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Corrosion inhibition by chromate and phosphate extracts for iron substrates studied by EIS and SVET

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Abstract

The anticorrosive performance of zinc chromate and zinc phosphate, used as extracts in 0.1 M NaCl, was studied using electrochemical impedance spectroscopy (EIS), the scanning vibrating electrode technique (SVET) and open circuit potential (OCP) measurements. Neither of the pigments managed to totally prevent corrosion. Phosphate had a much lower inhibiting efficiency and acted at a very low rate, whereas chromate acted immediately after immersion, but lost some of its action after a few hours of continuous immersion. Iron corroded uniformly in the phosphate extract, whereas in the chromate extract nucleation of metastable pits occurred. Evolution of the chromate layer with time was revealed in the EIS spectra in the form of a relaxation constant that developed during immersion, which was interpreted as being due to the formation of a porous healing layer formed on repassivated pits. The joint use of open circuit potential measurements, EIS and SVET is illustrated as a means of assessing different phenomena on the metal surface.

Keywords: Corrosion; Zinc chromate; Zinc phosphate; Anti-corrosion pigments; EIS; SVET

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1. Introduction

Zinc chromate has for many years been used as inhibiting pigment for steel. Its toxicity, however, is leading to great restrictions to its use and has caused intensive research on alternative pigments with better human compatibility. The inhibitive action of chromate seems to be based upon the oxidizing power of Cr(VI), which becomes reduced to Cr(III) in a reaction that neutral pH occurs as

$$2CrO_4^{2-} + 5H_2O + 6e^- \rightarrow Cr_2O_3 + 10OH^-$$

forming a layer of chromium oxide. The healing properties of chromate arise from the fact that this reduction occurs at sites where oxidation of iron initiates, as:

$$Fe \rightarrow Fe^{2+} + 2e^-$$

Although chromate ions participating in the cathodic reaction come essentially from the solution, it seems that conversion coatings formed on the surface of steel can have a high content of Cr(VI), which acts as a source of oxidising agent whenever corrosion starts [1,2]. Chromium oxides, namely Cr_2O_3, are thermodynamically stable above pH 5, but reports have been given that for acidic solutions the oxidising power of Cr(VI) ions can enhance corrosion of steel [3]. Chromate pigments are frequently used in the form called *zinc yellow*, a classical pigment consisting of a mixed salt of zinc chromate, potassium chromate and zinc hydroxide. The mode of action of this pigment, although essentially based upon the chromate, is reinforced by the hydroxyl ion from the zinc hydroxide, which raises the pH, enhancing the stability of Cr(III) salts, whereas Zn^{2+} precipitates as $Zn(OH)_2$ at cathodic sites, creating also a protective layer [4].

Zinc phosphate, which is probably the most important alternative to chromate, has been used either as soluble inhibitor, in the formulation of conversion coatings and also as anticorrosive pigment in paints [5–7]. Solubility of zinc phosphate is higher in acidic media [8] and therefore phosphate pigments are usually considered to be more effective at low pH.

In a previous work [9], on the action of zinc chromate and zinc phosphate pigments on zinc substrates it was concluded that both pigments were effective in 0.1 M NaCl, although the phosphate did not quite match the high efficiency of chromate. It was also observed that the inhibiting action of chromate was practically instantaneous, whereas phosphate took several hours to achieve total protection of the surface. In the present paper, which corresponds to the second part of that study, the action of the same pigments is studied for pure iron.

2. Experimental

2.1. Samples

The substrate consisted of iron foil (from Goodfellow, Ltd, UK, 99.5% pure). For the EIS study, 1 cm^2 samples were electrically connected via a copper wire with silver

Table 1
Characterization of the exposure media

	pH	Conductivity (mS cm^{-1})	Concentration (M)
Control, 0.1 M NaCl	6.20	9.44	–
Chromate extract in 0.1 M NaCl	6.55	10.90	$\lvert Cr \rvert_{total} = 1.1 \times 10^{-2}$
Phosphate extract in 0.1 M NaCl	6.43	9.46	$\lvert PO_4^{3-} \rvert = 4.8 \times 10^{-5}$

conductive adhesive and epoxy-mounted. For the SVET measurements, samples were also epoxy-mounted, leaving an exposed area of ≈1 mm^2. Prior to the tests, the iron surface was polished with SiC grit papers of grades 220, 500, 800 and 1000, washed in deionised water (Millipore) and ethanol and finally dried with compressed air.

