Use of the Faraday cage and the trend removal of the I_{DC} in measurements with electrochemical noise

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Abstract: The electrochemical noise tecnique (EN) can be employd for analize the electrochemical behavior of processes like passivation, corrosion, or inhibition of methals in contact with aggressive environments. The objective of this work was the evaluation with the electrochemical noise tecnique over electrode pair materials of mild steel (Ms-Ms) and other as galvanic pair of (Ms-Cu), under the influence and without it for the use of a Faraday cage. Also, methdologies were applied for fitting on the trade removal of the current I_{DC} generated with the EN measurements than in voltage as in current. The comparison of results was made with noise resistance (R_n) and the localized index (IL) as parameters. It was found that with the use of the Faraday cage and with the lineal method for the trend removal, better results were observed.
Keywords: Current DC, Faraday cage, Localized Index, Lineal and Polynomial Methods, Noise Resistance

Uso de la jaula de Faraday y eliminación de la tendencia I_{DC} en mediciones con ruido electroquímico

Resumen: La técnica de Ruido Electroquímico (RE) puede emplearse para analizar el comportamiento electroquímico de los procesos de pasivación, corrosión o inhibición de los materiales metálicos en contacto con un ambiente agresivo. El objetivo del presente trabajo fue evaluar con la técnica de ruido electroquímico en materiales de acero al carbono (Ac-Ac) y de un par galvánico (Ac-Cu) bajo la influencia y sin ella por el uso de una jaula de Faraday. Asimismo, se aplicaron metodologías de ajuste de tendencia por extrapolación lineal y polinomial para eliminar la tendencia de la corriente I_{DC} sobre las mediciones de RE tanto en voltaje como en corriente. Se compararon los resultados que se obtuvieron con los parámetros de resistencia de ruido (R_n) y el índice de corrosión localizada (IL). Se encontró que con el uso de la jaula de Faraday y la aplicación del método de ajuste lineal se observaron los mejores resultados en la remoción de la tendencia DC.
Palabras clave: Corriente DC, Índice de localización, Jaula de Faraday, Métodos, Lineal, Polinomial, Resistencia del ruido

Introduction

The electrochemical noise (EN) technique is used to study localized corrosion. When a metal corrodes at an open circuit potential (OCP), it experiences constant fluctuations of low frequency and magnitude which is called "*electrochemical noise*" in the corrosion current and potential; it occurs at the metal-electrolyte interface (Cottis R., 2008, Huet F., 2006 y Xia etal, 2020).

The pitting corrosion process is characterized by being insidious and difficult to detect. EN can be used to analyze the electrochemical behavior of current (I) and voltage (V) transients in passivation, corrosion and even inhibition processes (Mansfeld & Xiao, 1993, Zhang et al, 2007 and Klepper, et al 2007). Some of its main advantages compared

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to the conventional electrochemical techniques of Linear Polarization Resistance (LRP) and Electrochemical Impedance Spectroscopy (EIS) are the following:

- Ability to perform instantaneous monitoring of the speed of the corrosion process.
- DC technique that does not involve external disturbance of the corrosion process.
- With the use of statistical methods, it offers more information on localized corrosion and other mechanisms.

EN signals in voltage and current can be measured separately or simultaneously. During separate measurements, potential noise can be measured as the potential fluctuations between two nominally similar working electrodes. Current noise can be measured as the current fluctuation between two nominally similar working electrodes. Simultaneous measurement is preferred, in which the electrochemical potential and current noise are measured at the same time by coupling two nominally similar working electrodes connected by a zero resistance ammeter (ZRA) and measuring relative to a reference electrode.

