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Corrosion of Carbon Steel in Sour Water From the Oil Industry: the Effect of Temperature

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In this work, the corrosion behavior of carbon steel AISI 1018 immersed in a sour water sample solution collected from a refinery plant, was studied, varying the temperature between 25 to 50°C with and without stirring. Polarization curves indicate negligible modification of anodic branch without stirring; but, the cathodic branch is more sensitive to temperature change. Also, analysis of surface samples by scanning electron microscopy (SEM) shows no film of corrosion products. The values of electrochemical noise localization index (LI), indicate that the sites of localized corrosion increase at 30°C. However, when solution is stirred, polarization curves indicate more sensibility in anodic branch and SEM micrographs show different corrosion products. LI shows values approaching 1, indicating sites where localized corrosion is present. These results indicate that the variation in temperature can cause changes in the structure of the corrosion products and thus the severity of damage in the metal surface.

Keywords: Corrosion, sour water, carbon steel, temperature

1. INTRODUCTION

Among the corrosion problems, one of the most important present in the refining oil industry, is specifically the related to the sour water generated in primary distillation plants. Despite the efforts made in the industrial field, and in fundamental studies; the different operating conditions as well as different concentrations of pollutants, makes their control very complicated.

The studies that have been trying to simulate the operating conditions in sour waters [1-20] have increased in the last ten years, in which the characteristics of the raw obtained, have changed resulting in corrosive environments much more aggressive than before. The purpose of these studies was to try to find ways, in which various agents affect or minimize corrosion, with the purpose of determining the predominant mechanism of corrosion in such environments.

On the other hand, carbon steel AISI 1018 is widely used as construction material in many industries due to its excellent mechanical properties and low cost [21]. So, the evaluation of carbon steel corrosion in H_2S environments is very important in the petroleum industry, as this phenomenon is responsible for costly social, economic and sometimes even human losses [20].

The sour water is considered as waste water containing sulfides and chlorides, among other components; this kind of water is very harmful to metallic equipments that are in contact with it, producing high corrosion rates leading to failures and material drainage and spilling [22].

An important factor involved in the process of corrosion, is the temperature, because the corrosion rate increases as the temperature increase in the corrosion medium on the reactions proceeding in pure acids [23]. However, this does not happen in all systems, for example, the steel AISI 304 SS [24]; results indicated higher corrosion at high temperatures (more than 75 ºC), but at mid temperatures (between 50 and 75 ºC) corrosion rates were similar; and at low or moderate temperatures (between 25 and 50 ºC), the stainless steels are in true passive state below the transpasive region. From early studies in the same work on the corrosion behaviour of AISI 304 using anodic polarization method, it was observed, a smaller passive range at higher temperature and hence an unstable passive film.

Finally, failures due to wet H_2S cracking have been experienced extensively in production operations, in refining and in pipelines. Many commonly used steels and alloys are susceptible to cracking under the prevailing high temperatures, pressures and stresses [2]. On the other hand, many places where the refining industries are located have drastic changes in atmospheric temperature conditions and the damages in the equipment are severe.

The present study focuses in determining the corrosion effect on the carbon steel at different temperatures when it is in the real corrosive medium. Here, the behaviour of carbon steel AISI 1018 at temperatures between 25 and 50º C, is studied in the presence of a sour water sample from a primary distillation plant of a Mexican Refinery. The studies were performed with and without stirring using a rotating disk electrode to observe the flow effect, and trying to simulate operating conditions.

2. EXPERIMENTAL PART

Before collecting the samples of a sour water sample from a primary distillation plant of a Mexican Refinery, a statistical study (not showed in this work) was performed where all the parameters that change over a period of time, were identified. Afterwards, samples were collected from a primary distillation plant of a Mexican Refinery using a sampling point of the dome in the distillation tower. The composition ranges of the samples are showed in Table 1. Other studies are being performed varying parameters like pH and chloride content, but they will be published later.

For the electrochemical study a typical three electrode cell setup was used with saturated calomel electrode (S. C. E.) as reference and a graphite bar, as counter electrode. The working electrode was AISI 1018 carbon steel disks with 0.5 or 0.7 cm^2 of cross section area, embedded in a

Teflon support. Prior to each experiment, electrode surface was polished with 600 SiC emery paper. These electrodes were immersed in a sour water sample from the oil industry under 25, 30, 35, 40, 45 and 50 °C with and without stirring. The solution was deaerated with high purity nitrogen for 30 min prior testing. When stirring, was used a 500 rpm rotating disk electrode during the tests. Nitrogen was passed throughout the test, assuring de-oxygenating conditions as in the real operating conditions.

Table 1. Composition ranges for sour water samples from a Mexican primary plant refinery.

Component	mg/l
Al^{3+}	0.01 - 0.11
Ba^{+2}	0.02 - 0.12
Fe	2.41 - 2.87
Mn^{+2}	1.87 - 2.01
Na^{+}	19.23 - 22.31
Sulfide content as SO_4^-	62.31 - 64.32
NH_4^+	2.78 - 3.47
Ca^{+2}	6.32 - 8.56
Mg^{+2}	2.96 - 3.46
K^{+}	4.36 - 6.88
Sr^{+2}	0.11 - 0.12
Cl^{-}	110 – 240
Cr^{3+}	0.03 - 0.22
pH range	6.63 - 7.33

Polarization curves were traced at a 0.1 mV/s scan rate, in a range of E_{corr} ± 300 mV, following a 10 minutes immersion of the electrode in the corrosive solution, time in which the corrosion potential reached stability. Carbon steel AISI 1018 samples without exposure to corrosive medium, as well as samples for each experiment polarized anodic and cathodically, were analyzed in the Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometer (EDS).

Electrochemical Noise measurements were performed using simple cells which consisted of two “identical” samples of AISI 1018 carbon steel and a SCE reference electrode, immersed in sour water at the same temperature. Without stirring the samples have 0.5 cm^2 area. When stirring, the samples have 0.7 cm^2 area and two “identical” rotating disk electrodes at 500 rpm speed were used. The solution was deaerated with high purity nitrogen for 30 min. 1024 measurements with a sampling rate of 1 measurement per second was obtained. An ACM Gill AC and software for tests without stirring, and with stirring conditions an Autolab PGSTAT 30 potentiostat—galvanostat and software were employed. This was so, because the experiments were performed at two different laboratories, previously observing reproducibility.