2.2. Exposure media

Pigment extracts were produced by magnetic stirring 1 g of pigment in 0.5 L of 0.1 M NaCl aqueous solution for 24 h and filtering twice, as made in [3]. The pigments were zinc yellow ($K_2CrO_4 \cdot 3ZnCrO_4 \cdot Zn(OH)_2 \cdot 2H_2O$) and zinc phosphate ($Zn_3(PO_4)_2 \cdot 2H_2O$). The concentrations of chromate and of phosphate were determined by Inductively Coupled Plasma and by Molecular Absorption Spectrometry, respectively (Table 1). Plain 0.1 M NaCl solution, without pigment addition, was also used for control.

2.3. Electrochemical techniques

SVET measurements were made using the Applicable Electronics Inc. setup, with a 40 μm platinum tip microelectrode vibrating 200 μm above the surface, with an amplitude of 60 μm. Commercial adhesive tape glued around the sample holders worked as solution reservoir. Each scan comprised 20 × 20 points.

EIS was made in Gamry instrumentation, in the frequency range 50 kHz to 5 mHz, with a sinusoidal potential signal of 10 mV rms around the open circuit potential, using a three-electrode cell, with a platinum wire as counter electrode and a saturated calomel electrode as reference. Measurements were made at room temperature, inside a Faraday cage, and the solution was quiescent and exposed to air. Spectra fitting was made using ZView 2.70 software from Scribner Associates Inc.

3. Results

3.1. OCP

In the control solution, evolution of the open circuit potential is characterised essentially by a slow decay to approximately −0.7 V (Fig. 1(a)), which is slightly above the reversible potential for the oxidation from Fe to Fe^{2+}. This potential drop

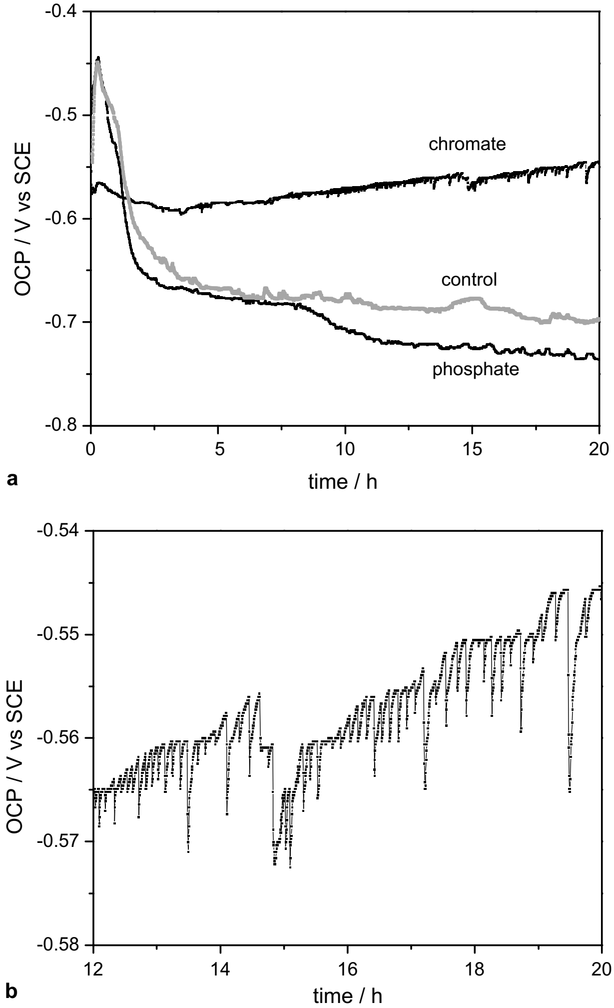

Fig. 1. (a): Open circuit potential variation of pure iron in 0.1 M NaCl, with and without pigment extracts; (b): Scale expansion for part of the curve relative to the chromate extract.