Different electrode arrangements have been proposed, as shown in Fig. 1 (Sanchez et al, 2009 and Xia et al, 2020), where arrangements A and B have been used for monitoring individual voltage or current transients. However, Cottis (2001) recommends that a basic arrangement such as arrangement C should be used when simultaneous monitoring of V and I is required, because it considers the measurement of current (I) between two exactly equal working electrodes (WE_1 and WE_2) and the measurement of potential (V) between the working electrodes and the reference electrode (RE).
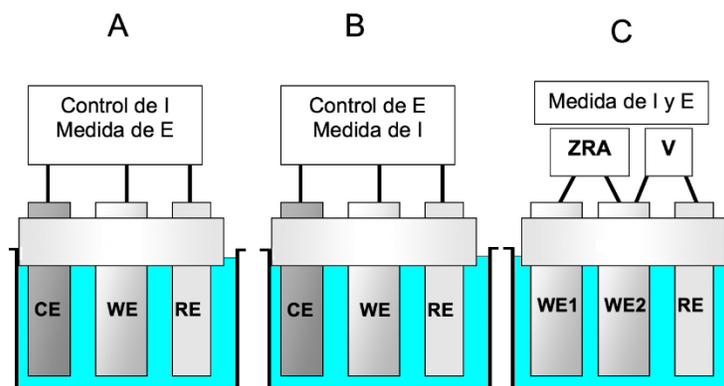

Figure 1. Electrochemical noise electrode cell arrangements.

In cases A and B, individual noise measurements are considered, where the current noise is measured between the working electrode and the counter electrode and the potential noise is measured between the working electrode and a zero noise reference electrode (Cottis, 2006). However, the simultaneous measurement of current and potential noise allows direct determination of the electrochemical noise resistance (R_n) (in the time domain), and the noise impedance, Z_n (in the frequency domain).

The electrochemical noise technique is highly sensitive and it can be used to study the kinetics of the corrosion process. However, there is the possibility that the electrochemical information can be interfered with by external factors that can alter the recorded signal (Malo and Uruchurtu, 2000). Given the high probability of interference, more sophisticated signal processing methods have been implemented, such as the removal of the DC trend, which represents the gradual variation in the continuous change of the voltage and current measurements recorded individually or simultaneous. The presence of the DC trend in an electrochemical signal can produce interpretation errors when conventional statistical analysis methods are applied, so its removal can be important for the analysis of electrochemical information in a noise signal (Amaya et al, 2009 and Lentka and Smulko, 2018). When EN measurements are performed on asymmetric electrode systems, the electrochemical response can be interfered with by a DC current signal (I_{dc}) introducing instability to the real electrochemical noise signal. Instruments are currently being fitted with filters that can remove I_{dc}. However, statistical treatments such as linear extrapolation (Homborg et al, 2012), polynomial and others (Lentka and Smulko, 2018) can also be carried out to find the best fit of the EN data for both I_E and V_E as is set forth below. For each measurement, in order to eliminate the DC trend, the current and potential information obtained is fitted to a straight line in the linear method (see Fig. 2a), or to a least squares polynomial when there is no linear trend (see Fig. 2b). In the linear method, a slope A and an intercept B are obtained according to the following equations.

