

EIS study of the corrosion behavior of an organic coating applied on Algerian oil tanker

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Abstract. Organic coatings are widely employed in the corrosion protection of most metal surfaces, particularly steel. They provide a barrier against corrosive species present in the environment, due to their high resistance to oxygen, water and ions transport. This study focuses on the evaluation of corrosion protection performance of epoxy paint on the carbon steel surface in chloride environment (3% NaCl) by Electrochemical Impedance Spectroscopy (EIS). The electrochemical behavior of painted surface was estimated by EIS parameters that contained paint film resistance, paint film capacitance and double layer capacitance. On the basis of calculation using EIS spectrums it was observed that pore resistance (R_{pore}) decreased with the appearance of doubled layer capacitance (C_{dl}) due to the electrolyte penetration through the film. This was further confirmed by the decrease of diffusion resistance (R_{d}) which was also the indicator of the deterioration of paint film protectiveness. Microscopic analyses have shown that oxidation dominates the corroded surfaces.

Keywords: corrosion mechanisms / carbon steel / electrochemical impedance spectroscopy / epoxy paints

1 Introduction

Electrochemical impedance spectroscopy (EIS) is widely used with good results for the electrochemical characterization of protecting organic coating on metals [1–4]. The substrate surface can be pretreated in many different ways before painting: chemical conversion treatment for coil coating (chromatation, phosphatation, new “chromium free” pretreatment, etc.) or mechanical surface preparations such as sand blasting or brushing. The protecting coating cycle often consists of some layers of paint which include adhesion promoters, primer surface tolerant, intermediate layers and top coatings. These different organic layers have different chemical and physical properties due to the different chemical composition of the matrix and the presence of anticorrosive pigments. All these parameters (metal substrates, surface pretreatments, painting cycles) can influence the electrochemical behavior measured by EIS, which is also a function of the environment and the general condition of measuring (temperature, oxygen concentration, etc.)

The final purpose of the EIS characterization of protecting organic coatings is to obtain information about the system properties such as presence of defects, reactivity of the interface, adhesion, barrier properties to water, etc.

Knowledge of these parameters is very useful for predicting anti-corrosive behavior in service of paints. It is evident that to obtain this result it is necessary to distinguish the impedance values as a function of the frequency in the single contribution due to the components of the system, for example distinguishing the part of the impedance due to the coating from that part due to the substrate. In other words, proposing some equivalent electrical circuits which can model the impedance results. It is possible to choose a limited number of electrical elements (capacitance, resistance...etc.) with an impedance equivalent to the impedance of the system studied for every frequency. From the complex situation previously described about organic coatings and metal, it clearly emerges that such models (or equivalent electrical circuits) will be different according to the different materials, and they will have to be chosen carefully. In order to use the correct equivalent circuit, all the information obtained about the system, including using non electrochemical methods can be very useful, also because it is important to remember that mathematically there are a large number of different equivalent electrical circuits with the same impedance [5]. The second step, which is also very important, is the effort to pass from the values of the equivalent electrical circuit (resistance, capacitance, etc.) to physical properties characterizing the system, which are the final aim of the EIS characterization of organic coatings. However, not all the elements of the equivalent electrical circuit have the same importance for this aim, and particular care must be used to choose the

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parameter to discuss. In order to obtain this kind of information, it is necessary to understand how the physical properties, for example adhesion or water barrier properties can influence the electrical parameters measured by EIS. The aim of this work was to determine the protective performance of epoxy coating against 3% NaCl solution before and after damage.

2 Experimental part

2.1 Sample material and preparation

The metallic substrate was X52 carbon steel (according to NF10027 standard) in conformity with the norm API (American Petroleum Industry), used in the tanker, the chemical composition of the tested carbon steel is given in Table 1. Before coating application, the metallic substrate was sandblasted to Sa 2.5 (Swedish Standard SIS 05 59 00/67) (roughness Ra 6.2 μm) or polished with emery paper up to G 400. Commercial epoxy was immediately applied onto steel panels using a brush or a roller (Fig. 1). Once cured, the samples were stocked in a desiccator until the moment of testing. The coating thickness was measured using an Elcometer gauge and was found around 80 μm for all panels, the composition of the coating is proprietary information. Coated panels were cut out (100 \times 60 \times 4 mm) and an electrical wire was added in order to allow electrochemical measurements.

