating in correspondence with the R_{ct} displacements.

Discussion

Selection of pigments for corrosion protection with organic coatings creates serious problems for the paint formulator. When more details about the coating material itself and the pigment as well as the interactions between them are available, the chance of getting the best formulation for each practical situation is higher.

From the point of view of the coatings, the barrier properties and permeability seem to be of great interest, since they determine the water transport and/or dissolution and supply of pigments to the metal substrate. Water permeability and ionic diffusion as well as corrosion potential and impedance measurements provide insight related with those organic coating properties. In such sense, the heterogeneous nature of the electrical properties of polymeric films are physically represented as a heterogeneous group of electrolytically conducting paths. Thus, in localized areas, the coating does not behave like a dielectric, but shows an electrolytical conductivity due to electrolyte dissolution

Figure 2 (a-d) paint film and (e-f) steel substrate parameters vs Immersion time (t) of all the painted steel/artificial sea water systems



inside the structural defects of the polymer or as a consequence of its penetration through the film pores. In other localized regions, the coating has the properties of a relatively inert dielectric material. At the metal/metal oxide/organic coating interface, several complex processes of difficult interpretation may take place since after the corrodent species (H_2O , O_2 and ionic species) permeation different electrochemical reactions are possible (Zsauer and Brandt, 1984; Frydrych *et al.*, 1987).

When painted steel plates are submerged in an aqueous solution, the water absorbed by the organic coating can dissolve some pigments. It is known that a high barrier effect or a low diffusion of corrosion inhibiting species can keep or accelerate the active state even if there is a significant pigment content in the paint film. Thus, it is difficult to determine

whether the metallic substrate is passivated or not, particularly in the presence of anions like Cl^- , which are capable of breaking the passivating layer (IVP, 1975-1977; Piens, 1979). This can be only balanced either by increasing the minimum pigment volume concentration required to obtain such a layer (IVP, 1975-1977; Szklarska-Smialowska, 1978; Vetter, 1967) and/or the paint film barrier properties using a properly chosen painting system.

The poorer protective properties of the thin samples (B-E) could be explained assuming that, independently of the organic coating thickness, the water permeability coefficient was less for samples type A than for most others, the exception being the samples type B. This result is explained assuming that both the barrier but chemically inert pigments (red

iron oxide and titanium dioxide) and the sacrificial one (Zn) did not bond well with the epoxy resin causing, therefore, a water permeation increase due to the weakly associated pigment/resin interface. In such circumstances, a faster route for the water diffusion through and dissolution into the paint film is provided. As we will see later, such a behaviour was intimately associated with the protected properties given to the steel substrate by the tested paint films.

The change of the corrosion potential (E_{corr}) with the exposure time in sea water has been used successfully as a simple tool for the study of the corrosion protection by organic coatings (Armas *et al.*, 1992; Mayne, 1954; Meszáros and Lindquist, 1982; Morcillo *et al.*, 1990). After immersion of the painted steel plates in the electrolyte and due to both the relatively easy water permeation but mainly the low coating thickness, the E_{corr} of samples A-D changed quickly (Figure 1a) from an initial value of about $-0.1/-0.4\text{V}$ (vs SCE) to more negative ones, while sample E does it from -1.0V to more positive values, up to reaching a common level that lay close to the steel corrosion potential in sea water (Pourbaix, 1963). The stationary value of $E_{\text{corr}} \approx -0.60(0.06\text{V})$ attained after ca. 3-20 and 40 days immersion in the case of samples A-D and E, respectively, corresponds to a situation where the anticorrosive and/or barrier effect (samples A-D) and the zinc galvanic action (sample E) had ceased to be effective. Under these circumstances, attack of the substrate alone (red rust) or of the substrate and the zinc particles (white rust) could be visually observed at several localized spots on samples B-D and E, respectively. Exceptions to this behaviour were those obtained for the unpigmented sample A as well as for the thicker and pigmented samples F and G. In the case of sample A, after a rapid initial increase, the E_{corr} remained almost constant at a value of $-0.4 \pm 0.02\text{V}$, probably because the low film thickness could be in certain degree balanced by the greater film homogeneity, which consequently afforded higher resistance to the permeation of corrodent species. Instead, the higher thickness of samples F and G caused a different response, improving satisfactorily the barrier property of samples (type G) pigmented with chemically inert particles such that their E_{corr} stayed constant and near to $-0.04 \pm 0.01\text{V}$; however, this fact did not allow that in sample

F the Zn particles either were activated or their availability was low at the paint/steel interface since the corrosion potential values recorded were much more positive than those measured for the thinner sample E.

On the other hand, concerning to the dependence of E_{corr} values on the exposure time for the three painting systems (H, I, J) tested, data in Figure 1b are in agreement with the water permeation results given in Table I. Therefore, such data were used to rank, at least from an electrochemical point of view, those painting systems in terms of their protective properties. Thus, a much more efficient protection was obtained when the anticorrosive action and/or the barrier effect were provided by the pigments used in samples H than in samples I and J.

A fairly good description of the experimental impedance diagrams was obtained in terms of transfer function analysis using non-linear fit routines. Because of the complex nature of the processes taking place at the painted steel interface it was necessary to derive the appropriate model accounting for the time dependence of measure impedance for the different samples.