$$I_d[n] = A_i T[n] + B_i \qquad (1)$$
$$E_d[n] = A_e T[n] + B_e \qquad (2)$$

where $I_d[n]$ is the corrected current of point n, A_i is the slope of the fitted DC trend line in current, $T[n]$ is the time of point n, B_i is the intersection of the fitted current line , $E_{dc}[n]$ is the corrected voltage of point n, A_e is the slope of the fitted DC trend line in potential, $T[n]$ is the time of point n, B_e is the intercept of the fitted line of potential.
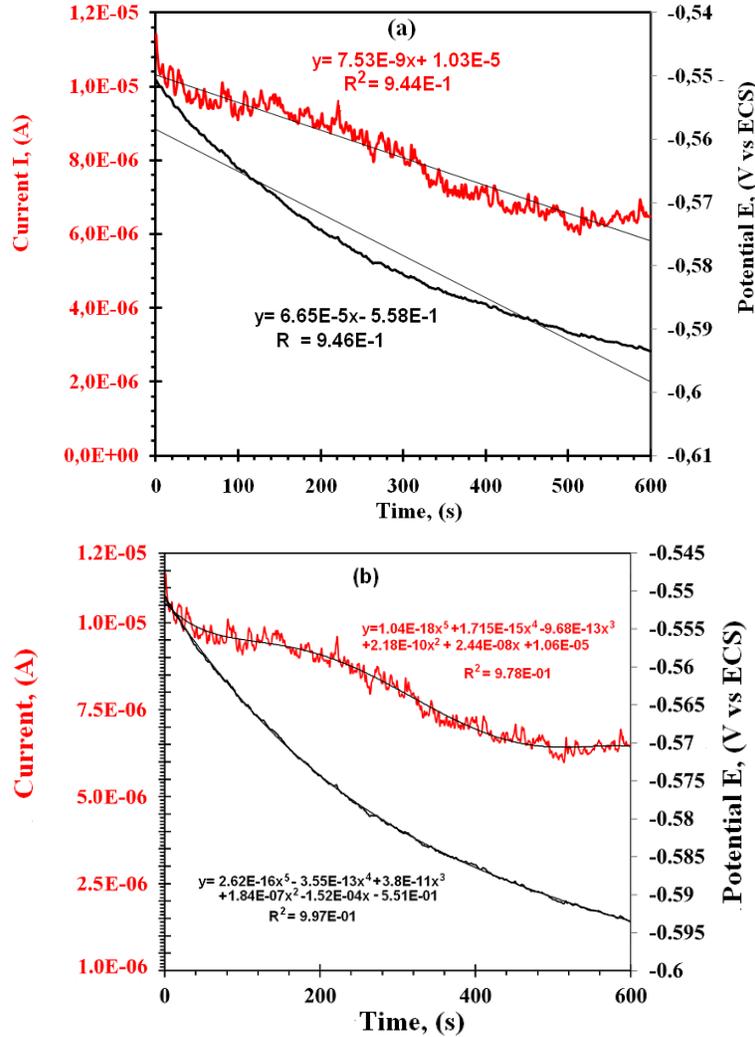
Figure 2. Examples of a) linear and b) polynomial fit of current and potential measurements

In the linear method, the current and potential noise are estimated according to the selected sampling period, subtracting the DC trend and calculating the root mean square RMS of the signal residue as follows

$$Irmsj = \sqrt{\sum_{i=n}^{N}(I[n] - Idc[n])} \qquad (3)$$

$$Ermsj = \sqrt{\sum_{i=n}^{N}(E[n] - Edc[n])} \qquad (4)$$

here $I_{rms}j$ is the current noise of block j, $E_{rms}j$ is the potential noise of block j, $I[n]$ is the measured current signal at time n, $E[n]$ is the measured potential signal at time n, and N is the total number of points. The estimate of the current and potential noise of each block is determined, the values of all these are plotted, and the mean of the noise and potential values is found as:

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$$Irms = \sum_{j=1}^{m} Irms \,[m] \tag{5}$$
$$Erms = \sum_{j=1}^{m} Erms \,[m] \tag{6}$$

where I_{rms} is the current noise of the sample and E_{rms} is the potential noise of the sample.

The polynomial method for an expression of order p_0, the approximate trend I_{rms} is adjusted at the measurement time and detrending the measurement data $x_n = [n]$, to determine the value of y_n, expressed in equation (7).

$$y_n = I_{dc} = \sum_{i=n}^{po} a_i * n^i \tag{7}$$

Now, considering the expression of a polynomial (8), for the current and voltage signals.

$$I_{rms,j= \min \sum_{n=1}^{po} (x_n - y_n)}^{\,2} \tag{8}$$

where a_i are the coefficients of the polynomial and that must be minimized to the least squares criterion with some software such as Excel or Matlab. The expressions of the adjusted least squares polynomial are shown in Figure 2(b).

The analysis of the measurements obtained by the EN technique can be performed by statistical treatment in the time and frequency domains (Mansfeld, 2005). In this work, the study was carried out in the time domain on the transient variations of the potential and current, the resistance of the electrochemical noise (R_n) was also analyzed through the standard deviation of the potential (σ_E) and current (σ_I) expressed in the equation (9) and finally the localization index (IL) was determined by means of equation (10), where the standard deviation of the current (σ_I) divided by the mean square root of the current $\sqrt{(I_{mean})^2}$, which also can be expressed as the square root of the current mean (I_{rms}) (Cottis and Turgoose, 1992, Botana et al 2002 and Lafront et al, 2010).

$$R_n = \frac{\sigma_E}{\sigma_I} \tag{9}$$

$$LI = \frac{\sigma_I}{\sigma_{rms}} = \frac{\sigma_I}{\sqrt{\sigma_I^2 + I^2}} \tag{10}$$

The objective of the present work was to work on electrochemical noise in mild steel-based materials (Ms-Ms) and to measure the influence of the use of a Faraday cage and without it, to evaluate the possible introduction of interferences on the sensitivity of the response of EN. Likewise, we worked with a galvanic couple of carbon steel and copper (Ms-Cu) with and without Faraday cage. The results will be evaluated using the parameters of electrochemical noise resistance (R_n) and the localization index (IL).

