

## Effect of Exposure Period and Temperature on the Corrosion of Incoloy<sup>®</sup> Alloy 800<sup>™</sup> in Hydrochloric Acid Pickling Solutions

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The corrosion of Incoloy<sup>®</sup> alloy 800<sup>™</sup> (Ni32.5/Cr21/Fe) in 1.0 M HCl pickling solutions after varied exposure periods, namely, 0.5 h, 2.0 h, 4.0 h, and 24 h were reported. The work was also extended to include the corrosion of the alloy at 20 °C, 30 °C, 40 °C, 50 °C, and 60 °C after 0.5 h immersion in the acid solution. The electrochemical impedance spectroscopy (EIS) measurements showed that the corrosion of Incoloy decreases when the exposure period increases and the highest surface and polarization resistances were recorded for the electrode after 24 h immersion. On the other hand, the increase of the temperature of the solution was found to highly increase the corrosion as a result of decreasing the resistance of the alloy under the increased corrosivity of the acid solution. The cyclic potentiodynamic polarization (CPP) measurements confirmed the EIS ones that the increase of immersion time decreases corrosion, while the increase of temperature pronouncedly increases it for the Incoloy in HCl solutions. This is because elongating the time of immersion leads to decreasing the values of  $j_{\text{CORR}}$  and  $R_{\text{CORR}}$  but rising the temperature greatly increases them. CPP curves also indicated that the Incoloy dissolves in 1.0 M HCl solution as a result of the occurrence of the uniform corrosion.

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**Keywords:** acidic pickling solutions; exposure period; Incoloy<sup>®</sup> alloy 800; impedance spectroscopy; polarization

### 1. INTRODUCTION

Incoloy<sup>®</sup> alloy 800<sup>™</sup> is a solid solution strengthened alloy of nickel, chromium and iron with small additions of aluminium and titanium. Nickel-iron-chromium alloy 800 was introduced to the market in the 1950s to fill the need for a heat- and corrosion-resistant alloy with a relatively low nickel content since nickel was, at the time, designated a “strategic” metal [1]. Two other alloys, namely

Incoloy 800H and Incoloy 800HT, were developed to control the carbon content and grain size to optimize stress rupture properties. The three nickel steel alloys are identical in composition except for the higher level of carbon in alloy 800H, and the addition of up to 1.20 percent aluminum and titanium in alloy 800HT [2].

The high nickel content in Incoloy 800 makes the alloy highly resistant to both chloride stress-corrosion cracking and to embrittlement from precipitation of sigma phase, as well as, excellent corrosion resistance [3]. The chromium and nickel contents of the alloy give excellent oxidation resistance, good resistance to carburization and resistance to sulphidation at low concentrations of oxidizing and reducing sulphur. Furthermore, the chemical balance allows this alloy to exhibit excellent resistance to oxidation, carburization, and nitriding atmospheres [1,4]. Incoloy 800 is widely used in equipment that must have high strength and resistance to oxidation, carburization and other harmful effects of high temperature exposure (below 1200° F). The alloy is typically designed for use in chemical, petrochemical and food processing, for nuclear engineering and for the sheathing of electrical heating elements [5].

We have been studying surface properties of metals and alloys, their corrosion reactions, and effects of organic additives on its corrosion inhibition [6-10] in different media. In the earlier work [6], the corrosion of iron and API X-65 pipeline steel in 4.0% NaCl solution has been reported. In another study, the corrosion and corrosion inhibition of high strength low alloy steel in 2.0 H<sub>2</sub>SO<sub>4</sub> acidic pickling solutions using 3-amino-1,2,3-triazole as a corrosion inhibitor have been investigated [9]. More recently [10], the electrochemical corrosion behavior of API X-70 5L grade steel in 4.0 wt. % sodium chloride solutions after different immersion periods of time was studied using different electrochemical and spectroscopic techniques. In this work, we aimed at investigating the corrosion of Incoloy® alloy 800<sup>TM</sup> after 0.5, 2.0, 4.0, and 24 h immersion in 1.0 M HCl pickling solutions. The objective was also to report the effect of the increased temperatures, namely 20, 30, 40, 50, and 60 °C on the corrosion of the alloy after its immersion for 0.5 h in the acid solution. The study was performed using different electrochemical tests, i.e., OCP, CPP, and EIS measurements.