can be related to the dissolution of the native oxides and thus to the activation of the metal, but also to other phenomena such as a raise in pH or some depletion of oxygen at the metal surface, both due to the cathodic reaction. The presence of phosphate extract did not significantly affect the potential, although a slight cathodic polarization seemed to occur after ~9 h. In the chromate extract, in contrast, the potential was much higher, which is a typical behaviour for a metal in the presence of a dissolved oxidant. This effect was observed since the first seconds of immersion, revealing that the formation of a passivating chromate layer occurred at a very high rate. The corrosion potential increased smoothly during the immersion period, probably due to thickening of the chromate layer. For the sample presented, the final potential was quite high, reaching 150 mV above the other samples. Towards the end of the period, numerous phenomena of potential drop followed by recovery suggest the nucleation and repassivation of metastable pits (Fig. 1(b)). For the several samples tested, the chromate efficiency was not totally reproducible. In some cases the potential was more anodic, approaching the behaviour of the other two systems, and corrosion took place in a relatively high extension, whereas in other cases the potential remained high, as in the case presented, but without any fluctuations. The result presented in Fig. 1(b) is thus one of an intermediate performance, but it is interesting because it is known that chromate pigments are sensitive to the presence of chloride ions [3], and can loose efficiency at high Cl^- concentrations. Being a strong oxidant, chromate passivates iron, shifting its potential to the passive range. It seems however that, as the film becomes either thicker or at least aged in the chloride solution, further shifting of the potential brings it to the vicinity of the pitting potential for iron in this solution, and therefore a competition between pitting nucleation and the healing power of chromate is established, leading to localized phenomena of breakdown and repassivation. Eventually, this competition will lead to the real development of stable pits.

3.2. SVET

Fig. 2 shows selected SVET current maps of pure iron immersed in the NaCl control solution, and also the effect of having the pigment extracts. In the chromate extract, the maps have shown nearly total absence of currents for the complete duration of the experiment in agreement with the open circuit potential. Inspection of the sample at the end of the experiment showed a shiny surface (Fig. 2–c4), although closer inspection revealed the existence of three small pits on the surface. The formation of these pits was not detected by the SVET, on any of the numerous maps obtained with this particular sample. The reason for this is that these pits nucleate and repassivate fast, and therefore the probability of the probe to be actually above one metastable pit at the exact time of one anodic burst is very low. Further, some of the pits may have nucleated during the interval between two subsequent scans. Nevertheless, this means that none of these pits proceeded for much time, due to the healing effect of chromate.

For the sample immersed in the solution with phosphate extract, and also for the control sample, the electrochemical activity was incipient in the first minutes of exposure, but became significant after 1 h. This is the period that corresponds to the observed drop of the open circuit potential, and confirms that the potential drop reveals an activation of the surface. The anodic activity is located in a small fraction of the area, with relatively high current densities, whereas the cathodic reaction is spread along the rest of the surface, with a comparatively weak signal. The action of phosphate was only observed for long exposure periods. In fact, after 24 h of immersion, the corrosion process was reduced again to very low values, in contrast to the on going activity of the control sample. Sample inspection at the end of exposure revealed extensive coverage of the surface by corrosion products with a gel-like aspect,