The corrosion rate measurement procedure was promoted by the Manchester group since the early 1980s (Eden et al, 1986), finding that R_n can be used to replace the linear polarization resistance (LPR) of the corroding electrodes and that it can be used to determine the corrosion rate, which is commonly expressed as a corrosion current density (i_{corr}). The IL with values ranging between 0 and 1 is a qualitative descriptor of corrosion phenomena (Eden et al., 1987). Table 1 details the IL intervals in a corrosion process.

Interval IL	Type of corrosion
$0.001 \leq IL < 0.01$	Uniform Corrosion
$0.01 \leq IL < 0.1$	Mixed Corrosion
$0.1 \leq IL < 1$	Pitting Corrosion

Table 1. Classification of the corrosion phenomenon according to the IL intervals.

Methodology

Preparation of the Working Electrodes

With silicon carbide sandpaper of 80, 120, 240, 400 and 600, the metallic surfaces of mild steel (low carbon) and copper were roughened until a uniform surface finish was achieved. Subsequently, copper wire was electrically joined to each sanded metal, the joint was soldered with tin material and soldering iron. Consequently, the electrical continuity between the copper cable and the work metal was verified with a multimeter. Once this was done, the metal

body was encapsulated in epoxy resin to isolate the metal faces except the working one. The areas considered for the electrodes were 5 cm^2 for each of the Ms-Ms electrodes and in the galvanic pair it was 5 cm^2 for Ms and 1 cm^2 for Cu. Figure 1 shows how the electrodes were used in this work.

Experimental Procedure

The electrode systems were positioned in the electrochemical cell with a 0.5M NaCl solution, to evaluate each pair of carbon steel materials (Ms-Ms) applying the EN electrochemical technique and the same procedure was applied for the paired configuration. galvanic (Ms-Cu). Electrochemical measurements were performed with and without the Faraday cage.

An ACM Instruments® Field Machine model potentiostat/galvanostat was used, to which an electrochemical cell with an arrangement of three electrodes was connected as shown in figure 3. The reference electrode was calomel (Hg/HgCl$_2$ saturated), the electrodes of work (WE$_1$) embedded individually in each specimen to be evaluated and the auxiliary electrode (WE$_2$) is another specimen with the same characteristics as the previous one. The use of a Faraday cage was important to ensure non-interference of current and voltage signals as R$_n$ transients.
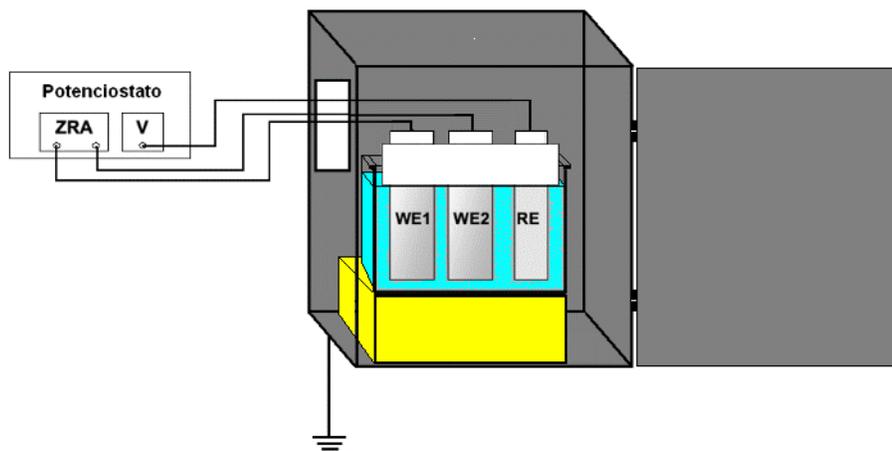

Figure 3. Instrumental arrangement for electrochemical noise measurements using the Faraday cage.

