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Electrochemical Evaluation of Chemical Treatment Effect on Corrosion Process of Carbon Steel in Oil Atmospheric Distillation Environments

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The effect of chemical treatment composed of mono ethanol amine and a commercial imidazoline based corrosion inhibitor on corrosion process of carbon steel in an acid sour solution typically found in oil atmospheric distillation plants was studied. Electrochemical impedance spectroscopy characterization showed the aggressiveness of the sour media alone. A protective behaviour is evidenced at different pH values, which is enhanced when inhibitor is added. The better corrosion control is attained at pH 6.5 and 30 mgL^{-1} of inhibitor. This behaviour is explained on the basis of the formation of a mixed corrosion film composed of inorganic – organic compounds. SEM characterization supports these results.

Keywords: Sour acid environment, Corrosion, Atmospheric distillation, EIS, pH.

1. INTRODUCTION

In the oil refining facilities, severe corrosion damages are frequent [1, 2]. The main evidence of this problem is a drastic decline of the useful life of equipment and accessories. Corrosion takes place preferentially in the low-temperature sites of the components of atmospheric distillation units [1], i.e. in the upper trays and domes, as well as in the overhead heat exchangers and vessels. This is caused by hydrolysis of calcium and magnesium chlorides favouring the formation of hydrochloric acid, as well as other species, such as hydrogen sulphide, hydrogen bisulphide, ammonium, carbon dioxide, low-molecular weight carboxylic acids, naphthenic acid, among others [3, 4]; however, hydrochloric acid and hydrogen sulphide remains as the more aggressive species generating the formation of a corrosive acid sour media reaching pH values around 2.0 [5].

Despite the extensive use of chemical treatments in real operations, corrosion – and its related economic and environmental consequences - remains a serious problem that needs to be controlled. Due to the complexity of the phenomenon, different approaches have been taken [6, 7]. At present, corrosion control is accomplished through the use of inhibitors and neutralizers whose performance depends on the column feed and the significant variation of salts [8-11]. About neutralizing compounds, a variety of amines have been traditionally utilized to maintain the pH of condensates near to 7 [12-14]. Furthermore, corrosion inhibitors (CI) are substances able to significantly decrease corrosion damage even at low dosages [15].

In order to investigate the effect of the chemical treatment composed by mono ethanol amine (MEA) as neutralizer and a commercial imidazoline based CI, on the corrosion phenomenon of SAE 1018 carbon steel (CS) in acid sour solution, an electrochemical characterization was carried out using electrochemical impedance spectroscopy (EIS) technique, using a representative solution of this media composed by hydrochloric acid 0.05 M and 500 mgL^{-1} of hydrogen sulphide (pH 2.0) as was established before [1, 4, 5].

2. MATERIALS AND METHODS

2.1 Preparation of electrodes

A carbon steel (CS) cylinder with an area of 0.5 cm^2 exposed to the solution and mounted in a Teflon tube was used as working electrode. The counter electrode was a graphite rod. As reference, a standard saturated calomel electrode (SCE) was employed. In all working electrodes, the exposed surfaces were prepared by mechanical grinding with up to 600-grit water-cooled SiC papers so as to obtain a reproducible surface and subsequently cleaned in an ultrasonic bath for 1 minute in acetone.

2.2 Preparation of test solutions

All reactive were analytic grade. To prepare the acid sour solution, a hydrochloric acid 0.05 M solution was made and then sodium sulphide was added in the proper quantity to achieve 500 mgL^{-1}, the pH was approximately 2.0. To study the effect of pH modifications, the acid solution was adjusted to the following pH values: 4.0, 5.0, 5.5, 6.0, 6.5 and 7.0 by adding mono ethanol amine (MEA). Also to evaluate a commercial corrosion inhibitor (CI) it was dosed at 5, 15 and 30 mgL^{-1} in the sour solutions previously adjusted to different pH values.

2.3 Electrochemical characterization

The electrochemical characterization was done in a conventional three-electrode cell. An Autolab potentiostat-galvanostat model PGSTAT 30 was utilized. The experimental conditions for the

Electrochemical Impedance Spectroscopy (EIS) tests were: ±10 mV amplitude; frequency sweep from 10 kHz to 10 mHz; 2000 rpm electrode rotation speed; 40 ºC and 3 h of immersion time. According to has been reported in previous work, these conditions are the most oxidizing conditions to investigate the corrosion process of CS in the test solutions here used [4].