With the aim to achieve the electrochemical measures in the best conditions it has been suited that the areas of about 15 cm^2 exposed to the electrolytic solution were sufficient. It seemed necessary to use a surface of paint relatively big in contact with electrolytic solution in order to compensate the insulating role of the sample as the thickness of the film grows. Mansfeld reports in a technical document [6] a study of Kendig and Scully suggesting the use of samples covered with a ratio area/thickness of the coating of at least 10^4 to assure satisfactory electrochemical measurement. Samples were exposed under open circuit potential conditions in sea water, for electrochemical impedance.

2.2 Electrochemical impedance spectroscopy measurements

The Electrochemical Impedance Spectroscopy (EIS) measurement is carried out in 3% NaCl solution, using a potentiostat/galvanostat EG&G A273. A frequency response analyser Solartron FRA 1260 connected to an electrochemical interface Solartron SI 1287 was used to perform EIS measurements. A filter (Kemo VBF 8) was also employed to improve the signal to noise ratio. The frequency domain covered was 100 kHz to 10 MHz with the frequency values spaced logarithmically (five per decade). The width of the sinusoidal voltage signal applied to the system was 10 mV. All the measurements were performed at the open circuit potential and at different immersion times. The electrolyte was confined in a glass tube which was fixed to the painted surface by an O-shaped ring. The total tested area was 15 cm^2 . Platinum gauze of large area

Table 1. Chemical composition of carbon steel (X52) (% in weight).

C	Mn	S	P
0.28	1.25	0.03	0.03

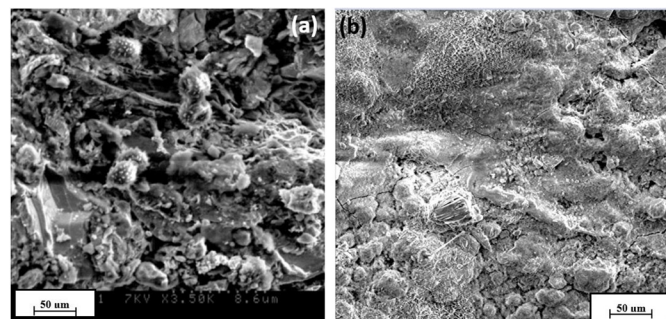


Fig. 1. (a) Cross section SEM micrograph on sandblasted steel (Sa 2.5); (b) Cross-section of the studied epoxy paint.

was used as a counter electrode. All the potentials in the current article are referred to Ag–Ag–Cl. During the intervals between EIS measurements, the painted specimen was kept in the electrolyte cell without reference electrode. The cell design for EIS measurement was described in detail in a previous work [7].

3 Results and discussion

3.1 Influence of the surface state on the electrochemical properties of the A283C carbon steel in 3% NaCl solution

3.1.1 Sandblasted state (Sa 2.5)

Electrochemical impedance diagrams, plotted for the sandblasted carbon steel after different periods of immersion to the corrosion potential, in the Nyquist plane, are represented in Figure 2, Bode diagrams shows the changes in module and phase of the impedance as a function of frequency. They are characterized by a single capacitive loop. The values of various parameters are summarized in Table 2. As the circular arcs are not centered on the real axis Z, for the calculation of the R_{ct} was extrapolated “low frequency” part at zero frequency.

Analysis of the various electrochemical parameters shown in Table 2, we note a significant increase in the charge transfer resistance R_{ct} during the first hours of immersion. This means that the process governing the kinetics is under joint control, charge transfer and diffusion and it is difficult to separate the two phenomena. However the double layer capacitance C_{dl} seems very high to be representative of the double layer capacitance (of the order of $\text{mF}\cdot\text{cm}^{-2}$) which is usually between 10 and 50 $\mu\text{F}/\text{cm}^2$. This situation was already met during our study of blasted steel in the same medium. This phenomenon Observed by Duprat [8] is due to the faradic current of corrosion products, attributed to the porous nature of this film,