## 2. EXPERIMENTAL PROCEDURES

Incoloy® alloy 800 (Ni32.5/Cr21/Fe) rod was purchased from Goodfellow, UK. Hydrochloric acid (HCl, Glass World, 32%) was used as received. A conventional electrochemical cell accommodates only 200 mL with a three-electrode configuration was used. The three electrodes were the Incoloy rod, platinum foil, and an Ag/AgCl electrode (in 3.0 M KCl) were used as working, counter, and reference electrodes, respectively. The Incoloy rod had a cylindrical shape and diameter of 10 mm. The working electrode for electrochemical measurements were prepared by welding an insulated copper wire to one face of the Incoloy rod, and cold mounted in resin. The surface of the Incoloy electrode to be exposed to the solution was first ground successively with metallographic emery paper of increasing fineness of up to 600 grits and further with 5, 1, 0.5, and 0.3 µm alumina slurries (Buehler). The electrode was then washed with doubly distilled water, degreased with acetone, washed using doubly distilled water again and finally dried with tissue paper.

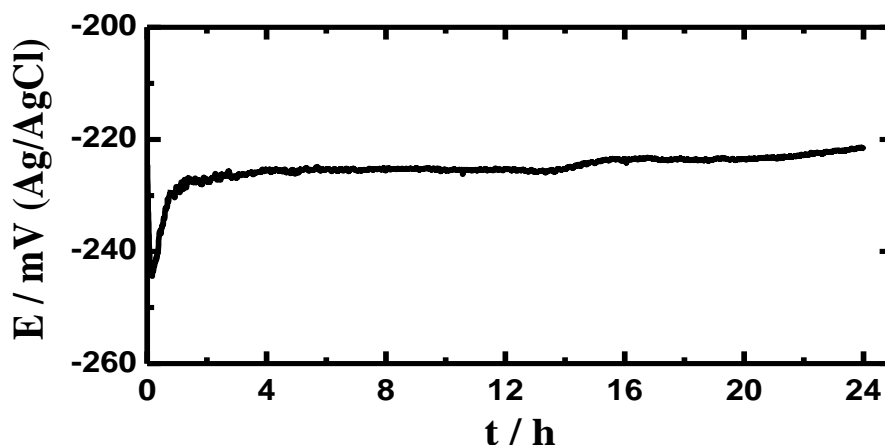
An Autolab Potentiostat (PGSTAT20 computer controlled) operated by the general purpose electrochemical software (GPES) version 4.9 was used to perform the electrochemical experiments. The open-circuit potential measurements were carried out for 24 h at room temperature. The electrochemical impedance spectroscopy (EIS) tests were performed at corrosion potentials over a frequency range of 100 kHz to 100 mHz, with an ac wave of  $\pm 5$  mV peak-to-peak overlaid on a dc bias potential, and the impedance data were collected using Powersine software at a rate of 10 points per decade change in frequency.

Cyclic potentiodynamic polarization (CPP) curves were obtained by scanning the potential in the forward direction from -0.80 V to 0.4 V vs. Ag/AgCl at a scan rate of 0.001 V/s. The potential was scanned again in the backward direction at the scan rate till the end of the run. Each experiment was carried out using fresh steel surface and new portion of the hydrochloric acid solution. All EIS and CPP experiments were carried out after different immersion periods and different solution temperatures.

### 3. RESULTS AND DISCUSSION

#### 3.1. Open-circuit potential (OCP) measurements

Fig. 1 shows the change of the OCP with time over 24 h exposure for the Incoloy electrode in 1.0 M HCl solution at room temperature.