2.4 Scanning Electron Microscopy, SEM

Characterization by scanning electron microscopy (SEM) of areas affected by corrosion in the absence and presence of chemical treatment (neutralizing amine, corrosion inhibitor) and the corrosion products formed was carried out with a Philips microscope XL30ESEM.

3. RESULTS AND DISCUSSION

3.1 EIS characterization in acid sour medium

In order to evaluate the electrochemical behaviour of the carbon steel (CS) immersed in the sour medium (hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide at pH 2.0), typical of distillation plants, in Fig. 1 is displayed the typical EIS spectrum (complex plane) rotating the electrode at 2000 rpm. In this plot is evident the aggressiveness of the aqueous solution under this experimental condition, because the impedance values are less than 50 **Ω** cm^2; likely related with an active dissolution of the steel. Taking into account the rapid oxidation of the metal in this medium, it can be considered the formation of corrosion films on the steel with non-protective properties; favouring the steel dissolution towards the aqueous solution. This fact could be related with an apparent linear response at high frequencies, commonly associated with a diffusion process through the films [16]. The oxidation of the steel and this diffusion process can be associated with a polarization resistance by extrapolating the semicircle observed in Fig. 1.

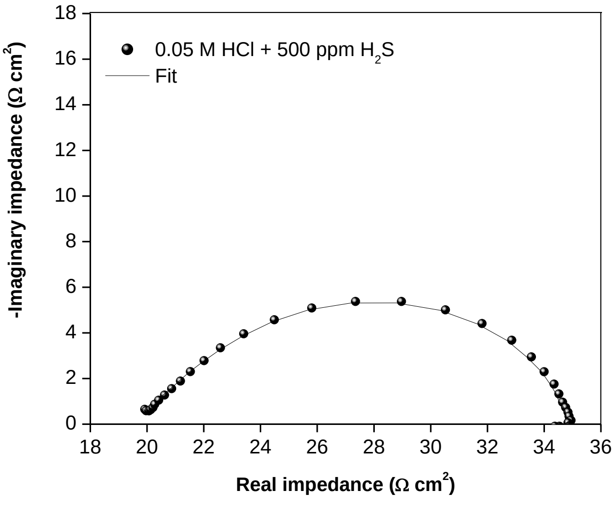

Figure 1. Typical Nyquist diagrams obtained on 1018 CS immersed 3h in the acid sour medium (hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide at pH 2.0), rotating the electrode at 2000 rpm and 40°C.

It is noteworthy the aggressiveness of the medium towards the steel, raising the important control in distillation plants using chemical treatments, i.e. neutralizing amine, corrosion inhibitors or both. To address the inhibition effect of these chemical compounds, first is modified the acid pH to a more neutral condition (varying the dosage of the MEA); after the CI is added to understand their influence on the corrosion of the steel immersed in this sour solution.

3.2 pH effect

In Fig. 2 are presented the complex diagrams of the CS immersed in the aqueous sour medium varying the pH by adding MEA. In these diagrams, it can be seen a similar capacitive response for each spectrum. Notice that the most aggressive condition towards the steel oxidation is at pH 2.0. Also is observed greater modifications in the impedance values, being evident an increase for pH 4.0, 5.0, 6.5 and 7.0; except for pH 6.0 where the semicircle diameter decrease. Based on these results the optimal corrosion control in distillation plants using MEA treatments, only can be performed at pH 4.0 or 5.0.

To explain this fact it has been reported that depending on the potential of zero charge (PZC) of the steel in the sour medium (positive or negative), the adsorption of the chlorides and MEA could be different. If the corrosion potential is positive with respect to the PZC, first occur the adsorption of chloride ions followed by the amino group; meanwhile whether the potential is negative, only the amine group is adsorbed on the steel [17]. In the case that the corrosion potential is similar to the PZC means that the steel is not charged and is considered a minimal adsorption of cations and anions; hence a minimal oxidation of the steel [17].