3. RESULTS AND DISCUSSIONS

3.1 Temperature Without Stirring

In order to compare different temperatures, Fig. 1 shows polarization curves. At temperatures of 25 to 40°C, anodic branches are very similar, but when the temperature increases at 45 and 50°C, the current increased. Fig. 2 shows SEM images of surfaces at different temperatures of the anodic scanned samples; it can be clearly seen the absence of corrosion products.

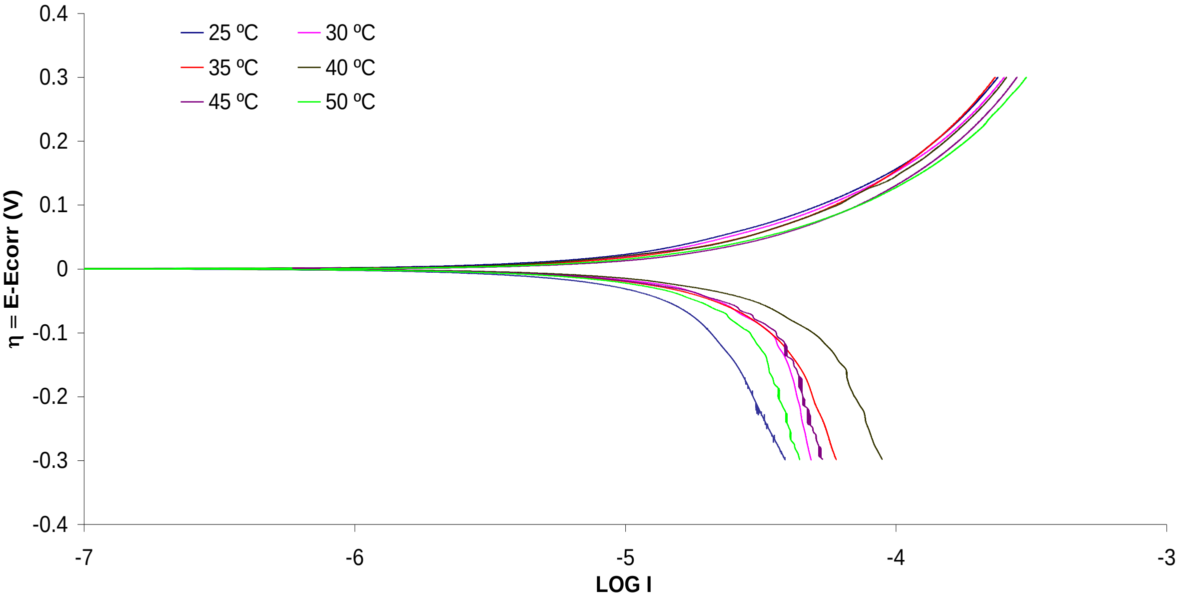
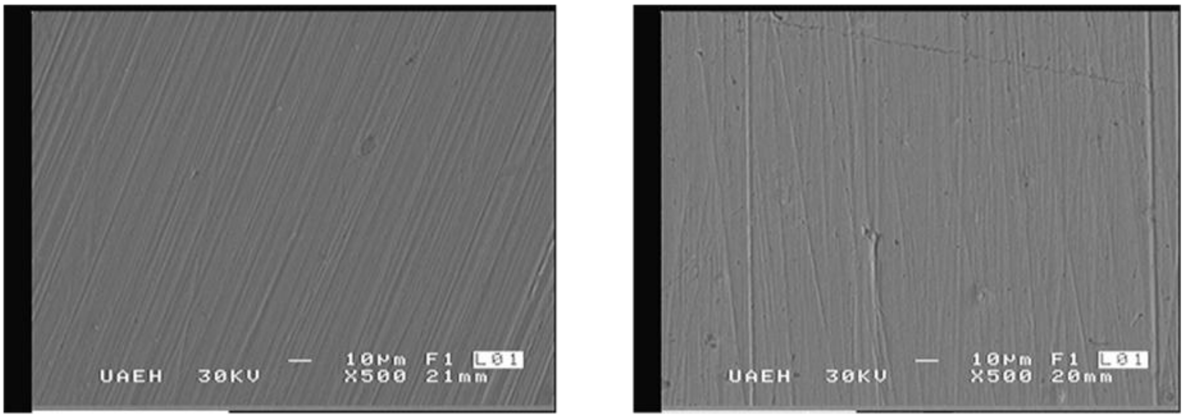
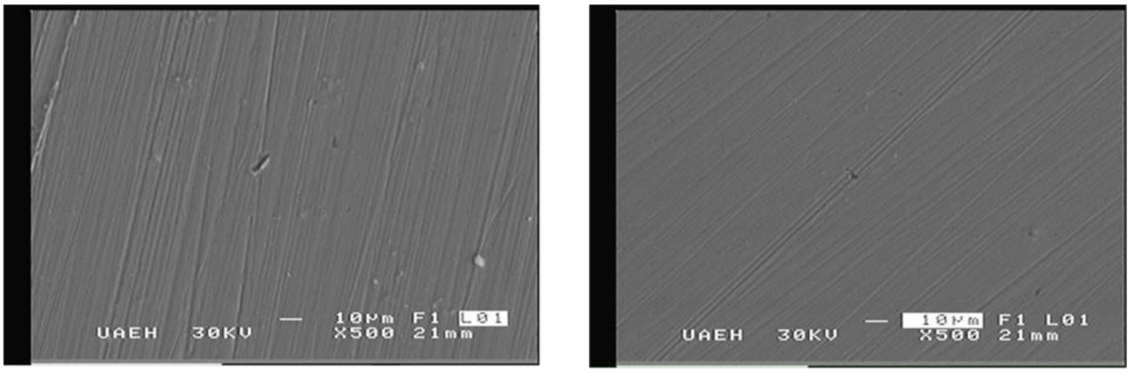


Figure 1. Polarization curves obtained from AISI 1018 carbon steel in sour water at different temperatures.



(a) (b)

Figure 2. Surface resulting from the exposure of carbon steel AISI 1018 at temperature 40°C (a) and 50°C (b), on the electrode with anodic scan.



(a) (b)

Figure 3. Surface resulting from the exposure of carbon steel AISI 1018 at temperature 30°C (a) and 35°C (b), on the electrode with cathodic scan.