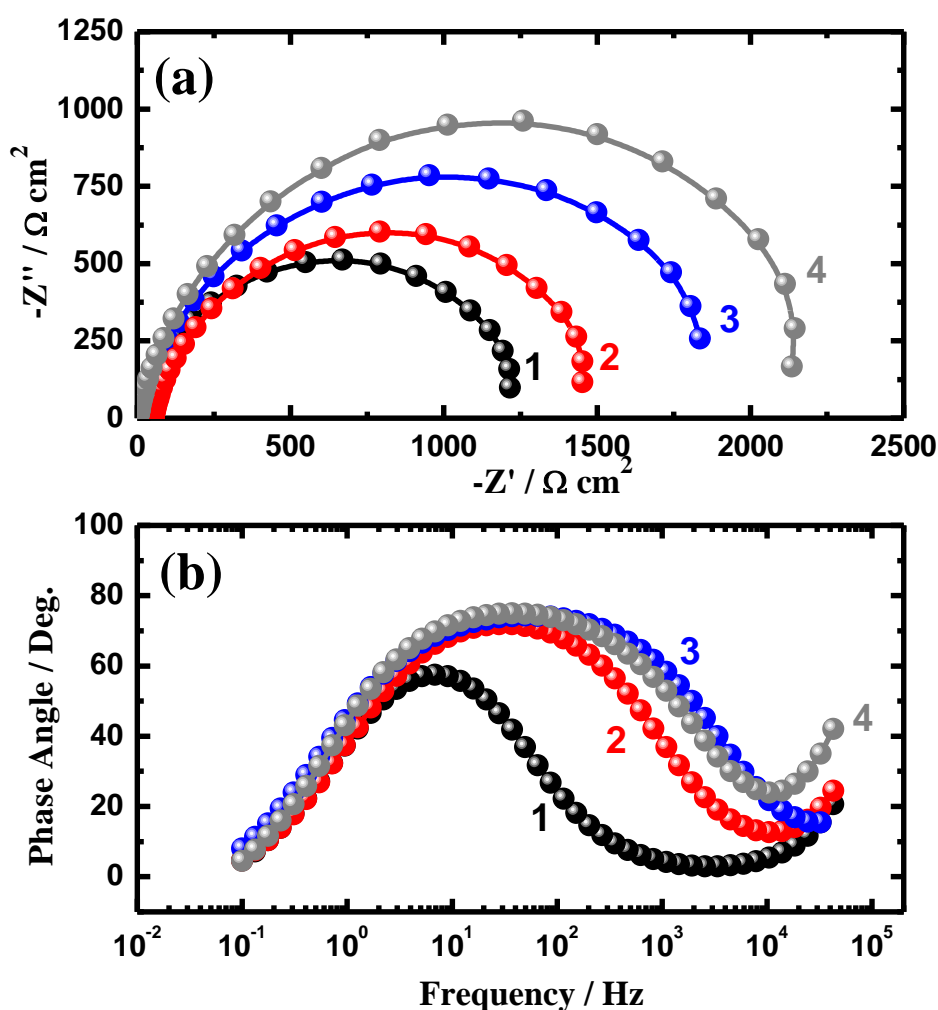
**Figure 1.** The change of the open-circuit potential with time curve obtained for Incoloy electrode in 1.0 M HCl solutions at room temperature.

It is seen from Fig. 1 that the potential of the Incoloy initially increased towards the more negative values from the first moment of electrode immersion as a result of the dissolution of an air oxide film was formed on the electrode before its immersion in the solution. The potential then started to have a rapid shift in the less negative direction, from  $\sim -0.245$  V to circa  $-0.228$  V vs. Ag/AgCl within only an hour time. This rapid positive potential shift indicates on the resistance of the Incoloy surface against corrosion due to the formation of an oxide film and/or a corrosion product layer. The

potential then slightly shifted towards the less negative value with increasing the time of the experiment to reach almost  $-0.222$  V at the end of the run. The change of potential with time thus indicates that the Incoloy dissolves in the acid solution upon its immersion, while increasing the immersion time decreases this dissolution via the formation of a corrosion product layer on the surface of the electrode.

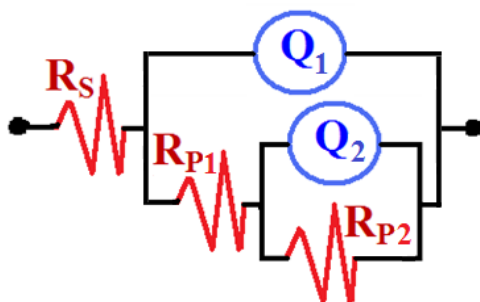
### 3.2. Electrochemical impedance spectroscopy (EIS) measurements

EIS is a powerful method that has been successfully used to understand the mechanism of corrosion and corrosion protection of metals and alloys in aggressive media [11-15]. In this work, the EIS technique was employed to report the kinetic parameters obtained from studying the corrosion of Incoloy® 800 after different exposure periods of time as well as different temperatures in 1.0 M HCl solutions.



**Figure 2.** (a) Typical Nyquist and (b) Bode phase angle plots obtained for Incoloy electrode after (1) 0.5 h, (2) 2.0 h, (3) 4 h, and (4) 24 h immersion in 1.0 M HCl solutions at room temperature.