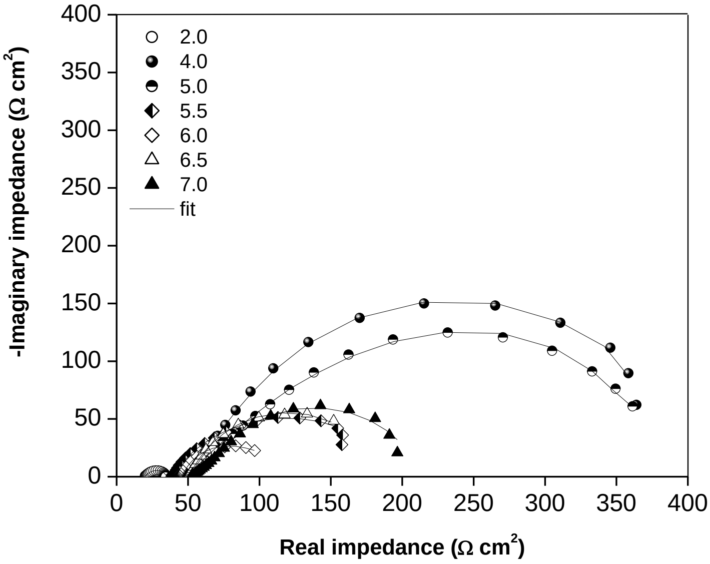

Figure 2. Typical Nyquist diagrams obtained on 1018 CS immersed 3h in the sour medium (hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide) varying the pH; rotating the electrode at 2000 rpm and 40°C.

It had been reported a potential of -530 mV vs SCE as the PZC for a carbon steel immersed in a 5 % hydrochloric acid solution (1.37 M) evaluated by Electrochemical Impedance Spectroscopy (EIS) and Ultraviolet-Visible Spectroscopy (UVS) techniques [17]. The potentials recorded for the different pH is as follows: -573 mV (pH 2.0), -547 mV (pH 4.0), -561 mV (pH 5.0), -539 mV (pH 5.5), -574 mV (pH 6.0), -545 mV (pH 6.5) and -569 mV (pH 7.0) all of them vs SCE. In general these values are negative than -530 mV; indicating that the electrode is negative charged. Based on this assumption it could be suggested the predominantly adsorption of the MEA on the steel instead of the halide ions. It is not surprising that the maxima values reached in the EIS diagrams (pH 4.0 and 5.0), are related when the corrosion potentials are closer to the PZC; meanwhile the most aggressive conditions towards the steel is evident at pH 2.0 and 6.0, when the corrosion potentials are more negative than this PZC. This fact could be due because the rotation and 40 °C, slightly modify the PZC for the steel immersed in the sour medium used in this study. Thus it can be considered an inhibition effect of the steel oxidation varying the pH by the MEA; however it could not be disregarded the possible formation of iron oxides on the steel enhancing its protective behaviour towards the corrosive medium.

In order to evaluate the synergic effect of a CI in the oxidation of the steel in presence of MEA, only are presented results obtained with the most aggressive condition (pH 2.0); when is reached a maximum protection in presence of MEA (pH 4.0); and 6.5, which is a pH value used as control in real conditions. For each experimental conditions it is varied the concentrations of the CI.

3.3 CI concentration effect at pH 2.0

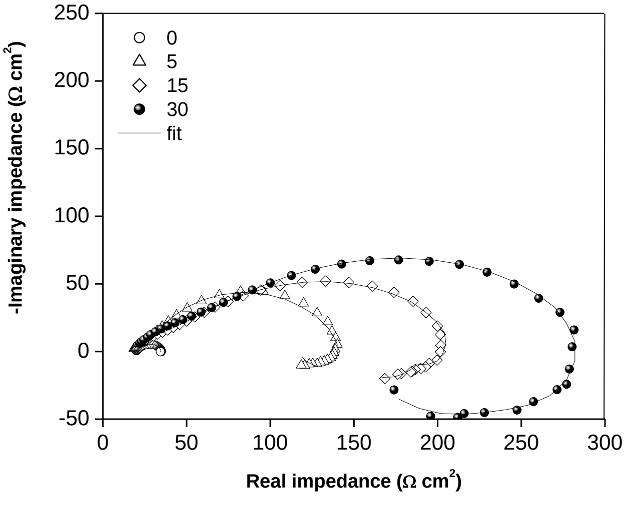

Figure 3. Typical Nyquist diagrams obtained on 1018 CS immersed 3h in the acid sour medium (hydrochloric acid 0.05 M + 500 mgL-1 hydrogen sulphide at pH 2.0) varying CI dosage; rotating the electrode at 2000 rpm and 40°C.