On the other hand, the cathodic branch is more sensitive to temperature change as it is observed in Fig.1, at 25 to 40ºC, the reduction currents increased when temperature increased; while at high temperatures, the current decreases and the slope increases in comparison with that at 35°C. However, as in the anodic scan, the images of SEM in the cathodic scan (Fig. 3) showed clean surfaces, as expected. Also the results of EDS at different temperatures did not show oxygen, which would indicate the absence of corrosion products (oxides or hydroxides).

Table 2 shows the corrosion potentials (E_{corr}), polarization resistances (R_p), Tafel slopes (b_a and b_c; anodic and cathodic, respectively) and corrosion currents (I_{corr}) obtained from the polarization curves at different temperatures; these calculations were made manually taking as a reference the junction at the corrosion potential. The values of the corrosion potential are more negative than in 25°C, indicating that the reduction reaction is favoured on the metal surface, with the increase of temperature. The cathodic slopes have higher values than the anodic ones, indicating the presence of diffusive mass transport processes, possibly related to hydrogen evolution. On the other hand, the polarization resistances show higher values compared to systems in sour acid mediums reported in the literature [25], indicating less active surfaces.

Table 2. Corrosion parameters obtained from polarization curves, for AISI 1018 carbon steel in sour water at different temperatures.

Temperature °C	E_{corr} (V vs. ESC)	I_{corr} (A/cm^2)	Rp (Ωcm^2)	b_a (V/dec)	b_c (V/dec)
25	-0.6796	5.49E-06	2056	0.2174	-0.8456
30	-0.6679	8.99E-06	1256	0.2957	-1.0647
35	-0.6905	7.92E-06	1426	0.2728	-0.6306
40	-0.6686	1.03E-05	1095	0.3232	-0.6581
45	-0.6961	8.32E-06	1357	0.2734	-1.2438
50	-0.6985	7.25E-06	1557	0.2289	-1.1614

The corrosion currents, calculated from the R_p values (as it is done sometimes in industrial corrosion monitoring), show low variation among them, as the increase in temperature had no effect. However, as mentioned before, the temperature effect is on the cathodic reaction, which is reflected in the cathodic slope values. This could be an indication of slight damage related with the hydrogen evolution reaction.

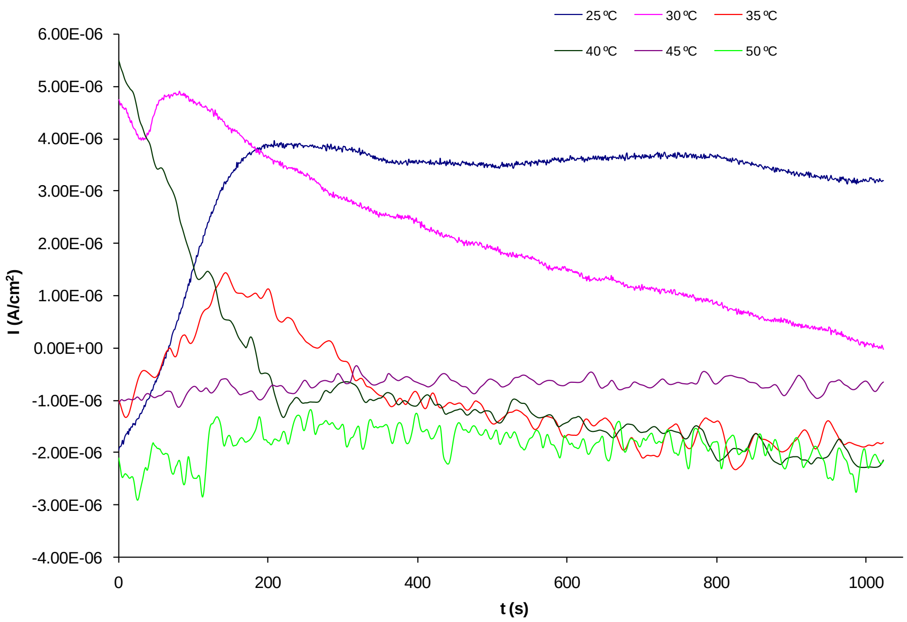


Figure 4. Current time records for AISI 1018 carbon steel in sour water at different temperatures.

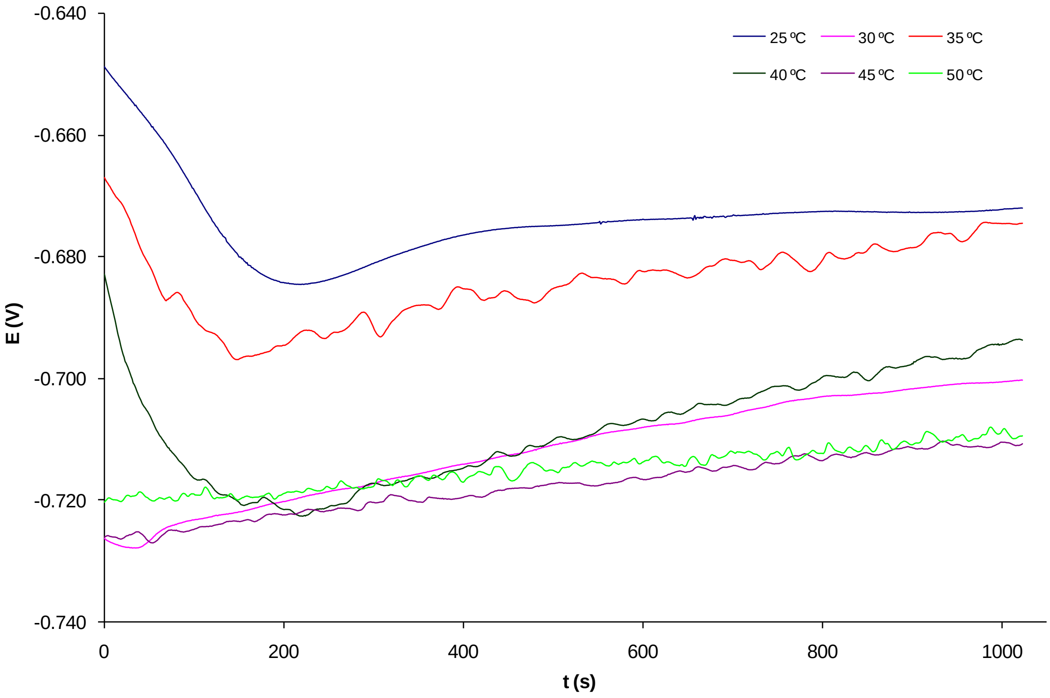


Figure 5. Potential time records for AISI 1018 carbon steel in sour water at different temperatures.