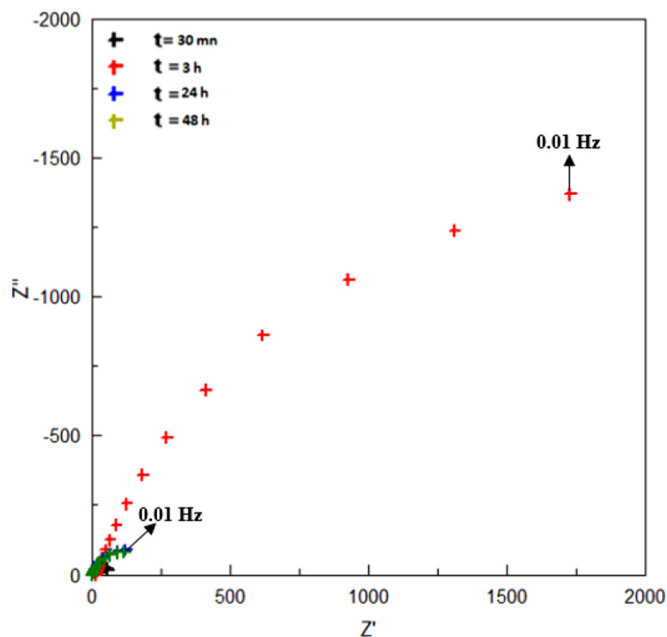


Fig. 2. Evolution of Nyquist and Bode diagrams as a function of immersion time in 3% NaCl solution for sandblasted steel. (Z' , Z'' $\Omega.cm^2$).

Table 2. Parameters values extracted from the fitting procedure.

Time (h)	E_{corr} (mV)	R_{ct} ($K\Omega.cm^2$)	C_{dl} ($mF.cm^{-2}$)
30 min	-685	0.069	53.05
3	-803	4.03	5.12
24	-798	0.25	64.03
48	-795	0.23	62.25

formed abandonment and present at the metal interface. Through these results, we deduced that the sandblasted steel has a mediocre behavior in this environment.

3.1.2 Polished state

The diagrams of Nyquist determined to different time of immersion, in the 3% NaCl solution normally aired and non-agitated are represented in the Figure 3, the values of the different parameters are gathered in the Table 3. In our study solution, semicircular responses are obtained. However each of them deviate from the ideal interface, because of the presence of heterogenists, roughness... etc.

Analysis of impedance diagrams, there is a significant increase in the R_{ct} during the first hours of immersion, translated the film formation of corrosion products that grows with the immersion time [9] as rust layers consist mainly of hydroxides such as $Fe(OH)_3$ or an iron oxide Fe_2O_3 . The film formed is stable and adherent but porous, whether we will go further in the immersion time there have been a decrease in the R_{ct} these phenomena are due to the evolution of corrosion products [10–12].

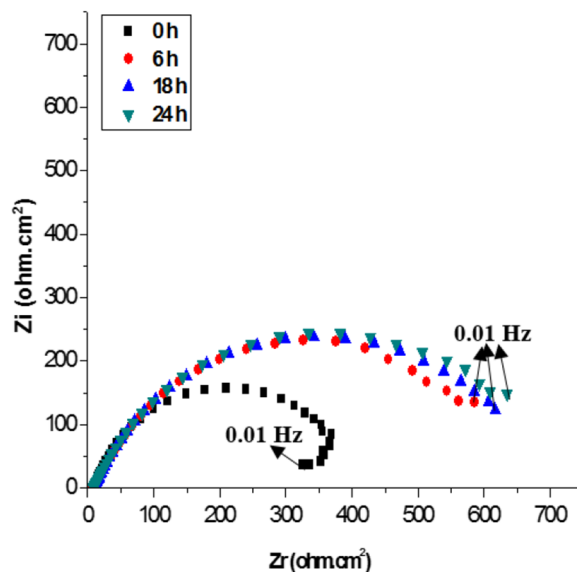


Fig. 3. Evolution of Nyquist diagrams as a function of immersion time in 3% NaCl solution for the polished steel.

Table 3. Parameters values extracted from the fitting procedure.

Time (h)	E_{corr} (mV)	R_e ($\Omega.cm^2$)	R_{ct} ($\Omega.cm^2$)	C_{dl} ($mF.cm^{-2}$)
0	-620	9.063	404.8	1.252
6	-666	10.28	640.6	1.242
18	-680	9.459	686.0	1.160
24	-683	9.204	709.2	1.418

3.1.3 Equivalent circuit for the EIS simulation

Interfacial impedance is modeled in terms of equivalent circuit [13]. The electrochemical kinetics control is under charge transfer control, the equivalent electrical circuit is shown in Figure 4, the contribution of the electrolyte resistance is simulated by the resistance R_e , the faradic impedance R_{ct} and the capacitance of the double layer C_{dl} are parallel. They are characteristic of the substrate.