Fig. 3, shows the complex plots recorded for the CS immersed in the most aggressive condition (pH 2.0) using different dosages of the CI (5, 15 and 30 mgL^{-1}); rotating the electrode at 2000 rpm. In this Figure is important to remark the increase in the impedance values for each concentration of CI. It is observed a modification in the electrochemical response; because is evident the apparent formation of two depressed semicircles in all the frequency range. The relaxation phenomena occurring through the interface could be related with the possible formation of corrosion films with different semiconductor properties on the steel surface [8,18]. It can be suggested the typical formation of iron oxides, as well as the contribution of an organic compound by the CI [19, 20]. It is important to note that extrapolating the semicircle for evaluating the polarization resistance, the values obtained are minor in comparison with those evaluated at pH 4.0 in absence of CI (Fig. 2); indicating a minor protection at this pH by using a CI. Furthermore, the inductive response at low frequencies could be related with dissolution of the steel; as well as an adsorption or desorption phenomena of the CI on the surface, because the inductive response increases whit CI concentration [19, 20].

3.4 CI concentration effect at pH 4.0

Fig. 4 display the typical Nyquist spectra obtained for the immersion of the CS in the sour medium at pH 4.0 in presence of 5, 15 and 30 mgL^{-1} of CI; rotating the electrode at 2000 rpm. It is outstanding the impedance values recorded for each CI concentration. The impedance spectra are one order of magnitude major in comparison with the same plot at pH 4.0 (in presence of MEA) and two orders of magnitude in comparison with the sour medium at pH 2.0 (in absence of MEA, Fig. 1).

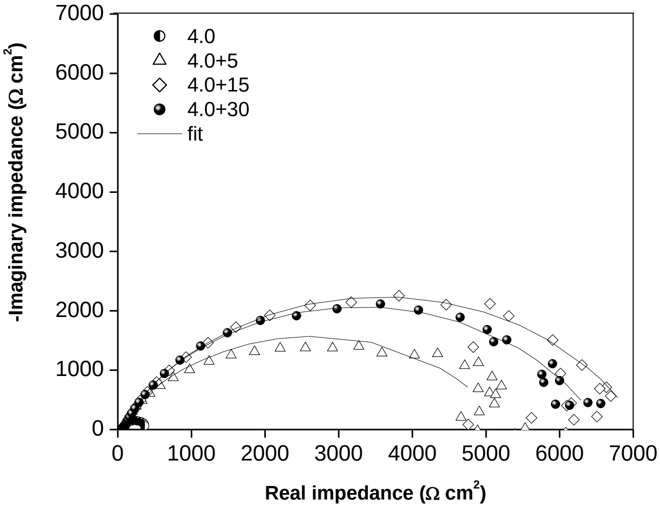

Figure 4. Typical Nyquist diagrams obtained on 1018 CS immersed 3h in the sour medium (hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide) varying CI dosage at pH 4.0, rotating the electrode at 2000 rpm and 40°C.

Also it can be observed that 15 mgL^{-1} of CI is an optimal concentration, reaching similar impedance values than 30 mg L^{-1}. It means that the protective behaviour by using a mixed treatment of MEA and CI is increased at pH 4; as was already mentioned, likely due to the formation of corrosion films with a different morphology and probably different chemical composition [2,19-20]. The chemical analysis of these films is out of the scope of this paper.

3.5 CI concentration effect at pH 6.5

In Fig. 5 are presented the typical EIS spectra recorded for the CS immersed in the sour solution at pH 6.5 in presence of 5, 15 and 30 mgL^{-1}; rotating the electrode at 2000 rpm. In the complex plots is observed an important modification in the electrochemical responses for each concentration; reaching higher impedance values as a function of the CI. It can be seen a capacitive response for 5 and 15 mgL^{-1} in comparison when the CI is added in 30 mgL^{-1}, recording a linear response at low frequencies. These results only could be related with the formation of corrosion products on the steel with a heterogeneous composition (MEA and CI), being adherent, less porous, among others; decreasing its oxidation in the sour solution [2].