The experimental results obtained for carbon steel AISI 1018 in sour water conditions at different temperatures without stirring, with sequential collection of potential and current noise data are displayed in Figs. 4 and 5. Figure 4 shows experimental current noise data. For the 35°C temperature condition and above, after transient behaviour dies out reaching steady state conditions, some higher frequency and amplitude oscillations or higher noise level were observed, and therefore more sites of localized corrosion. Although at 25 and 30 ºC, measurements suggest generalized corrosion conditions, with no visible oscillations.

Figure 5 shows a slow fall in the potential at temperatures of 25, 35 and 40ºC, towards cathodic potentials, associated with the active region; and then reaching the stationary state. At 30ºC temperature, it increases toward anodic potentials. However, at 35, 45 and 50°C (Figs. 4 and 5), data show steadily increase in current, which then falls rapidly, with little detectable current and potential decay. As with noise current (Fig.4), some higher frequency and amplitude oscillations or higher noise level were observed. Then the potential consists of the response of the passive surface (that must provide the cathodic current) to the anodic current produced by pits; although these periods are much less predictable in both amplitude and in time of occurrence than for carbon steel with 0.025M NaCl [26].

Table 3. Corrosion parameters obtained from electrochemical noise, for AISI 1018 carbon steel submerged in sour water at different temperatures.

Temperature ºC	σ of I (A)	σ of P (V)	Rn (Ωcm²)	LI
25	8.34E-08	7.54E-04	6442	0.0475
30	2.63E-07	3.15E-03	8527	0.4824
35	2.63E-07	2.95E-03	8005	0.1550
40	3.45E-07	4.73E-03	9762	0.1964
45	9.89E-08	2.27E-03	16345	0.1439
50	2.31E-07	1.75E-03	5412	0.1199

The noise resistance as a function of time is presented in Fig. 6, with average noise resistance in the 100-1000 ohm-cm^2 with some transients up to 10 000 ohm-cm^2 observed, for the 30-40°C temperature conditions. A local noise in the range 500-1024 seconds, were obtained with the results of electrochemical noise parameters, presented in Table 3. The standard deviation of the current and potential, the noise resistance (Rn) which is the standard deviation of the potential between the standard deviation of the current ($Rn = \sigma_v / \sigma_i$), and the localization index (IL), obtained from the ratio of the current standard deviation over the root mean square (rms) [27]:

$$IL = \sigma_i / i_{rms} \tag{1}$$

$$rms = \sqrt{\frac{1}{n}\sum_{t=1}^{n}(x_i^2)} \tag{2}$$

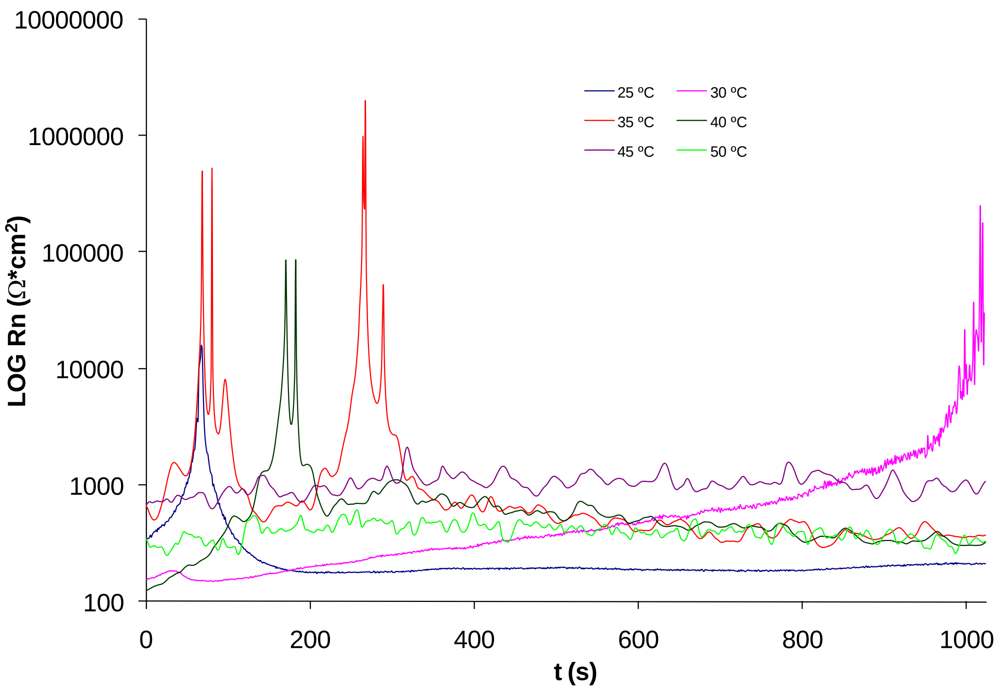


Figure 6. Resistance time records for AISI 1018 carbon steel in sour water at different temperatures.

Table 3 shows that LI increased at 30ºC, which is the highest value of IL obtained, this suggests localized events taking place over the surface. But when temperature is increasing, IL decreased indicating that the sites of localized corrosion would be lower. It must be due to the solubility of the corrosion products. Although, LI values suggest some localized attack, their values close to 0.1 approach the mixed corrosion attack condition (LI 0.01-0.1), more likely since no corrosion products were observed (Table 3). Also, the standard deviation of the current is greater for temperatures of 30, 35, 40 and 50°C, suggesting that the corrosion rate is higher than with other temperatures with less standard deviation of the current.

The different Rn did not show a trend in their behaviour, obtaining the greatest resistance at 45ºC and the less at 50°C. Also at temperatures between 30 and 40ºC, there is an intermediate effect on the corrosion resistance, which could indicate that the effect of temperature allows the formation and re-dissolution of corrosion products as a result of the cathodic reaction, reflected in the overall corrosion rate. This has been reported before in the literature [20, 26].