3.2 Influence of environment on the electrochemical behavior of a primer damaged in situ

3.2.1 Open circuit potential (OCP) measurements

This method is to follow over time the evolution of the open circuit potential of our damaged primer in 3% NaCl solution and in sea water. The potential is noted from the immersion of the sample surface in our environment, we follow the evolution of the potential all minutes until almost total stability of the potential (Fig. 5). The analysis of the curves in Figure 6, there is some degradation of our primer in different media (3% NaCl and in sea water), this degradation causes a change in our surface state due to the formation of corrosion products that cover the surface, these more or less adherent cause stability of corrosion potential. We note that for our primer in sea water

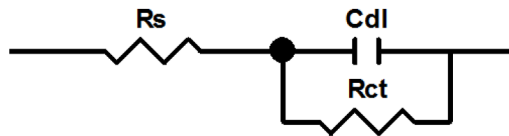


Fig. 4. Equivalent electric circuit of the electrochemical interface.

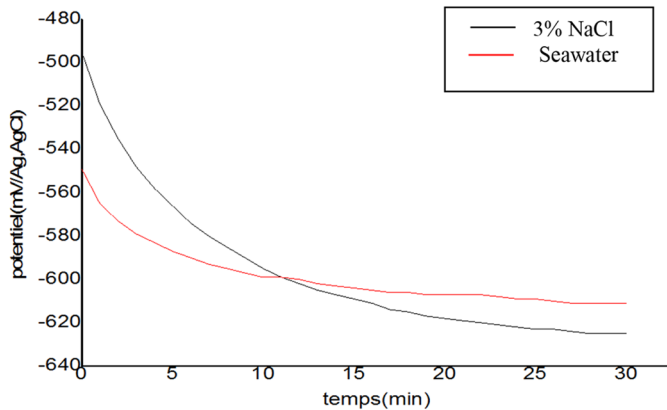


Fig. 5. Variation of open circuit potentials vs. immersion time of the damaged primer in 3 wt.% NaCl solution and sea water.

environment the reading potential (-611 mV/Ag, AgCl) is greater than in 3% NaCl solution (-625 mV/Ag, AgCl). This fact allows us to say that the 3% NaCl solution was more aggressive than sea water, with a large value of the polarization resistance of the order of 3.29 $\text{k}\Omega\cdot\text{cm}^2$ in sea water and 2.19 $\text{k}\Omega\cdot\text{cm}^2$ in 3% NaCl solution.

3.2.2 Potentiodynamic polarization curves

The curves given in Figure 6 relative to the behavior of our damaged primer, characterize the general shape of the polarization curves obtained in different media: 3% NaCl aqueous solution and sea water in the above experimental conditions. These curves are plotted after potentiokinetics stability free corrosion potential.

According to the shape of polarization curves of Figure 6 and the results summarized in Table 4, we note that the damaged primer has better behavior in sea water than in 3% NaCl solution. It is confirmed by the values of i_{corr} (0.172 mA/cm^2 for sea water and 0.210 mA/cm^2 for 3% NaCl). We also note that our tested primer has good resistance in sea water environment: 219.01 $\Omega\cdot\text{cm}^2$ for 3% NaCl: 145.88 $\Omega\cdot\text{cm}^2$. This fact is due to the conductivity of the medium and the concentration of salts in the sea water.

3.3 Study of barrier properties of damaged epoxy paint by Electrochemical Impedance Spectroscopy in 3% NaCl solution

It is very difficult or impossible to obtain information on the mechanism occurring in coated metal electrolyte interface with classic electrochemical technique. The electrochemical impedance spectroscopy has the advantage of using low-amplitude signals, without disturbing the