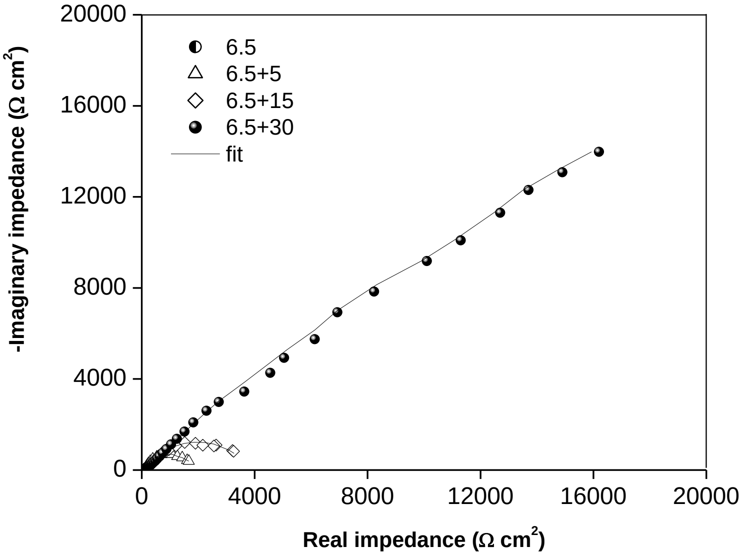

Figure 5. Typical Nyquist diagrams obtained on 1018 CS immersed 3h in the sour medium (hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide) varying CI dosage at pH 6.5, rotating the electrode at 2000 rpm and 40°C.

In order to further analyse the influence of the MEA and CI in the sour medium pH 6.5, in Fig. 6 is presented the Bode plots (phase angle vs frequency); which is sensible detecting the formation of corrosion products with different semiconductor properties on the steel surface [19-21]. It is noteworthy that similar responses were attained in presence of MEA and CI at pH 2.0 and 4.0. In Fig. 6 is observed a similar response for each curve increasing the angle values in presence of CI as a function of its concentration. Also is evident the formation of different maxima in all the frequency range being better defined in presence of CI. It is important to emphasize the increase in phase angle at high frequencies (10 kHz to 10 Hz) for the different CI concentrations (5, 15 and 30 mg L^{-1}); suggesting a modification in the semiconductor properties of the corrosion films formed on the steel [2,19,20]. This fact could be related whether both MEA and CI are forming a mix inorganic – organic corrosion films on the steel surface. This is supported because the electrochemical response in absence of CI (pH 6.5) only is observed an apparent resistive contribution which could be related with the predominantly formation of iron oxides and sulphides; still in presence of MEA [2,22-24].

In other hand, for intermediate to low frequency region the capacitive behaviour is detected and it could be related to the contribution of the electrical double layer or a diffusion phenomenon occurring through the corrosion films [2,21,22,25]. These differences assigning the contributions on the EIS measurements are dependent of the interface under study, i.e. in absence or presence of MEA, CI or both.

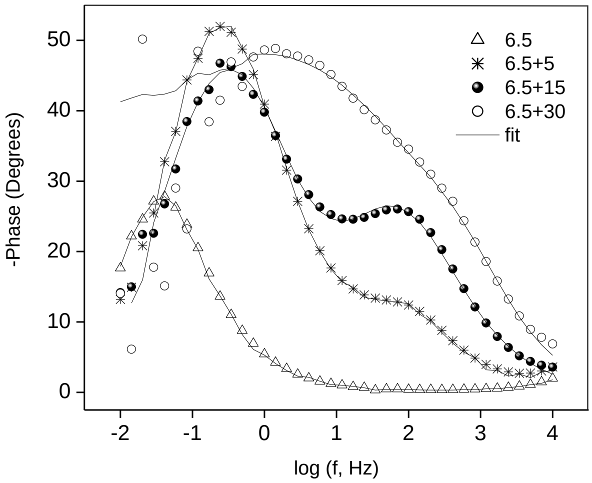

Figure 6. Typical Bode diagrams obtained on 1018 CS immersed 3h in the sour medium (hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide) varying CI dosage at pH 6.5, rotating the electrode at 2000 rpm and 40°C.

In general the steel protection in presence of MEA and CI seems to be acting in a different way to decrease the steel oxidation. As was mentioned above the MEA avoid the specific adsorption of the chloride ions [25,26]; meanwhile the CI modifies the semiconductor properties of the films and its protective properties [25].

3.6 Quantitative analysis

Based on the EIS analysis, to describe the oxidation of the steel in sour media, the equivalent circuit shown in Fig. 7 is suggested. For practical purposes, all experimental impedance spectra were related to the series-parallel equivalent circuit associated with two time constants and they were fitted using the Boukamp software [29]. This equivalent circuit considers the following elements: R_s is the solution resistance; R_{cp} and C_{cp} are the resistance and the pseudocapacitance associated with the corrosion products, respectively; R_2 and C_2 is an electric arrangement to describe a diffusion process through the corrosion films. The pseudocapacitance (C) values were evaluated using the following expression:

$$C = \frac{(YR)^{\frac{1}{n}}}{R} \qquad (1)$$

The terms Y and n were obtained using a constant phase element (Q) [30]. The continuous lines in Figs. 1 - 6, represent the fitting obtained for each experimental condition; being observed a reasonably adjust using the equivalent circuit shown in Fig. 7. Table 1 and 2 summarizes the electrical parameters derived from the best fitting of the experimental data.