Using the potentiostat, the measurement of the variations in current and potential was programmed for a time of 600 seconds, with the following experimental parameters:
- Measurements were made every second, that is, at a sampling frequency of 1 Hertz.
- For each measurement block, in order to eliminate the DC trend (I$_{dc}$), the information obtained on current and potential was adjusted to a straight line with slope and intercept according to equations (2 and 3).
- The standard deviation of potential and current was estimated after subtracting the DC trend.
- Electrochemical noise resistance (R$_n$) was determined according to equation (8):
- Finally, the IL was estimated according to what is expressed in equation (9).

Analysis: Results and Discussion

The experimental measurements of electrochemical noise (EN) were performed as time series, it was necessary to evaluate with and without Faraday cage for nominally identical mild steels and in galvanic couple with copper. A solution of NaCl 0.5M was used as the electrolyte medium and in a three-electrode configuration, with four experiments carried out to achieve the objective of the study. For the RE, potential and current were monitored in the time series during 600 seconds of exposure. The results of EN without Faraday cage are shown in Figures 4(a) and 4(b), with the linear fit for the Ms-Ms and Ms-Cu galvanic couple systems and in Figures 5(a) and 5(b). The polynomial fit to those of Ms-Ms and galvanic couple Ms-Cu respectively. These graphs show the expressions that contain the trends obtained by adjusting the experimental results by linear regression and by least squares in which they were adjusted.
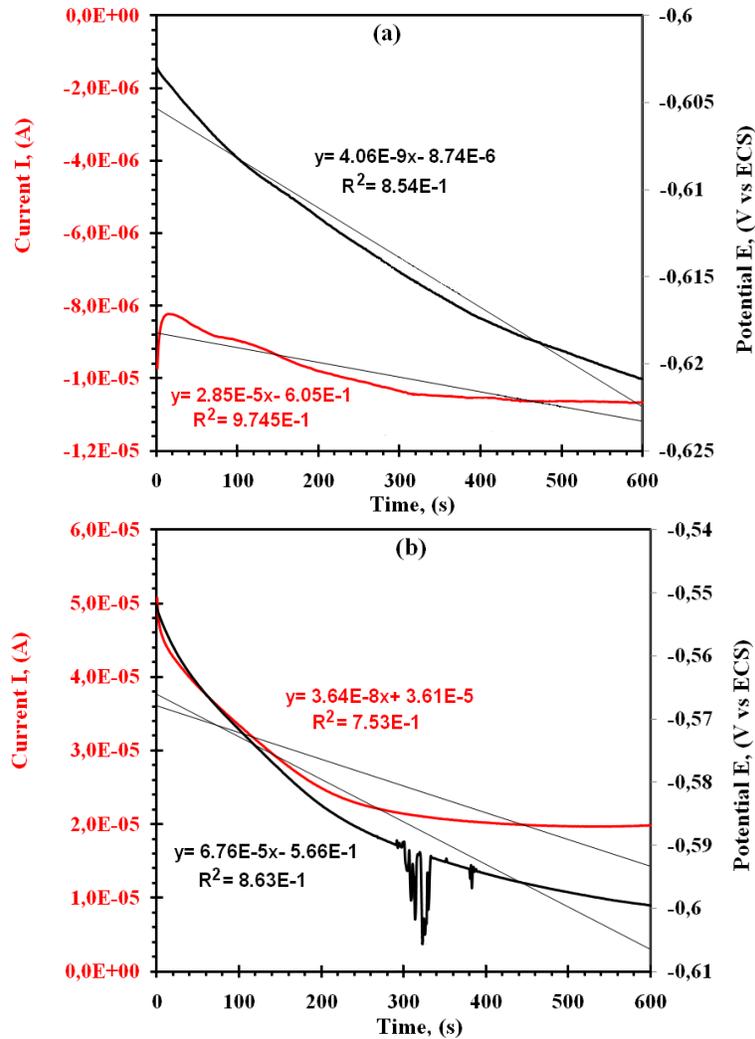
Figure 4. Linear fit graphs of EN for steel systems without Faraday cage, a) Ms-Ms and b) Ms-Cu.