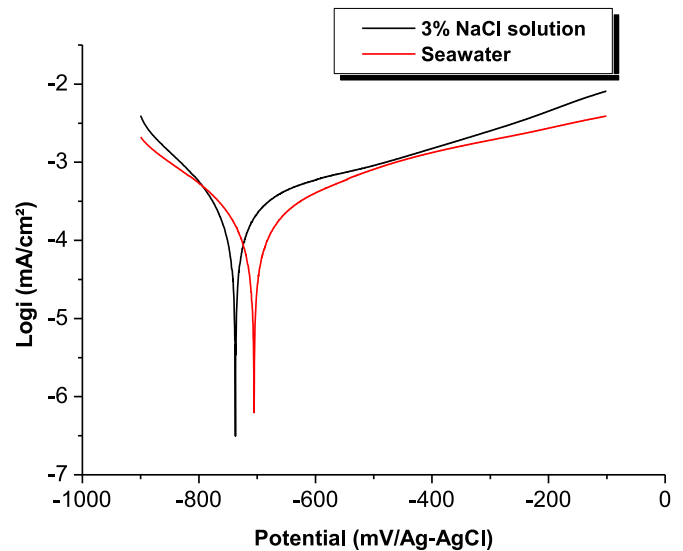


Fig. 6. Influence of environments on the potentiodynamic polarization curves of damaged primer.

Table 4. Values of E_{corr} , R_p , and i_{corr} of damaged primer in different environments.

Medium	E_{corr} (mV)	i_{cor} (mA/cm^2)	R_p ($\text{ohm}\cdot\text{cm}^2$)
3% NaCl	-738.5	0.210	145.88
Sea water	-706.6	0.172	219.01

measured properties, thereby; it offers a convenient way to evaluate the performance of a coated metal system. [14]. Epoxy paints have the ability to actively protect the metal substrate. The sample is coated by a primer paint layer of epoxy zinc whose thickness is of the order of 80 ± 1 μm damaged in sea water. The frequency domain covered was 100 kHz to 10 mHz with the frequency values spaced logarithmically (five per decade). The width of the sinusoidal voltage signal applied to the system was 10 mV. The electrochemical impedance diagrams determined at different immersion times in the 3% NaCl solution normally aerated and unstirred are shown in Figure 7. The values of various parameters are summarized in Table 5.

The impedance spectra represented in the Nyquist plane permits to conclude the existence of two capacitive loops (two constant of time). According to the Nyquist diagrams, several authors assign the loop at high frequencies to the paint. Under the low frequencies, the processes become more and more slow. Thus, in some cases, the transfer resistance can be determined [15,16]. At the limit of the low frequencies, a straight line is attributed to Warburg, interpreting this way the electrolyte diffusion phenomenon through the paint layer [17].

During the 18 h of immersion two separate processes are very well differentiated in the Nyquist diagram (Fig. 7), the EIS spectra can be fitted using the equivalent electrical circuit as shown in Figure 8a. Initially, the appearance of pores on the surface of the coating due to its damage, gives rise to a decrease in R_f according to Table 5, which, in this

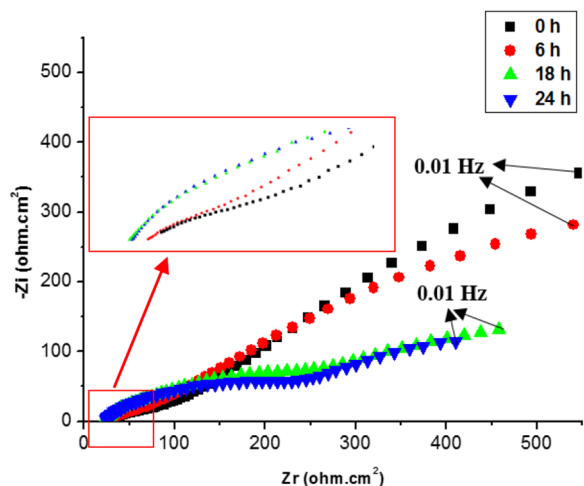


Fig. 7. Evolution of Nyquist diagrams as a function of immersion time in 3% NaCl solution for damaged (in-situ) paint.

Table 5. Parameters values extracted from the fitting procedure.