3.2 Effect of Temperature under Stirring

In order to compare different temperature effects when the solution was stirred, Fig. 7 shows polarization curves. At temperatures of 25 to 40°C, anodic branches are very similar and these processes are also observed without stirring; however, when the temperature increases at 45°C, the currents increased, but at 50°C anodic currents decreased again considerably, registering the lowest values.

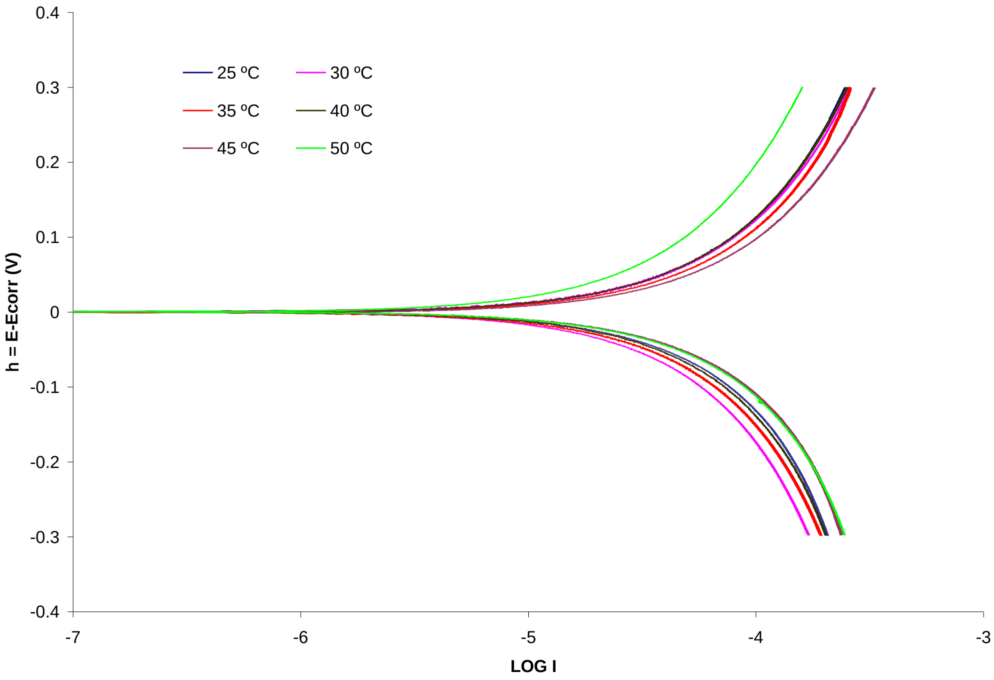


Figure 7. Polarization curves obtained from AISI 1018 carbon steel in sour water at different temperatures with stirring.

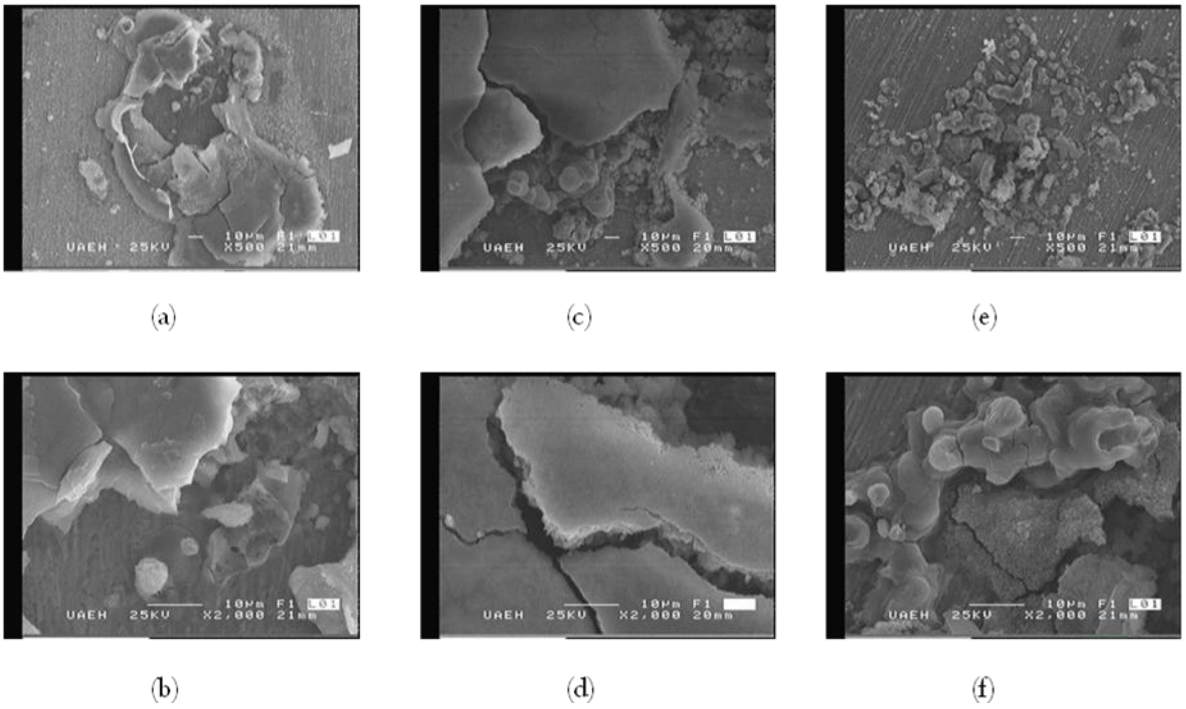
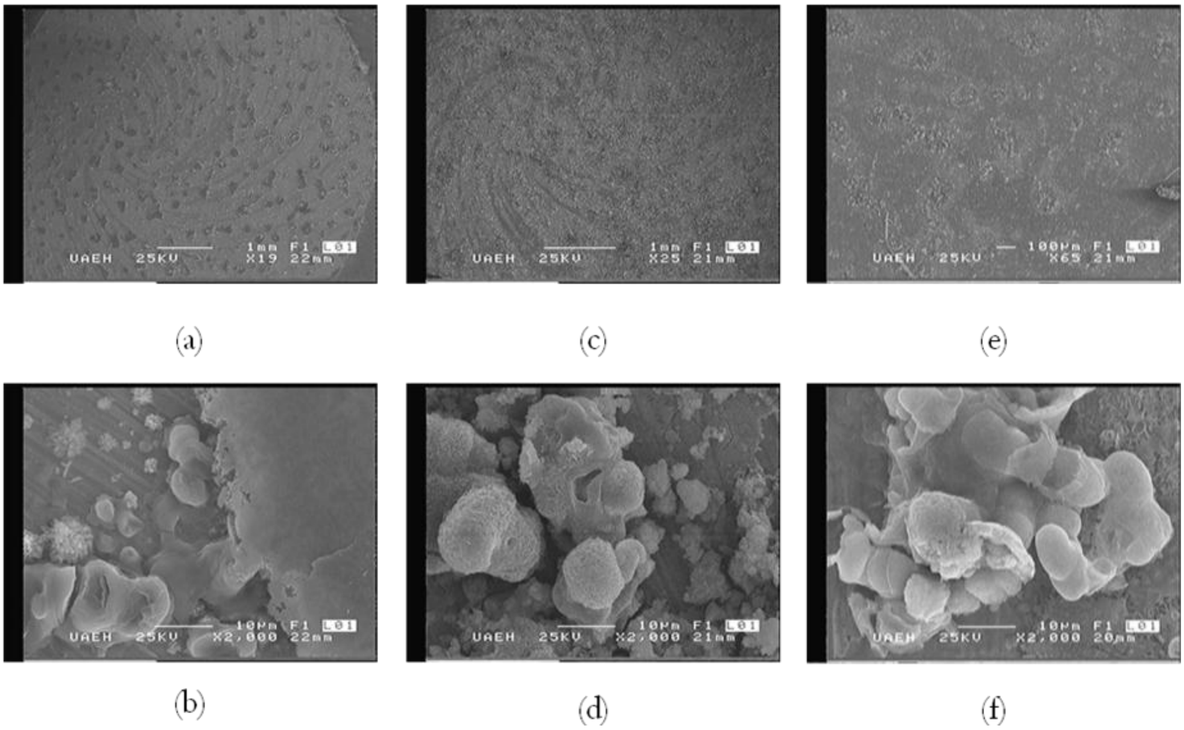