The EN results with the Faraday Cage are shown in Figures 6(a) and 5(b) with the linear fit for the Ms-Ms and Ms-Cu galvanic couple system and in Figures 7(a) and 6(b) the polynomial adjustment to those of Ms-Ms and galvanic couple Ms-Cu, respectively. The expressions containing the trends obtained by fitting the experimental results by linear regression and by least squares in which they were fitted are shown.

According to Figures 4(a) and 5(a), mild steel immersed in 0.5 M NaCl tends to an active direction as time goes by. That is, the measured potential tends to decrease. In turn, the recorded current tends to quasi stabilize as time goes by. This behavior of the current can be correlated with a tendency of the metal to mixed corrosion in the chlorinated medium. This is an indication that the metallic material is probably being attacked mainly by uniform corrosion and less presence of pitting. Similar trends were observed in the caged systems of Figures 6(a) and 7(a) for the Ms-Ms electrode samples. For the case of the Ms-Cu galvanic couple, figures 4(b) and 5(b) show that the tendency to decrease activity was also observed, however, both current and voltage signals do not show stable activity at the end of the test, which indicates that the anodic surface is probably kept active by the incidence of localized corrosion on the steel that works here as anode and the copper that works as cathode for the oxygen-reducing activity, and in a very similar way for the results with cage shown in figures 6(b) and 7(b) respectively.

In previous studies it has been reported that this behavior is due to a transient decay of the potential signal as the activation of the anode surface develops, since being in galvanic couple, and with the aggressiveness of the medium as an adjuvant, generates greater sensitivity in the EN signal (Uruchurtu and Malo, 2002). However, the results with the Faraday cage show a better fit in the correlation factor in the current data up to 3% deviation for Ms-Ms and 25% in Ms-Cu compared to those of the system without cage, also observing a greater sensitivity. The deviation of the

correlation factor for the voltage showed some inconsistency, since there is no significant variation that can be attributed to the use of the Faraday cage.

Although the polynomial correlation factor was more accurate, it does not mean that DC trend removal is more effective, as can be seen in the noise resistance (R_n) and localization index (IL) results obtained from the linear and polynomial fit to the experimental data and carried out with and without a Faraday cage, as shown in Table 2.

The behavior observed in the results of R_n on the Ms-Ms systems with Faraday cage presented deviations of 1 to 2 % between the values of R_n without DC removal and with the linear adjustment method, instead the deviation observed with respect to those without the cage it was greater than 50%. The results of R_n with the polynomial method did not show validity when registering deviations greater than 90% in both systems with and without Faraday cage. For the results of R_n with the Ms-Cu galvanic couple systems, it was not possible to corroborate the behavior observed for the trends with and without the Faraday cage. It is important to mention that the asymmetries generated in the areas of the electrode systems used in chlorinated media and the RE response in a galvanic couple with asymmetric areas that influence the anodic and cathodic reactions involved in the EN signal that is measured experimentally (Bautista et al, 2001).
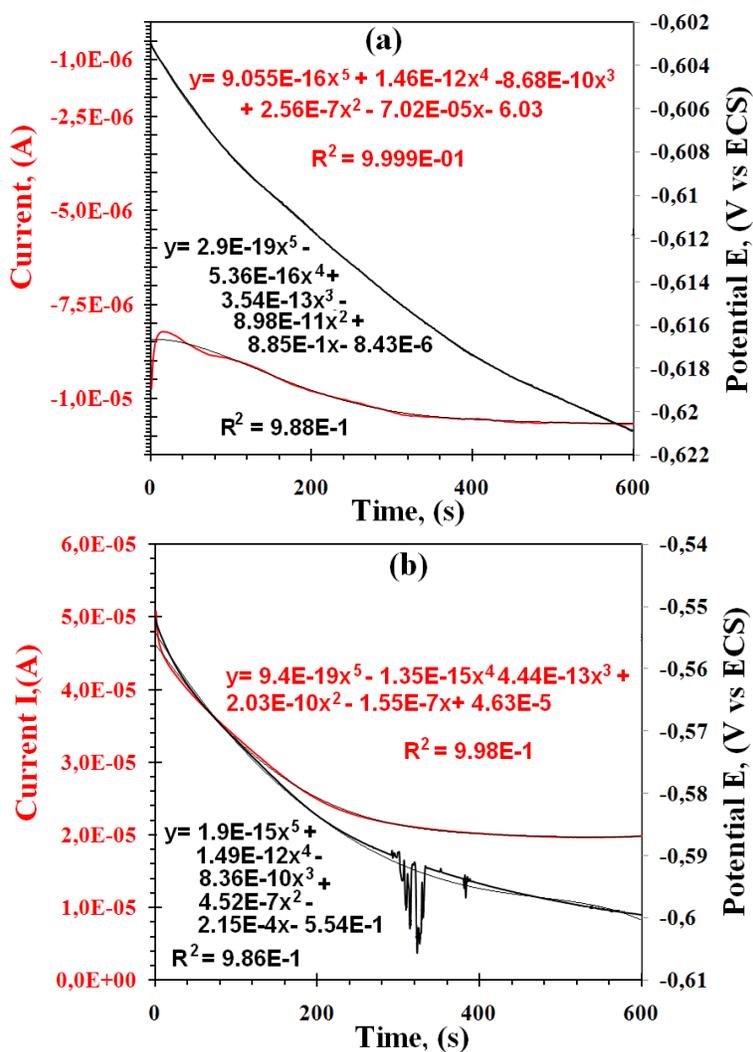