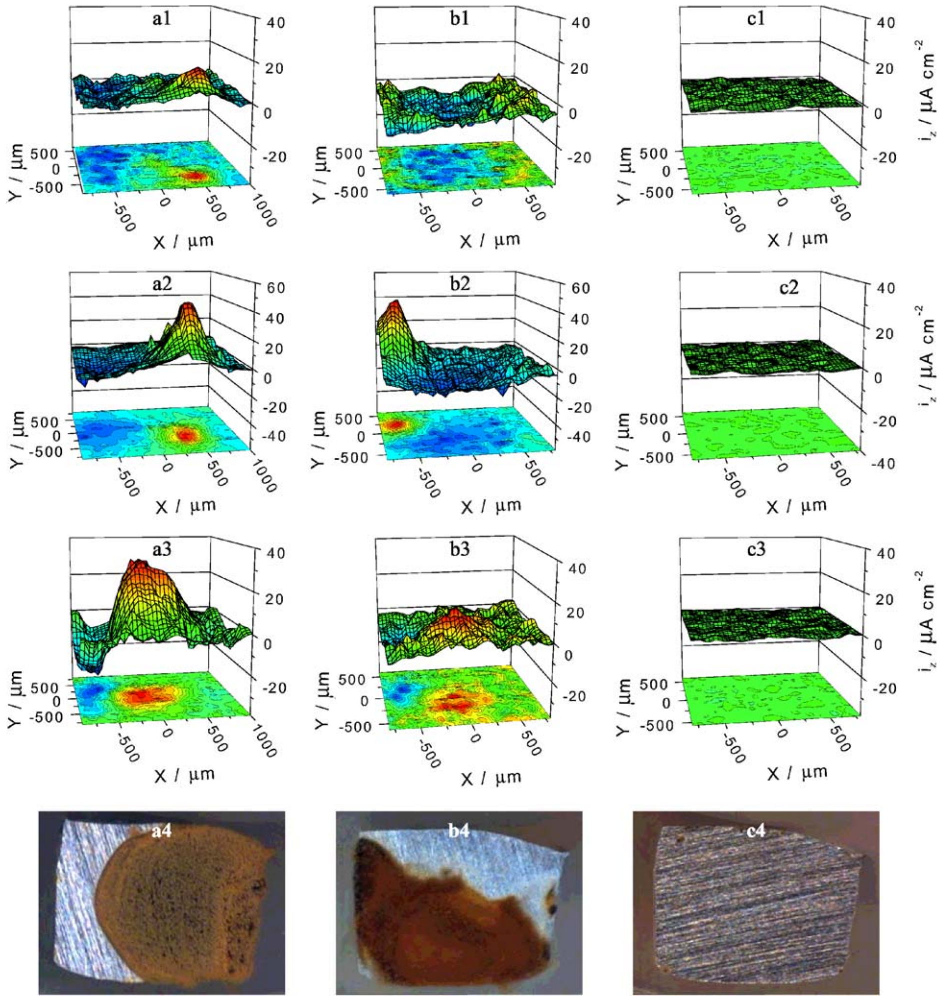

Fig. 2. Ionic current mapping of pure iron exposed to: 0.1 M NaCl without pigment (a); with zinc phosphate extract (b); and with zinc chromate extract (c). Times of immersion are (top to bottom): ~5 min, 1 h and 1 day. Also shown are video pictures of the surface at the end of the experiment (a4 to c4).

probably consisting of a mixture with the pigment. This gel-like structure was not observed in the control sample.

3.3. EIS

Impedance spectra obtained in the three solutions for selected times of immersion are presented in Fig. 3, where both the Bode and the Nyquist plots are included, for better understanding of the systems. In the absence of any pigments (Fig. 3-(a1) and (a2)) the spectra reveal only one relaxation process, with the low frequency resistance

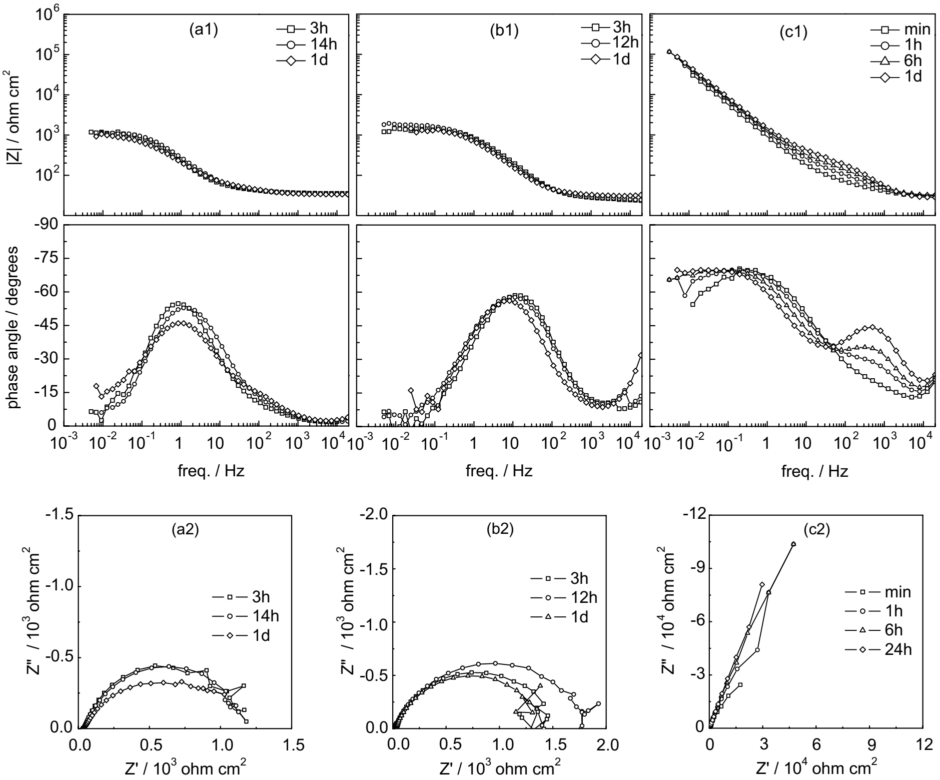

Fig. 3. Impedance spectra obtained for pure iron at different times of exposure to 0.1 M NaCl (a), phosphate extract (b) and chromate extract (c).