Fig. 2 shows (a) typical Nyquist and (b) Bode phase angle plots obtained for Incoloy electrode after (1) 0.5 h, (2) 2.0 h, (3) 4 h, and (4) 24 h immersion in 1.0 M HCl solutions at room temperature. The spectra represented in Fig. 2 were analyzed by best fitting to the equivalent circuit model shown in Fig. 3. The EIS parameters obtained by fitting the equivalent circuit shown in Fig. 3 are listed in Table 1. The parameters of the equivalent circuit model can be defined according to the usual convention as follows;  $R_s$  represents the solution resistance between the Incoloy surface and the counter (platinum) electrode,  $Q_1$  represents the first constant phase elements (CPEs),  $R_{p1}$  is the polarization resistance of a film layer formed on the surface of the Incoloy electrode and can be also considered as the charge transfer resistance,  $Q_2$  is the second constant phase elements, and  $R_{p2}$  accounts for the polarization resistance of the oxide and/or corrosion product layer formed on the Incoloy surface.



**Figure 3.** The equivalent circuit model used to fit the experimental data obtained by EIS measurements; the symbols of the equivalent circuit are defined in the text and their values are listed in Table 1 and Table 2.

It is seen from the Nyquist plots of Fig. 2(a) that the Incoloy electrode showed only one semicircle whose diameter gets wider with increasing the immersion time from 0.5 h to 2.0 h, 4.0 h and further to 24 h before measurements; wider diameter of the semicircle reflects on a better corrosion resistance [11-14]. The effect of increasing the immersion time was also found to increase the maximum degree of the phase angle shown in Fig. 2(b). Here, EIS behavior indicates that the increase of immersion time decreases the corrosion of Incoloy in HCl solutions. This is because the Incoloy<sup>®</sup> 800 suffers uniform corrosion, which is characterized by its higher rates in short exposure periods then slows down with increasing the immersion time due to the formation of corrosion product layers on the metal surface that block or isolate it from being further attacked by the surrounding environment. This was further confirmed by the parameters listed in Table 1, where the increase of immersion periods increased the values of all obtained resistances, i.e.  $R_s$ ,  $R_{p1}$  and  $R_{p2}$ . The first constant phase elements ( $Q_1$ , CPEs) with its  $n$  values exactly 1.0 for all samples represent double layer capacitors [6,9,10]. This is because and depending on the value of  $n$ , CPE can represent capacitance ( $Z(\text{CPE}) = Cdl$ ,  $n = 1$ ), Warburg impedance for ( $n = 0.5$ ), or resistance ( $Z(\text{CPE}) = R$ ,  $n = 0$ ). Hence, the CPE for the Incoloy electrode is substituted for a capacitor to fit the semicircle more exactly [9]. The impedance of CPE in the case of Incoloy electrode in the hydrochloric acid, 1.0 M HCl, solutions can be expressed as following;

$$Z_{\text{CPE}} = 1 / Y_0 (j\omega) \quad (1)$$

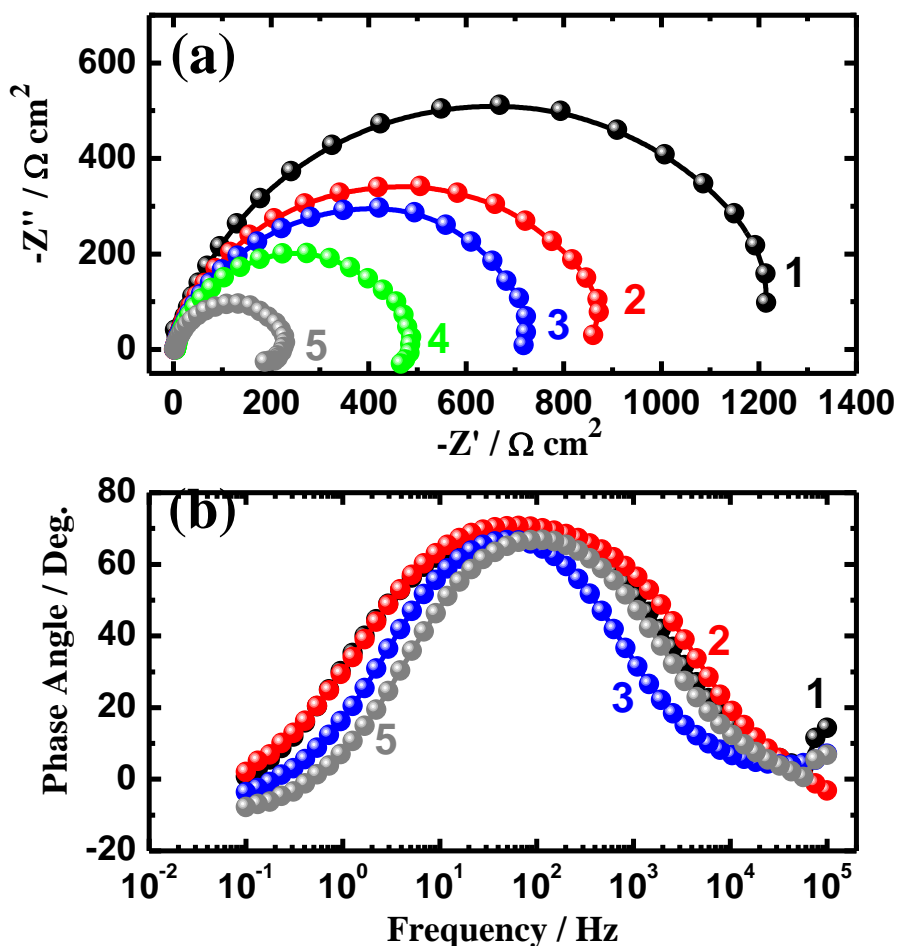
Where,  $Y_0$  is the magnitude of CPE,  $n$  is the exponent (phase shift),  $\omega$  is the angular frequency and  $j$  is the imaginary unit. The values of the CPEs decrease with increasing time. Also, the second constant phase elements,  $Q_2$ , with their  $n$  value was 0.84 for 0.5 h that increased to 0.92 for the electrode after 24 h immersion, which means that the CPEs here represent double layer capacitors with some pores; these pores were to cover the charged Incoloy surfaces under the corrosive attack of the acid solution [9]. The values of the CPEs also decreased with increasing the immersion time, which confirms the fact that the corrosion of Incoloy® alloy 800 in 1.0 M HCl solutions decreases with time.