Figure 5. ER graphs with polynomial fit for steel systems without Faraday cage, a) Ms-Ms and b) Ms-Cu.
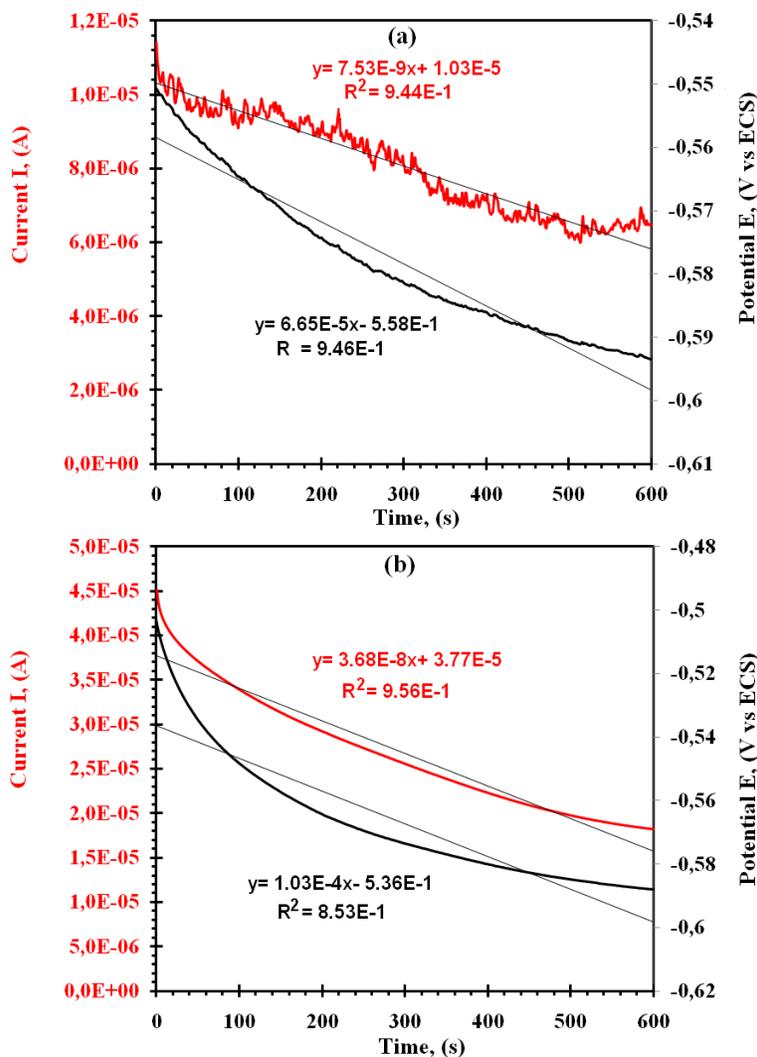
Figure 6. Linear fit plots of EN for Faraday cage electrode systems, (a) Ms-Ms and (b) Ms-Cu.