Figure 8. Surface resulting from the exposure of carbon steel AISI 1018 at temperature 25°C (a) , (b), 30°C (c), (d) and 50°C (e), (f), on the electrode with anodic scan.

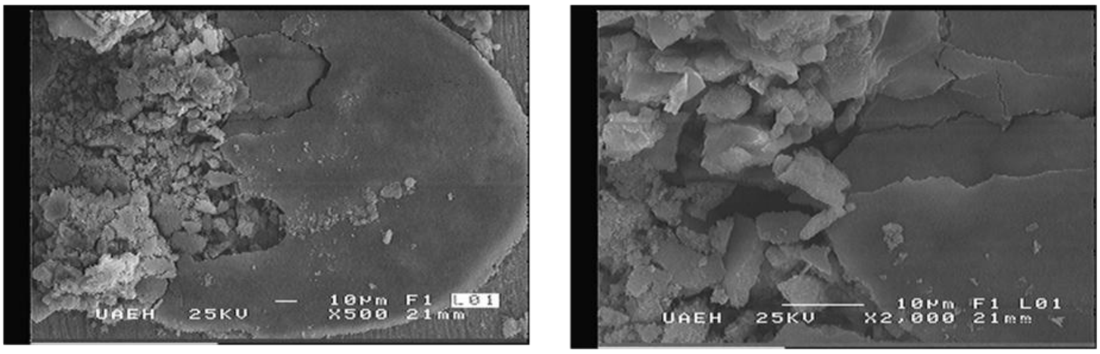
SEM images of anodic scanned samples (Fig. 8) shows a compact layer with small areas of cracked film and formation of new corrosion products, which may be responsible for the similarity of the anodic currents at 25 to 40°C. At 50ºC the image shows layers of porous corrosion products, which probably are not electrically conductive and would lead to the anodic oxidation currents being lower than for the rest of the other temperatures.



(a) (c) (e)

(b) (d) (f)

Figure 9. Surface resulting from the exposure of carbon steel AISI 1018 at temperature 25°C (a), (b), 35°C (c), (d) and 45°C (e), (f), on the electrodes with cathodic scan.



(a) (b)

Figure 10. Surface resulting from the exposure of carbon steel AISI 1018 at temperature 30°C (a) and (b), on the electrodes with cathodic scan.

On the other hand, the system shows that cathodic branch samples (Fig. 9), are not very sensitive to temperature change between 25 and 40ºC and both 45 and 50 °C, because the reduction currents are very similar. Although, the currents of the latter are higher than the rest of the temperatures; at 30ºC the lowest reduction currents were obtained. On the contrary, when the system is not stirred, the cathodic branch is more sensitive to temperature change. This is indicative that the medium evolution is taking place in the system. SEM images of the electrode with cathodic scanned samples (Fig. 9) show the formation of protective corrosion products. However at 45 and 50°C, the surface of the electrodes have less corrosion products, but at 30°C (Fig. 10) shows a porous corrosion layer, which may be responsible for the lowest reduction current.

Also, the results of EDS on the surfaces of the electrodes tested at different temperatures with stirring, show the presence of oxygen compounds, which indicates, that the only corrosion products formed are oxides or hydroxides with different stoichiometry, because they show differences in morphology.

Table 4 shows the corrosion potentials (E_{corr}), polarization resistances (R_p), Tafel slopes (b_a and b_c; anodic and cathodic, respectively) and corrosion currents (I_{corr}) obtained from the polarization curves at different temperatures. The values of the corrosion potential show that any reaction is favoured when the temperature increases; exception made at 45ºC, which is 60 mV more negative. This is observed in the anodic and cathodic slopes, because the values are similar between them, though distant from the typical Tafel values, indicating that the system is controlled by mass transfer. On the other hand, the polarization resistances show higher values with respect to the systems in sour acid media reported in the in literature [25], but similar to the system without stirring (Table 2); indicating the presence of slightly protective corrosion products or porous films. Also, the system at 50°C, has the greatest polarization resistance, although at 45°C, the lowest Rp was registered, which may be related to the morphology of the corrosion products (see Figs. 8 and 9).

Table 4. Corrosion parameters obtained from polarization curves, for AISI 1018 carbon steel in sour water at different temperatures, with stirring.