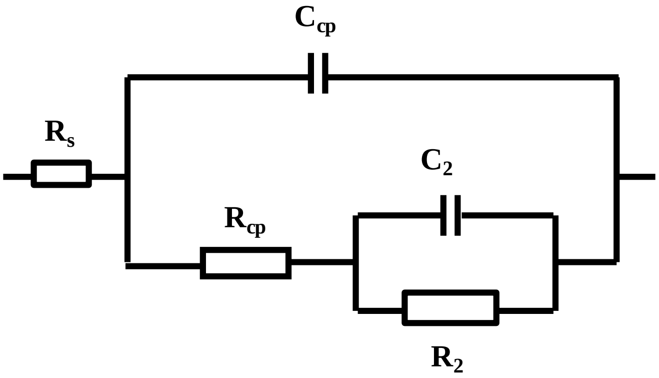

Figure 7. Equivalent circuit used to fit the experimental data shown in figs. 1 to 6.

Table 1. Parameter values obtained after the fitting using Boukamp program and the equivalent circuit shown in Figure 7 for the CS immersed in hydrochloride acid 0.05 M + 500 mgL^{-1} hydrogen sulphide + CI.

CI (mgL^{-1})	R_s (Ω cm^2)	R_{cp} (Ω cm^2)	C_{cp} (μF)	R_2 (Ω cm^2)	C_2 (μF)
0	19	4	108	11	240
5	18	23	19	103	77
15	17	49	6	149	64
30	17	78	7	201	79

In Table 1 the solution resistance varied from 19 to 17 Ω cm^2, for the different CI dosage at pH 2.0, indicating that the strength of the solution resistance is unchanged. In this Table is important to note the variation in the capacitance related with the semiconductor properties of the corrosion products formed on the steel in the sour medium (pH 2.0) in presence of CI. The maximum value is obtained in absence of CI (108 μF), mainly related with the formation of iron oxides; being negligible the contribution of sulphides because they could be dissolved at this pH [12, 31]. Typical values in this order have been reported in the formation of iron oxides or sulphides [2,23,24,32]. However in presence of CI, it seems that the semiconductor properties are different because this capacitance decrease; indicating the formation of a more resistive film probably mixed by iron oxides and/or organic compound.

Among the resistive elements (R_{cp} and R_2), is evident an increase as a function of the CI concentration. The first resistance is related with the corrosion films formed on the steel surface; meanwhile the second one could be associated with a diffusion process through the films, from the metal film interface; likely related with the dissolution of the steel. This fact is plausible whether is considered that the double layer capacitance should increase by considering a reduction of the non-covered areas of the steel by the CI, which is not observed in Table 1. Thus in presence of CI is considered that the electrical arrangement (C_2-R_2) is related with the diffusion of iron ions towards the aqueous solution. In order to support these assumptions, in Table 2 are presented the fitting values obtained for pH 4.0 and 6.5 in presence of different dosages of CI (5, 15 and 30 mgL^{-1}).

Table 2. Parameter values obtained after the fitting using Boukamp program and the equivalent circuit shown in Figure 7 for the CS immersed in hydrochloric acid 0.05 M + 500 mgL^{-1} hydrogen sulphide + MEA + CI.

pH + CI (mgL^{-1})	R_s (Ω cm^2)	R_{cp} (Ω cm^2)	C_{cp} (μF)	R_2 (Ω cm^2)	C_2 (μF)
4.0	51	66	3760	302	4150
4.0 + 5	61	2163	1.62	3635	11.81
4.0 + 15	68	6193	2.77	744	60.5
4.0 + 30	73	5865	3.06	552	76.4
6.5	50	21	28100	114	27200
6.5 + 5	60	67	74.0	1791	1280
6.5 + 15	63	235	28	3455	690
6.5 + 30	122	233	2.65	82481	363

In this Table is observed variations in the electric parameters for the different pH and CI concentrations. The solution resistance varied from 50 to 122 Ω cm^2. This variation could be related with the consumption of species probably due to the formation of iron compounds involving the inhibitor molecules onto the steel.