Time (h)	0	6	18	24
R_s ($\Omega \cdot \text{cm}^2$)	26.02	27.80	16.88	18.10
R_f ($\text{ohm} \cdot \text{cm}^2$)	151.6	117.9	411.5	–
CPE_f ($\mu\text{F}/\text{cm}^2$)	33.16	53.95	966.7	–
R_{ct} ($\Omega \cdot \text{cm}^2$)	2179	1524	655.8	323.9
CPE_{dl} ($\mu\text{F}/\text{cm}^2$)	9200	13 150	30 570	491.3
σ	–	–	–	0.35
α	–	–	–	0.99

case, is a measurement of the resistance of the electrolyte filling such pores in our paint. The in-depth growth of those pores would allow the electrolyte to penetrate the polymer and eventually reach the electrode surface and spread over it. This new electrode–electrolyte interface is easily detected in the impedance spectra by the appearance at low frequencies of a second semicircle, which is fitted to a resistor/capacitor parallel combination in the equivalent circuit, namely a charge transfer resistance R_{tc} and a double-layer constant phase element CPE_{dl} respectively [18]. The increase in CPE_{dl} values is an indicator of the deterioration of our paint due to an increase in the porosity of our film [19]. Depending on the values of R_f and the change in CPE_f , it can be concluded that the film of the corrosion products which form at the metal/solution interface through the pores is not stable. The Randles circuit is the simplest equivalent circuit that describes an electrochemical cell where a single-step Faradaic process in the presence of diffusion may occur at the coating/steel interface (Fig. 8b). It combines four components, namely the solution resistance between working and reference electrodes (R_s), constant phase element CPE_{dl} and resistance R_{ct} show the double layer capacitance and charge transfer resistance of electrochemical dissolution of zinc particles, respectively [18]. After specific exposure time and the so-called Warburg Impedance (Z_w), which

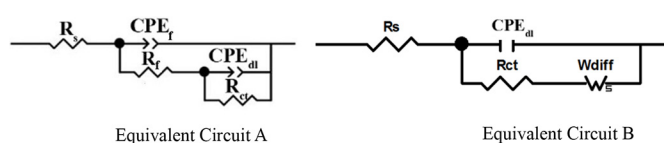


Fig. 8. Electrical equivalent circuit used to model the experimental impedance results.

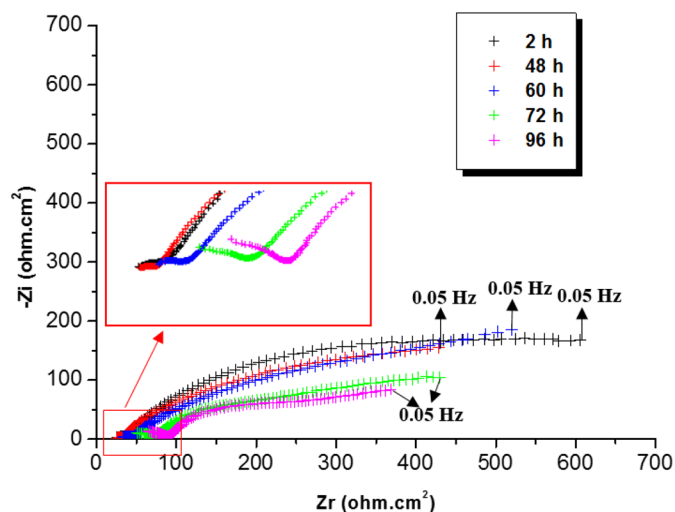


Fig. 9. Evolution of Nyquist diagrams as a function of immersion time in 3% NaCl solution for coated steel.

reflects the influence of the mass transport of the electroactive species on the total impedance of the electrochemical cell. In this case the diffusion process is suggested as being the controlling step and, with regard to impedance, the electrical behaviour of the interface is dominated by the Warburg element.

The R_{ct} and CPE_{dl} are the two parameters which are normally used to specify the disbanding of the coatings and the onset of corrosion at the interface. These parameters decrease the R_{ct} and increase the capacitances during the immersion time (shown in Tab. 5), indicating the entry of electrolyte into the epoxy coating [20], this penetration can be passed through breakdown sites of the coating [21]. The change in CPE_{dl} values may depend either on disbanding of the coating or on the accumulation of corrosion products during the corrosion processes [22]. Variation in capacitance with immersion time could be induced with water uptake in coatings. The penetration of Na^+ and Cl^- ions in the coatings can also influence the capacitance [23].

3.4 Study of barrier properties of non-damaged epoxy paint by Electrochemical Impedance Spectroscopy in 3% NaCl solution

At the beginning of the immersion period, the impedance measurements made on $50 \mu\text{m}$ polyamide epoxy paint in a frequency range of $10^5 \text{ Hz} - 10^{-2} \text{ Hz}$ revealed a fully capacitive behavior. The Nyquist impedance diagrams

Table 6. Parameters values extracted from the fitting procedure.