Temperature °C	E_{corr} (V vs. ESC)	I_{corr} (A/cm^2)	Rp (Ωcm^2)	b_a (V/dec)	b_c (V/dec)
25	-0.5960	1.99E-05	873	0.4160	-0.4696
30	-0.5826	1.98E-05	879	0.3638	-0.5045
35	-0.6015	2.00E-05	871	0.4343	-0.5876
40	-0.5902	1.50E-05	1161	0.4037	-0.4516
45	-0.6850	2.13E-05	817	0.3994	-0.4829
50	-0.6213	1.29E-05	1345	0.5117	-0.3484

The values of corrosion currents, which are directly related to the corrosion rate, are in the same order of magnitude for all temperatures tested, indicating that apparently, the increase in temperature has no effect. It shows that the corrosion rate calculated with R_p values cannot be a reliable parameter, because the changes in both anodic and cathodic slopes and the morphology of the corrosion products is different, as mentioned above.

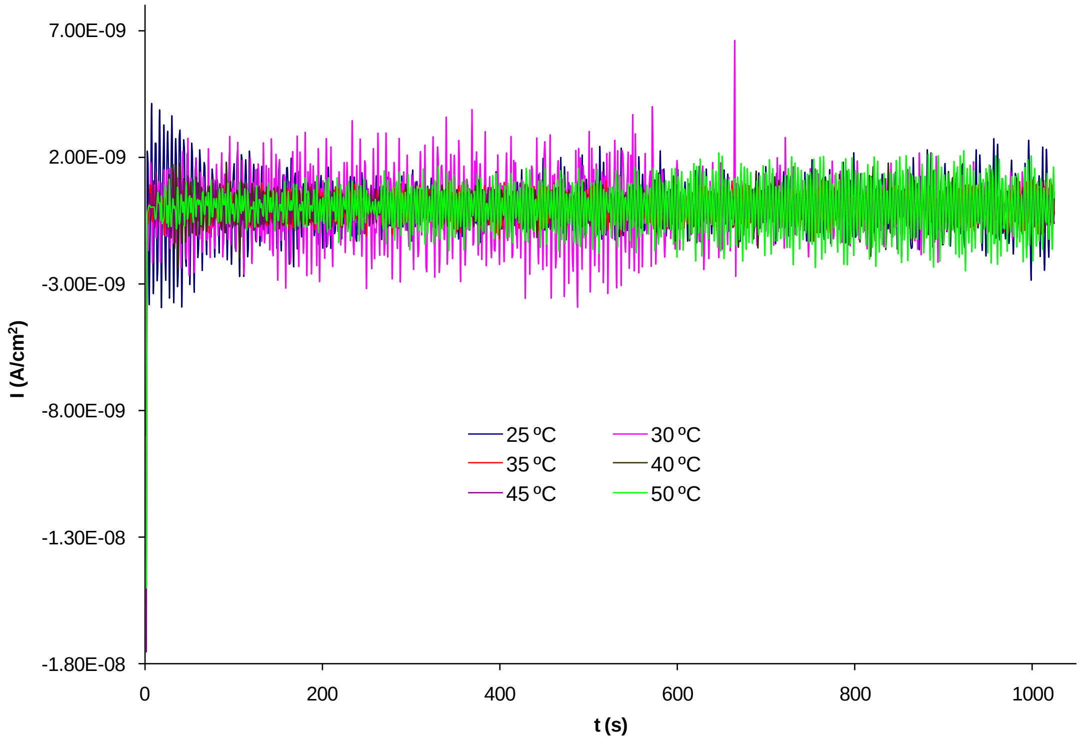


Figure 11. Current time records for AISI 1018 carbon steel in sour water at different temperatures with stirring.

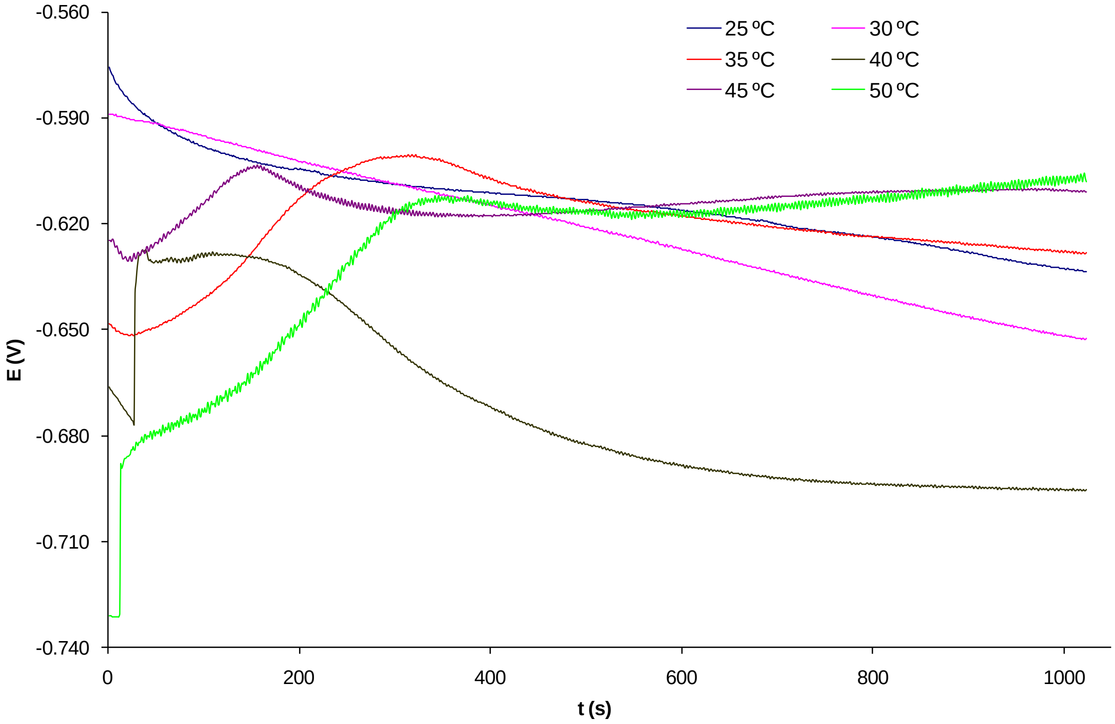


Figure 12. Potential time records for AISI 1018 carbon steel in sour water at different temperatures with stirring.