corresponding practically to the charge transfer resistance (added to the solution resistance, although this is not significant), whereas at higher frequencies the response is due to the double layer capacitance. The charge transfer resistance remained practically constant for the period of the test. In the presence of phosphate, the general shape of the spectrum was the same (Fig. 3-(b1) and (b2)), although the values for the charge transfer resistance were slightly higher, possibly due to the phosphate inhibiting effect. The chromate, in contrast, produced not only a growth in the total impedance (Fig. 3-(c1) and (c2)), but also a total change in the equivalent circuit describing the spectrum. The low frequency region of the spectra is capacitive and only a slight decrease of the phase angle near the end of the spectrum suggests the existence of a resistance plateau below the window of frequencies measured. This initial capacitive spectrum changes with time, with the development of a second relaxation process, at frequencies above 100 Hz. This new relaxation process has a low resistance, but it cannot reveal active corrosion, since the total impedance of the system remains high. It will therefore be ascribed to a chromate layer, with properties different from those of the film formed in the first instants of immersion, and possibly including iron ions from the corrosion bursts occurring at pits before they became repassivated. The shape of the spectrum is identical to that observed by Jüttner [10] for porous oxide layers.

In the control solution and also in the presence of the phosphate extract, the spectra can be described by a simple Randles circuit, as presented in Fig. 4(a). For the sake of fitting quality, all the capacitors were taken as Constant Phase Elements (CPE or Q), whose impedance is given by $Z_Q = Y_0^{-1}(j\omega)^{-n}$, where Y_0 is the frequency independent admittance, n is the power of Q, ω is the angular frequency (in radians) and $j = \sqrt{\,} - 1$. The value of n gives information on the nature of the CPE, being $n = 1$ for a pure capacitor and $n \sim 0.5$ for a mass transfer process. Since the impedance of a capacitance is $Z_C = (j\omega C)^{-1}$, the fitted values of Y_0 will be used for the assessment of the capacitance. Example of a fitting obtained with the Randles equivalent circuit is shown in Fig. 4(b), in which only the values below ~2 kHz were considered. For the chromate extract solution, the equivalent circuit comprises a second R–C circuit, as presented in Fig. 5(a), which represents the effect of the chromate layer. Adjustment of the theoretical to the experimental data is quite satisfactory, as can be seen in Fig. 5(b).

Evolution of the fitted parameters with time of immersion is presented in Fig. 6. The charge transfer resistance in Fig. 6(a) does not change significantly during immersion and is of the order of $10^3\ \Omega\,\mathrm{cm}^2$ in the control solution and also in the presence of phosphate. In the chromate the charge transfer resistance is not clearly defined in the spectra, but a rough estimate can however be made based upon the lowest frequency measurements, for which the phase angle starts to decrease. Based on this estimate (presented in Fig. 6(a) by a dashed line), the charge transfer resistance is several orders of magnitude higher than in the other cases, starting at