As explained earlier, the capacitance element evaluated for the steel in sour solution could be related with important modifications in the semiconductor properties of the corrosion films; by considering the formation of iron oxides (approximately 108 μF) at pH 2. Based on this assumption it can be suggested modifications in the dielectric properties of the films formed in presence of MEA at pH values of 4.0 and 6.5, because the capacitance values are 3760 and 28100 μF, respectively. These values could be related in one hand with an increase in the roughness and thickness of the films; meanwhile in other hand, it could be considered the presence of different iron compounds, i.e. iron oxides and sulphides or both; also reported in the literature [2,30-32]. Thus is probably that the adsorption of the MEA or the pH of the medium, modify the dissolution rate of the steel, as well as the rotation of the electrode, favouring the formation of corrosion films on the steel surface.

In other hand, when the corrosion films are formed varying the pH in presence of different CI concentrations is evident a decrease in the capacitance values; reaching C_{cp} values less than 5 μF; except some cases at pH 6.5 at 5 and 15 mgL^{-1}. This important variation in this parameter only could be explained with the mixed formation of organic compounds (amine and corrosion inhibitor) and iron oxides at the same time. The presence of this organic – inorganic layer is supported by an increase in the R_{cp} values for the different CI dosages reaching resistance values above 1000 Ω cm^2 for pH 4.0; meanwhile for pH 6.5 these values are in the order of 230 Ω cm^2. For pH 4, it can be suggested a modification in the chemical structure of the organic compounds (MEA and CI) interacting with formation of iron oxides and probably in minor extent iron sulphides; affecting their resistive properties. In the case of pH 6.5, it could be considered in minor extent the contribution of the MEA, resulting in the formation of an organic / inorganic structure similar to that obtained at pH 2.0, as well as the possible participation of iron sulphides; modifying its protective characteristics. This assumption is supported because is known the poor inhibition behaviour of the iron oxides on the steel for different pH values, reporting a high dissolution rate in acid conditions; being more protective at neutral pH [8, 12, 31]. It is suggested the formation of an organic – inorganic layer well defined in most part on the steel surface.

Likewise, the action of the corrosion inhibitor is supported by the rise in the diffusion resistance (R_2) for the pH 4.0 and 6.5 and larger concentrations of CI. It is remarkable that for pH 6.5 is observed the highest values, thus disclosing a substantial decrease in the oxidation of the carbon steel. From the above analysis it can be inferred that the optimum protection conditions come about at pH 6.5. It is worth mentioning that the corrosion control in the primary distillation columns is achieved by keeping the pH of streams within the interval of 5.5 to 6.5.