Figs. 11 and 12 show the sequential collection of potential and current noise data, showing current noise data (Fig.11) at 25 and 30ºC temperatures, presenting random behaviour or *white* noise, reaching these oscillations amplitudes around (-5X10^{-9} to 5X10^{-9} A/cm^2), but for another temperatures these oscillations are of lower amplitude (-2X10^{-9} to 2X10^{-9} A/cm^2). So, the first temperatures have increased levels of noise, but in systems without stirring the solution, there are smaller amplitudes registered. Thus stirring the solution, results in a greater increase in the noise amplitude. This is presumably as a result of enhanced mass transport, as in 304 SS [28].

Figure 12 shows some initial transient behaviour reaching stability. All measurements did not present oscillations, except for the initial transient. A slow fall in potential at 25, 30 and 40ºC, toward more cathodic potentials associated with the active region. However, at 35, 45 and 50ºC, the potential rose anodically, while at 40, 45 and 50°C reached the steady state. Also, these effects are present in systems without stirring (Fig. 5), but at different potential values, because when stirring the potential increase becomes more anodic.

A local analysis of noise data on the 500 to 1024 seconds range, were obtained with the results of electrochemical noise showed in Table 5. Table 5 shows that the LI is similar for all temperatures, and its value is close to 1.0, which suggests that the different samples at different temperature conditions, present localized attack. So, the standard deviation of the current is greater at 25, 30 and 50ºC, implying that the corrosion rate is higher than in other temperatures, which have lower standard deviation of the current.

Table 5. Corrosion parameters obtained from electrochemical noise, for AISI 1018 carbon steel submerged in sour water at different temperatures with stirring.

Temperature ºC	σ of I (A)	σ of P (V)	Rn (kΩcm²)	LI
25	6.44E-10	6.06E-03	6700	0.9996
30	7.53E-10	9.49E-03	8977	0.99997
35	3.98E-10	4.06E-03	7276	0.9978
40	4.59E-10	3.54E-03	5499	0.9991
45	2.85E-10	1.88E-03	4706	0.9982
50	8.16E-10	3.45E-03	3018	0.9992

As well as without stirring Rn, did not present any trend for the different temperature conditions because the greatest resistance is at 30ºC, and less at 50°C (Fig. 13). For other temperatures, there is an intermediate and variable effect on the corrosion resistance. These could indicate that the effect of temperature allows the formation and redissolution of corrosion products as a result of the cathodic reaction.

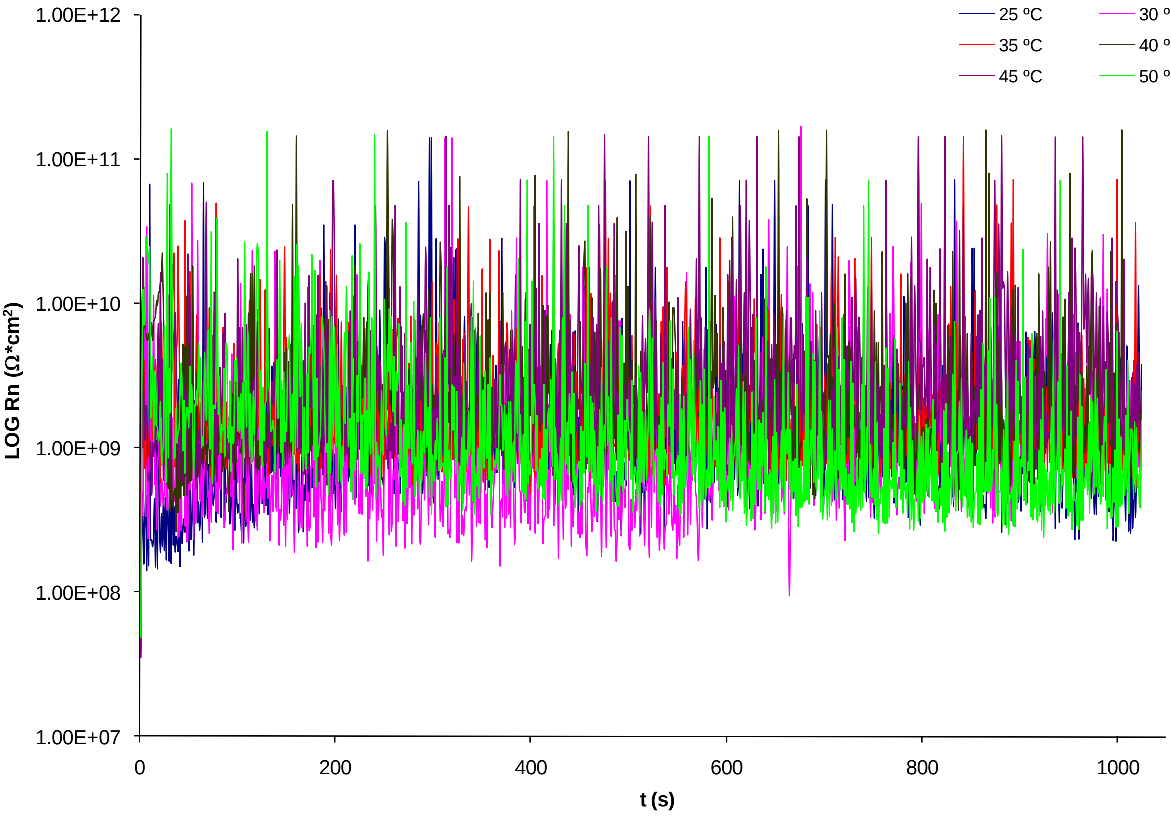


Figure 13. Resistance time records for AISI 1018 carbon steel in sour water at different temperatures with stirring.

4. CONCLUSIONS

The system under study at different temperatures without stirring did not show the presence of corrosion products and the temperatures where changes in behaviour were observed are at 30 and 45ºC.

On the other hand stirring the solution, shows results indicating that the variation in operating temperature can cause changes in the structure of the corrosion products and therefore the damage in the exposed material, even when the corrosion rate obtained through R_p did not show any differences. Also, the temperatures where changes in behaviour where observed were at 30 and 50ºC. This implies that equipment submitted to extreme temperature changes could be affected in an irreversible way, because of the corrosion products formed and the possibility of localized corrosion. For this reason it is very important the strict control of the operating temperature in all the pipelines and equipment where the sour water contents are present.

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