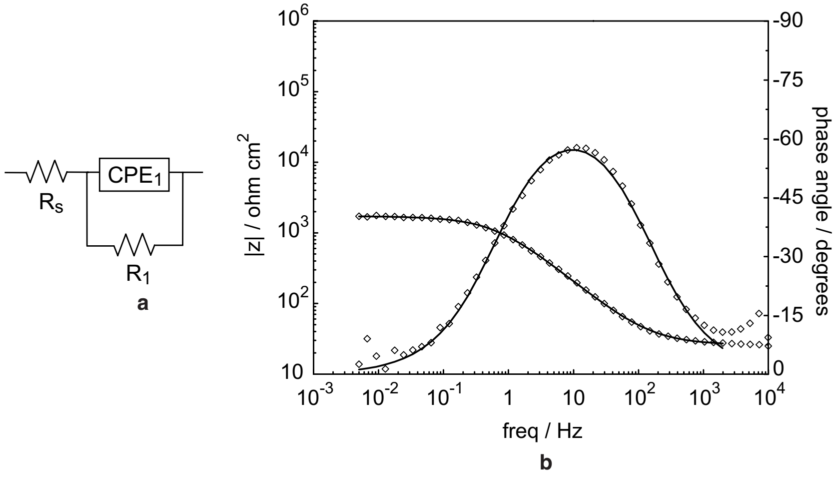

Fig. 4. Equivalent circuit (a) and spectrum fitting (b) for pure iron in phosphate extract + 0.1 M NaCl. Fitting parameters: $R_s = 26\ \Omega\,\mathrm{cm}^2$; $Y_0(\mathrm{CPE}_1) = 1.98 \times 10^{-4}\ \mathrm{F\,cm}^{-2}\,\mathrm{s}^{n-1}$; $n(\mathrm{CPE}_1) = 0.78$; $R_1 = 1.7\ \mathrm{k}\Omega\,\mathrm{cm}^2$; $\chi^2 = 1.4 \times 10^{-3}$.

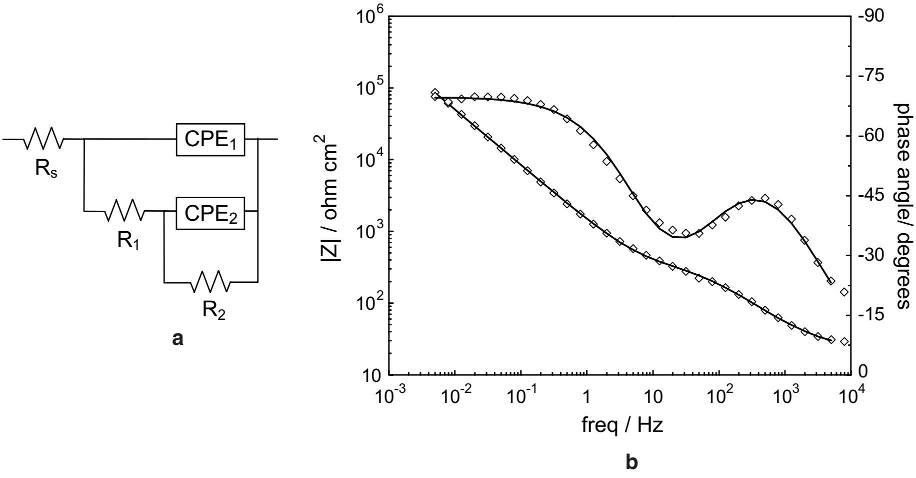

Fig. 5. Equivalent circuit (a) and fitting (b) for pure iron in chromate extract + 0.1 M NaCl. Fitting parameters: $R_s = 22\ \Omega\ \mathrm{cm}^2$; $Y_0(\mathrm{CPE}_1) = 4.1 \times 10^{-5}\ \mathrm{F\ cm}^{-2}\ \mathrm{s}^{\mathrm{n-1}}$; $n(\mathrm{CPE}_1) = 0.73$; $R_1 = 387\ \Omega\ \mathrm{cm}^2$; $Y_0(\mathrm{CPE}_2) = 1.3 \times 10^{-4}\ \mathrm{F\ cm}^{-2}\ \mathrm{s}^{\mathrm{n-1}}$; $n(\mathrm{CPE}_2) = 0.8$; $R_2 = 7 \times 10^{11}\ \Omega\ \mathrm{cm}^2$; $\chi^2 = 1 \times 10^{-3}$.

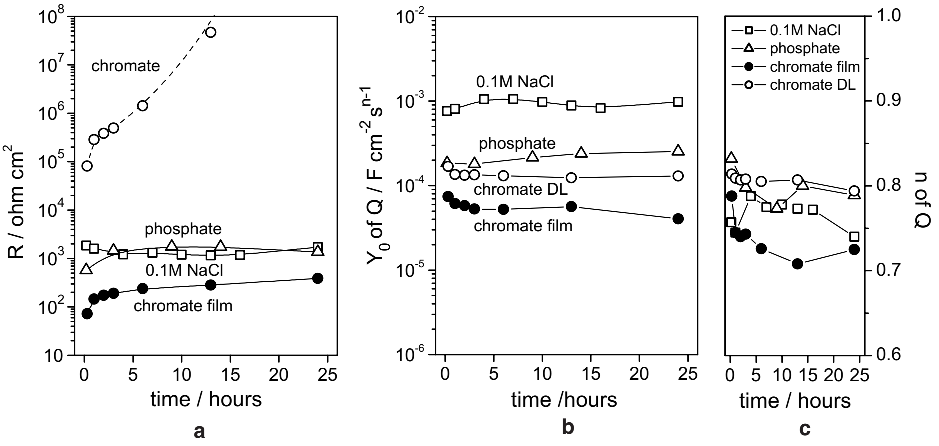

Fig. 6. Fitted parameters of resistance (a), Y_0 (b) and exponent n (c), for the impedance spectra obtained for pure iron at different times of exposure to 0.1 M NaCl, with and without pigment extracts.