Figure 7. EN plots with polynomial fit for Faraday cage electrode systems, (a) Ms-Ms and (b) Ms-Cu.

The type of attack shown by the linear adjustment method of the IL index corresponds to mixed corrosion in both experimental arrangements for the Ms-Ms system, observing an inconsistent response in the Ms-Cu system with and without the Faraday cage, which is interpreted as a greater sensitivity for the case of the galvanic couple, where the electrochemical noise activity is seriously affected by the increase in corrosiveness that it produces in a galvanic couple on the anodic electrode (Ms), while Cu is cathodically protected , with the corresponding reduction of Oxygen dissolved in the electrolyte (Uruchurtu and Malo, 2002).

System and Condition	Fitting Method	Rn ($\Omega*cm^2$)	IL	Type of Corrosion
Ms-Ms without Faraday Cage	No	6571.58	0.08	Mixed
	Lineal	2748.7	0.03	Mixed
	Polynomial	437.98	0.01	Uniform
Ms-Cu without Faraday Cage	No	1735.48	0.28	Pitting
	Lineal	1289.28	0.14	Pitting
	Polynomial	4815.95	0.01	Uniform

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Ms-Ms with Faraday Cage	No	8821.69	0.16	Pitting
	Lineal	8671.33	0.04	Mixed
	Polynomial	957.41	0.02	Mixed
Ms-Cu with Faraday Cage	No	2972.65	0.08	Mixed
	Lineal	5453.9	0.05	Mixed
	Polynomial	3320.6	0.03	Mixed

Table 2. EN parameters obtained for systems with and without Faraday cage in individual metallic materials and in galvanic couple.

Countless studies have been reported on the influence of NaCl 0.5 as an aggressive medium and exposure to metals such as carbon steel and even copper. Although not visible to the naked eye, microscopically it is possible to observe how the chloride ions present in the medium can alter the surface of metals such as carbon steel and copper, since pitting is produced on the surface of the metal attributable to corrosion by pitting. However, in this work the measurement times are very short, which makes it necessary to carry out studies with a longer exposure time of at least 24 hours and also to analyze the surface morphology attacked with high-resolution microscopy to obtain convincing information that allows us to corroborate and compare the electrochemical response and identify the associated corrosion mechanisms.

Although in this situation both linear and polynomial fitting methods were applied to the source electrochemical noise signal to remove the trend in current (Idc), the usefulness of the linear method had previously been demonstrated in the works of Bertocci et al, (2000). and Xia and Benhamian, (2015). Thus, the uncertainty about the validity of the physical interpretation of the information was also reported, by increasing the order of the polynomial Mansfeld et al (2001), Chao et al, (2019), even some prefer to use the methods of the moving average and the Wavelet transform to remove the trend and reported in the works of Cao et al, (2019) and Botana et al, (2002).

Conclusions

Experimentation with the electrochemical noise technique under various conditions and analysis methods offers us the conclusions listed here.

It was possible to demonstrate the advantages offered by the linear fit method to remove the trend of the I_{dc} current involved in the noise response, compared to the results of the polynomial fit. However, it is necessary to extend the experimental phase to convince the community of EN experts about the improvement in the sensitivity of the noise response and to exercise better control over the exposure areas. It was also possible to demonstrate the advantages of using the Faraday cage, which allows to removing the external interferences that normally influence the sensitivity of the noise response, mainly current, a response that could be seen graphically and with the noise resistance values in the Ms-Ms system.

The statistical analysis, as well as the Rn and IL values, shed light on possible mechanisms such as adsorption and incorporation of chloride ions, producing an attack dominated by mixed corrosion, attributable to the quasi-stability of the metal-solution interface.

The initial experimental exposure conditions did not give an opportunity to identify the meta-stable pitting attack reported (Mansfeld & Xiao, 1993), which occurs during induction periods with longer exposure time to establish the conditions of localized corrosion at the concentration of chlorides used in our study.

As a final remark, it is recommended to establish educational practices about the interferences involved in the electrochemical measurements and also for the validation of the sensibility in the electrochemical noise signals, for instance by the use of the Faraday cage as a general-purpose procedure. However, it is important the mention that a more exhaustive experimental data with the Faraday cage design is necessary to better understand the current signals trends removal from the electrochemical noise measurements.

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