EIS data in Figures 2a-f show clearly that the one coat pigmented paint (samples B-D) as well as the painting systems H and I suffered degradation and occurrence of corrosion at the steel/epoxy paint interface during exposure to artificial sea water for some weeks, while samples A, F, G and the system H remained more or less unchanged.

As the coating degraded, the ionic resistance R_f (Figures 2a and 2c) decreased as a result of the formation of conductive paths due to the penetration of corrodents (Cl^- , Na^+ , O_2 , etc.) and water uptake. On the contrary, the cyclic increase of R_f along the exposure time can be related to the formation and accumulation of insoluble corrosion products within the paint film defects, which decreases the effective defects area reinforcing the barrier effect. These results are in agreement with the gradual changes of the corrosion potential towards more negative or positive values, respectively, as depicted in Figures 1a-b. After prolonged exposure time such a trend may be influenced by perforation of other less resistant localized areas in the coating layer, causing the deterioration of the protective system which prevailed over the anticorrosive and/or barrier-type effects

already enhanced by the substrate and/or the pigments corrosion products.

The initial C_f increase (Figures 2b and 2d) was attributed to the water uptake since the dielectric constant of water is more than 20 times larger than that of the coating (Mansfield *et al.*, 1982). The smaller increase showed by samples A, F, G and systems H and I was attributed to the fact that the unpigmented and, therefore, structurally more homogeneous epoxy paint film as well as the pigmented and thicker ones delayed water penetration. The also cyclic decrease of C_f with time observed in some samples suggested that the sealing effect of the corrosion products within the coating matrix causes a water displacement out of the organic film.

The relatively small changes of R_f and C_f with the exposure time in the electrolyte for samples A, F, G, and systems H and J were ascribed to the fact that the paint films retained their good barrier properties leading to a highly limited, if any, increased in the corrosion rate of the substrate. Thus, the best dielectric performance observed for these samples, i.e., low C_f values (Figures 2b and 2d), indicated that they behaved according to a lower water uptake, which in turn is necessary to complete the electrical circuit in order that the faradaic processes at the steel/paint interface take place. Therefore, the beginning of these last was longer delayed because either no electrochemical double layer was formed or else it was so small that the resistive and capacitive parameters associated with it were masked by those related with the impedance of the organic coating film (see Figures 2e-f).

On the other hand, when the coating paint non properly protected the steel surface as was the case of samples C-E, the initial value of the charge transfer resistance R_{ct} (Figure 2e) is lower than that of samples A-B, independently of the film thickness. However, the time dependence of R_{ct} is affected by the type of film employed because while this parameter stayed high and almost constant for sample A, it dropped pronouncedly for sample B, to reach the continuously low resistance values provided by the paint films type C-E. This suggested that due to an insufficient coating thickness and to a high Cl^- concentration in the electrolyte solution either the anticorrosive pigment concentration in the steel/paint interface and/or the

sealer pigment barrier effect was unable to protect the substrate. That is to say, after a short exposure time the corrodent could arrive to such interface provoking the recorded consequences on the potential and impedance values as well as on the sample visual appearance. As was mentioned above the, in general, oscillating behaviour of the electrochemical double layer C_{dl} (Figure 2f) with time observed for samples A-E was also associated with the accumulation of corrosion products at the bottom and within the paint film defects, which diminished the electrochemically active area at the steel/epoxy paint interface. On the contrary, the perforation of new coating defects caused an increase of such areas and, consequently, of the C_{dl} values which, in some cases, reached values as high as several mF (Figure 2f), which could not be related to C_{dl} at the metal/coating interface. For samples C, D and E open blisters were observed. Experimental values of C_{dl} and R_{ct} were related primarily to these blisters. This process went on up to the total delamination of the paint film or else the metal/coating bonds were able to resist the wet adhesion.

Conclusions

- (1) Dynamic system analysis with small perturbation allows the determination of system specific parameters which characterize the deterioration of painted steel with immersion time in sea water. Also the water uptake in the early stage of exposure from the rate of changes in the paint dielectric capacitance was determined. Corrosion of paint-coated steel involves defect formation, penetration of corrodents, loss of adhesion, and attack of the substrate.
- (2) Electrochemical results indicate that the type of pigment as well as the thickness of one paint or painting scheme used are variables having a paramount importance on the effectiveness of each protective period. These are formed by an initial stage of barrier protection followed by a barrier + anticorrosive one, when an inhibitive pigment has been added to the paint formulation. From the analysis of the whole set of data obtained from the non-destructive and reproducible

electrochemical methods above described it was found that:

- in order to protect the steel against the attack of a strongly aggressive marine medium, the protective properties provided by the epoxy painting scheme built up by an anticorrosive (pigmented with red lead) + a top coat (pigmented with red iron oxide) were much more lasting and effective than the other systems and the paints used alone; and
- such performance was attributed to the fact that both the effective adhesion provided by the chemical bonding between the steel surface and the epoxy groups of the paint, and the constant high barrier effect afforded by this type of top coat were a perfect complement for the high corrosion inhibitive capacity of the red lead pigment.

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