$10^5\ \Omega$ and increasing beyond $10^8\ \Omega$. Concerning the double layer capacitance, it has values of approximately 10^{-3} F for the sample in the plain salt solution, but the presence of any of the extracts lowers it by nearly one order of magnitude, as a consequence of a reduced active area. The values of the exponent n are always above 0.7, which also excludes the possibility of a Warburg impedance in Fig. 6(c). Finally, the high frequency process, attributed to the chromate film, had a resistance of 80–300 $\Omega\ \mathrm{cm}^2$ and a relatively high capacitance, of 40–80 μF cm^{-2} (Fig. 6(b)).

The efficiency of an inhibitor can be calculated from

$$\eta = \left(1 - \frac{R_{\mathrm{ct},0}}{R_{\mathrm{ct}}}\right) \times 100\%$$

where R_{ct} and $R_{\mathrm{ct},0}$ are the charge transfer resistance values in the inhibiting solution and in the control solution, respectively. For the chromate solution, the charge transfer resistance cannot be determined with certainty because it is not defined in the spectrum, but it can be taken as being higher than $10^5\ \Omega\ \mathrm{cm}^2$. Taking for the charge transfer resistance the approximate values of $1.2\ \mathrm{k}\Omega\ \mathrm{cm}^2$ and $1.8\ \mathrm{k}\Omega\ \mathrm{cm}^2$ for the control sample and the one in the phosphate extract, measured after 12 hours of immersion, then the inhibiting efficiency of the phosphate can be estimated as 33%, whereas the one for chromate is above 99%.

4. Discussion

The two pigments tested in this work proved to be quite different, not only in the time required for their action, but also in the morphology of the attack when they failed, and in the efficiency estimated from the impedance measurements. Chromate acted initially at an extremely high rate, totally stopping any corrosion activity since the first seconds of immersion. It is known that the time for surface blocking by chromate is affected by concentration. According to Armstrong et al. [11], for concentrations as low as 5×10^{-5} M, the time to full coverage of iron was approximately 30 s. Since in our case the concentration was higher by more than two orders of magnitude, the time for chromate adsorption was certainly below the time spent for the start of the measurements. Phosphate, in contrast, only seemed to act after several hours of immersion, thus not being able to prevent corrosion onset. The action of phosphate was poor, with an inhibiting power of only 20%. The pigment seems to act at a low rate, leading to a gel-like structure that was observed at the end of the experiment. Corrosion in this case proceeded along the surface, tending towards uniform corrosion, whereas with chromate, it was originated at localized spots, starting by phenomena of metastable pitting.

Chromate acted as a strong oxidant, raising the open circuit potential by $\sim$150 mV, whereas phosphate did not significantly affect the potential, therefore acting probably by a barrier effect. According to the literature, phosphates act by a "pore-plugging" mechanism, stabilizing the oxide layer presumably through the formation of hydrated $\mathrm{FePO_4}$ [12,13]. Further, phosphates are oxygen-dependent inhibitors [14], and thus their rate of action can ultimately be determined by the rate of oxygen diffusion to the surface, especially after the metal itself has consumed most of the oxygen in its own oxidation. Finally, another crucial variable in the action of inhibiting pigments is pigment solubility. Although the initial amount of solid pigment was the same, the concentrations achieved were quite different, the concentration of phosphate in the extract was three orders of magnitude lower, a factor that can be responsible for its comparatively low efficiency. As mentioned above, solubility is strongly influenced by pH, and phosphate has lower solubility at neutral than at acidic pH [8].

It is interesting to note the difference between the impedance response and the SVET. In the phosphate solution and also in the control solution, relevant features were observed in the SVET mapping, namely the spatial distribution of cathodes and anodes and their variation in time, i.e. the localized character of corrosion was evidenced. Also the inhibiting effect of phosphate, at long exposure times, was observed. For the same systems, the impedance spectra reveal corrosion proceeding at a practically constant rate, with no significant changes in the spectra. For the chromate extract, however, the SVET mapping reveals practically no activity on the surface, whereas the impedance spectrum clearly shows the changes associated with the slow growth of a second time constant.