Time (h)	2	48	60	72	96
R_s ($\Omega \cdot \text{cm}^2$)	29.26	27.31	41.10	70.09	88.07
E_{corr} (mV)	-714	-610	-635	-575	-546
R_f ($\text{ohm} \cdot \text{cm}^2$)	809.2	722	–	–	–
CPE_f ($\mu\text{F}/\text{cm}^2$)	786.6	3.08×10^3	–	–	–
R_{ct} ($\Omega \cdot \text{cm}^2$)	–	–	588.2	272.7	201.2
CPE_{dl} ($\mu\text{F}/\text{cm}^2$)	–	–	481.6	233.3	249.9
σ	–	–	0.251	0.143	0.184
α	–	–	0.99	0.96	0.99

determined at different immersion times in 3% NaCl solution are shown in Figure 9. The values of various parameters are summarized in Table 6.

At the beginning of immersion the Nyquist diagrams show one semicircle in short exposure times and the loop come to be smaller with increasing exposure time. The EIS spectra at 48 h can be fitted using the equivalent electrical circuit as shown Figure 10a. In this circuit, R_s represents the NaCl electrolyte resistance, CPE was applied rather than the “ideal” capacitance taking into consideration heterogeneous, roughness of the surface, and porous nature of the zinc-rich film [24,25], R_f represents the charge transfer resistance processes occurring within the pores of the film. The decrease of the diameter of the semi-circles in the early stage of immersion is in agreement with the rapid decrease of corrosion potential as shown in Table 6. R_f has been generally explained as the pore resistance of the coating due to electrolyte penetration [26] or as area where solution easily uptake, particularly, pre-existing holes or porous areas [27]. When zinc particle was integrated into the organic binder, the pore could be created around the particle due to weak adhesion between the particle and binder. Through the pore, solution could percolate into the coating film easily decreasing the value of R_f , as can be seen in Table 6.

The corrosion potential shifted anodically to positive values because of a reduction in Zn/Fe area ratio caused by wetting steel substrate with electrolyte penetration through the porous coating. Rapid increase in E_{corr} towards less negative potential values typical of steel and close to -546 mV (Ag/AgCl). After 60 h evolution of one time constant in the high frequency range and Warburg impedance element in the low frequency range characteristic for diffusion in a finite layer can be seen. The EIS spectra can be fitted using the equivalent electrical circuit as shown Figure 10b. The fitting procedure using a constant phase element is related to the double layer capacitance CPE_{dl} in parallel with a charge transfer R_{ct} (Faradaic due to reaction of zinc with electrolyte) followed by a Warburg-type impedance (diffusion through a film). The first loop in the high frequency range is related to the coating properties and the second loop at lower frequencies represents the corrosion process, i.e. zinc dissolution process. The impedance reduction during the immersion time is due to zinc particle activation, while the subsequent increase is due to a decrease in the active surface area

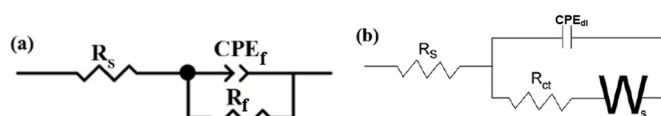


Fig. 10. Equivalent circuit used for numerical fitting of the impedance plots obtained for the different immersion times: (a) short immersion times; (b) long immersion times.

caused by zinc depletion and zinc corrosion product accumulation in the pores of the coatings. Spectra depression is also ascribed to the porous nature of the film [26,27]. CPE_{dl} is accepted as a measurement of the area over which the coating has disbonded [27,28]. In contrast, Amirudin recognized CPE_{dl} as a measurement of the electroactive area rather than degradation area [29]. The integrated of zinc particles in the coating increases the electroactive area in the coating because zinc particles have their own active surfaces [26]. For that reason, the integrated of active zinc particles increased the double layer capacitance remarkably.

4 Conclusion

In this work is shown the approach of electrochemical impedance data interpretation by equivalent electrical circuits of our organic coated metals showing some example relevant to our metal substrates (carbon steel). The influence of the presence of defects on the EIS measurements was also discussed for the different examples. All these parameters can influence the electrochemical behavior measured by EIS, and therefore also the electrical models (equivalent electrical circuits) used to explain the impedance results.

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