3.7 Characterization by SEM

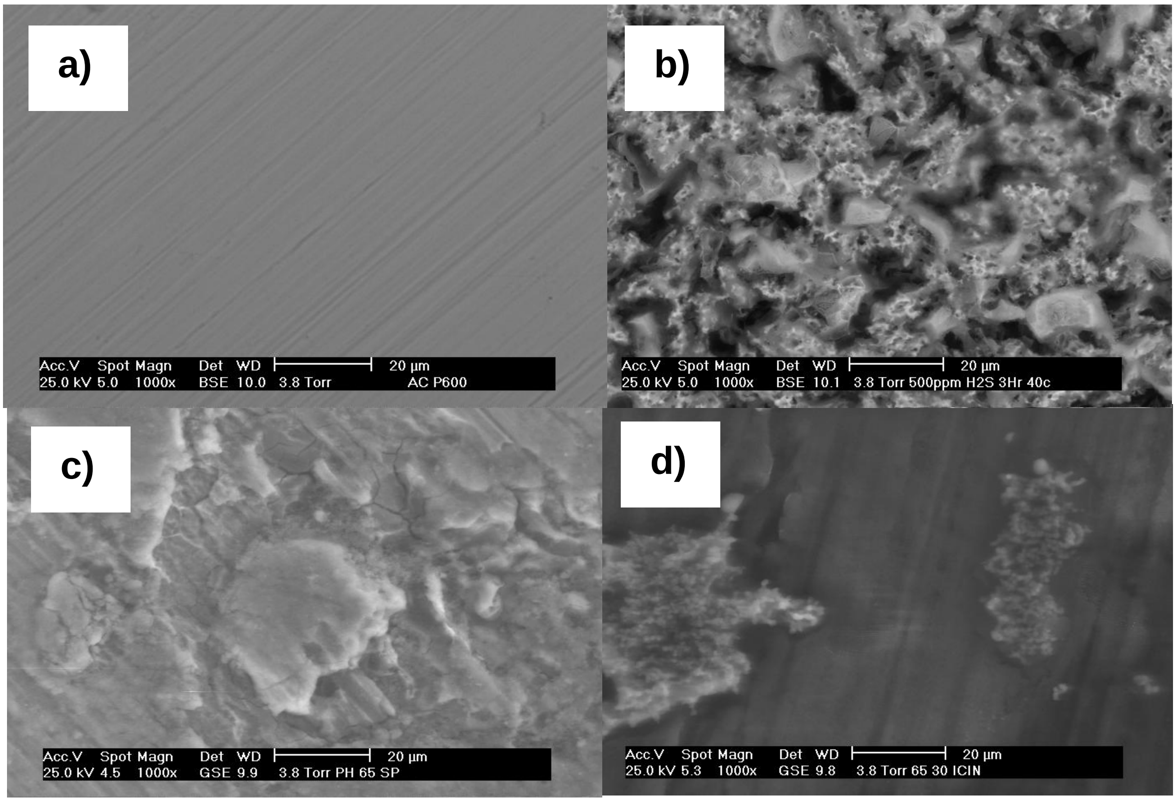

Figure 8. SEM images of CS surfaces exposed to: a) CS surface; b) HCl 0.05 M + 500 mgL^{-1} hydrogen sulphide, pH 2.0; c) pH = 6.5; and d) pH = 6.5 +30 mgL^{-1} of CI.

In order to support the electrochemical analyses of the metal-solution-interfaces, after the electrochemical tests, the carbon steel electrodes were further characterized by scanning electron microscopy (SEM). The examination was performed on surfaces where corrosion products were formed to determine the extent of deterioration and to correlate the SEM findings with the EIS analyses of carbon steel.

Fig. 8 shows, for instance, the SEM images of the freshly polished steel surface, 8a; and different surface conditions obtained after 3 h of immersion of carbon steel electrode in acid sour solution at pH 2.0, Fig. 8b; pH adjusted to 6.5, Fig. 8c; and the same solution at pH of 6.5 with 30 mgL^{-1} of CI, Fig. 8d. In this Figure is important to emphasize the different morphology of the corrosion films formed on the steel (Figs. 8b, 8c and 8d); in comparison with the scratches lines observed on the polished surface (Fig. 8a). It is evident the accelerated oxidation of the steel in the sour medium at pH 2.0; being evident a severe damage on the entire surface, indicating an active oxidation of the steel.

Conversely, greater modifications are obtained in the SEM images in the sour medium at pH 6.5 (Figs. 8c and 8d) by the presence of MEA, CI or both. In presence of the MEA (Fig. 8c), it seems the precipitation of different chemical compounds on the steel surface; showing its entire coverage on the steel. This fact allows supporting the minor contribution of the non-covered areas on the steel by the corrosion films. According to these results could be suggested its minimal contribution on the EIS measurements; supporting the diffusion of iron ions through the films. The coverage extent in the formation of corrosion films on the surface is supported, because is not evident the scratches polished lines observed in Fig. 8a.

In other hand, when the carbon steel is exposed to the sour acid solution at pH 6.5 + 30 mgL^{-1} CI (Fig. 8d), exhibits in one hand the formation of precipitates in some regions on the steel surface; meanwhile, in other hand non covered areas, showing the formation of a homogeneous surface. In this case is suggested the possible oxidation of the steel favouring the formation of a homogeneous film on the substrate; because the characteristic lines obtained by the polished treatment, seems to be different to those shown in Fig. 8a. This fact could be related with the formation of protective corrosion films on the steel, likely due to the presence of the iron oxides and sulphides and the CI. These results are in agreement with the analysis of the EIS diagrams using the equivalent circuit to describe the characterization of the steel in the sour media. Finally, it can be asserted that these chemical treatments are two plausible candidates to implement a more suitable corrosion control in the primary distillation columns.

4. CONCLUSIONS

The electrochemical characterization results show the complexity of carbon steel phenomenon corrosion typical of the petroleum industry. In order to reduce the corrosion damage in the atmospheric distillation towers is important to consider the pH control. It was evident that small pH variations (using MEA) enhance the activity of the metal surface; however better inhibition behaviour is observed when a corrosion inhibitor is added which is associated with a synergistic effect between amine and CI. The mixture of amine and inhibitor requires an important study as reported here, in order to establish the pH value, which the major corrosion protection is obtained. Under evaluation conditions here used, the best corrosion control was attained at pH 6.5 by MEA and 30 mgL^{-1} of CI.

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