This time constant has a resistance that increases with time, from ~ 60 to $\sim 300\ \Omega\ \mathrm{cm}^2$, whereas its capacitance decreases. For a dielectric layer, the capacitance is inversely proportional to the thickness d, according to

$$C = \varepsilon\varepsilon_0 A/d$$

where ε is the dielectric constant, ε_0 the permittivity of vacuum and A the area, whereas the resistance is directly proportional to d, provided that the resistivity remains constant. Thus, both the resistance rise and the capacitance drop are consistent with a process of thickening of a film, but probably only at the sites where nucleation and repassivation of pits has taken place. This film thus covers only a small fraction of the surface, but is highly porous, very thick and consisting of a mixture of chromate, iron cations and other ions from the solution, which can lead to a high dielectric constant and therefore to a high resulting capacitance.

The action of chromate can thus be described in two stages, in agreement with what has been described by other authors, and each of these stages can be associated with one time constant in the impedance spectrum: initially, an instantaneous monolayer of chromate is formed on the surface, protecting the substrate by anodic polarization into the passive region. This results in a capacitive behaviour on the impedance spectrum. This initial film is probably made of Cr(III) and Cr(VI), and it will then adsorb more Cr(VI) ions from the solution. This film is transparent and covers the entire surface. Later, pit initiation at weak sites on the surface will be followed by fast repassivation. A thick layer, rich in Fe(II) oxides and hydroxides and also in Cr_2O_3, is then formed on these pits. This layer is coloured due to the presence of iron oxides and has a relatively low resistance because it is porous. Although it is not a passive layer, it can inhibit the growth of the pit by a barrier effect, as confirmed by the high values of the total impedance of the system.

In the first part of this study [9], it was observed that zinc, under the same conditions of exposure to the inhibitor, was protected from corrosion with both the pigments. It was also determined that the chromate layer formed spontaneously on zinc had a smaller resistance ($10^5\ \Omega\ \mathrm{cm}^2$) and a smaller capacitance, in the range $10^{-6}\ \mathrm{F\ cm}^{-2}$. Further, there were no signs of the high frequency time constant, but this difference is thought to be due to the inexistence of pitting corrosion for zinc in the presence of chromate inhibitor.

In our previous studies it was also observed that zinc phosphate decreased the corrosion rate by one order of magnitude. With iron, however, the inhibiting effect was much weaker, possibly due to the non-protective character of iron oxides. According to Isaacs et al. [2], chromate ions compete with oxygen for blocking of the surface. The way in which they block the surface, however, is not totally clear. According to Onochukwu, $Cr_2O_7^{2-}$ becomes adsorbed on a Fe atom on the surface [15], liberating its two free electrons, which can be used for the reduction of H^+. This adsorption, which occurs within the first few seconds of exposure, produces on the surface a storage of Cr(VI) that will promptly become reduced to Cr_2O_3, healing the passive film, whenever iron ions start to develop locally. The reaction is not only thermodynamically favourable, but is also stoichiometrically efficient, since each mole of ions reduced produces five moles of hydroxyl ion, which increases the pH locally and therefore helps to inhibit pitting growth.

5. Conclusions

Zinc phosphate provided no significant inhibition to iron corrosion in 0.1 M NaCl in the first hours of exposure. Inhibition by the phosphate was only achieved after long exposure times, due to the slow kinetics of phosphate deposition. Corrosion attack in the presence of phosphate occurred on a relative large area, probably leading to uniform corrosion.

The chromate pigment was capable of inhibiting corrosion of iron since the first seconds of immersion, but could not prevent nucleation of metastable pits at a later stage. It is believed that formation of the chromate layer occurs in two stages, with different rates. The first one consists of instantaneous blocking of the surface and dramatically reduces the metal dissolution, whereas the second corresponds to the formation of a mixed layer of chromate and corrosion products and is formed at nucleated pits on the surface. This does not correspond to significant activity, since it is accompanied by an increase of the total impedance of the system.

The joint use of the scanning vibrating electrode technique, electrochemical impedance and open circuit potential gave information on the several aspects of the system properties, corrosion rates and morphology. Namely, the SVET could not detect the nucleation and repassivation phenomena on the surface, whereas only the impedance was sensitive to the changes on the properties of the chromate film. Finally, the OCP was the only technique that could detect the fast phenomena of pit nucleation and repassivation.

Acknowledgements

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