**Table 1.** Parameters obtained by fitting the EIS data shown in Fig. 2 with the equivalent circuit shown in Fig. 3 for the Incoloy in 1.0 M HCl solutions after different immersion periods of time.

Exposure period	Kinetic EIS parameters						
	$R_s / \Omega \text{ cm}^2$	$Q_1$		$R_{p1} / \Omega \text{ cm}^2$	$Q_2$		$R_{p2} / \Omega \text{ cm}^2$
		$Y_{Q1} / \text{F cm}^{-2}$	$n$		$Y_{Q2} / \text{F cm}^{-2}$	$n$	
0.5 hour	12.63	0.000708	1.0	33.75	0.000146	0.84	976.8
2.0 hour	13.52	0.000581	1.0	40.36	0.000133	0.88	1458
4.0 hour	14.68	0.000467	1.0	58.71	0.000119	0.89	1890
24 hour	15.88	0.000461	1.0	70.42	0.000085	0.92	2200

In order to study the effect of temperature on the corrosion of Incoloy® alloy 800 in the hydrochloric acid solution, the EIS measurements were performed after 0.5 h immersion at different temperatures of the HCl solutions. Fig. 4 shows (a) Typical Nyquist and (b) Bode phase angle plots obtained for the Incoloy after 30 min immersion in 1.0 M HCl solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C, (4) 50 °C and (5) 60 °C, respectively. The data presented in Fig. 4 were best fitted to the same equivalent circuit shown in Fig. 3 and the values of the parameters of the circuit are listed in Table 2. The parameters of the equivalent circuit were defined as mentioned earlier. It is clearly seen from Fig. 4(a) that the Nyquist spectra show only one semicircle at all temperatures. The increase of the temperature of the hydrochloric acid solution led to decreasing the diameter of the semicircle, which indicates that the raise of temperature increases the corrosion of the Incoloy in the acid solution. Fig. 4(b) also shows that the increase of the temperature decreased the maximum degree of the phase angle. The Nyquist and Bode plots thus reveal that the increase of the temperature increased the corrosion of the Incoloy.

Table 2 shows that the increase of the solution temperature from 20 °C to 60 °C decreased the values of the resistances  $R_s$ ,  $R_{p1}$  and  $R_{p2}$ , increased the values of  $Q_1$  and  $Q_2$ . The decrease of the solution and polarization resistances with temperature of the solution was due to the increased corrosion of the Incoloy. Where, the increase of temperature increases the active centers on the surface of the Incoloy alloy and thus increases its reactivity to dissolve rapidly under the aggressiveness action of the hydrochloric acid solution.



**Figure 4.** (a) Typical Nyquist and (b) Bode phase angle plots obtained for the Incoloy after 30 min immersion in 1.0 M HCl solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C, (4) 50 °C and (5) 60 °C, respectively.

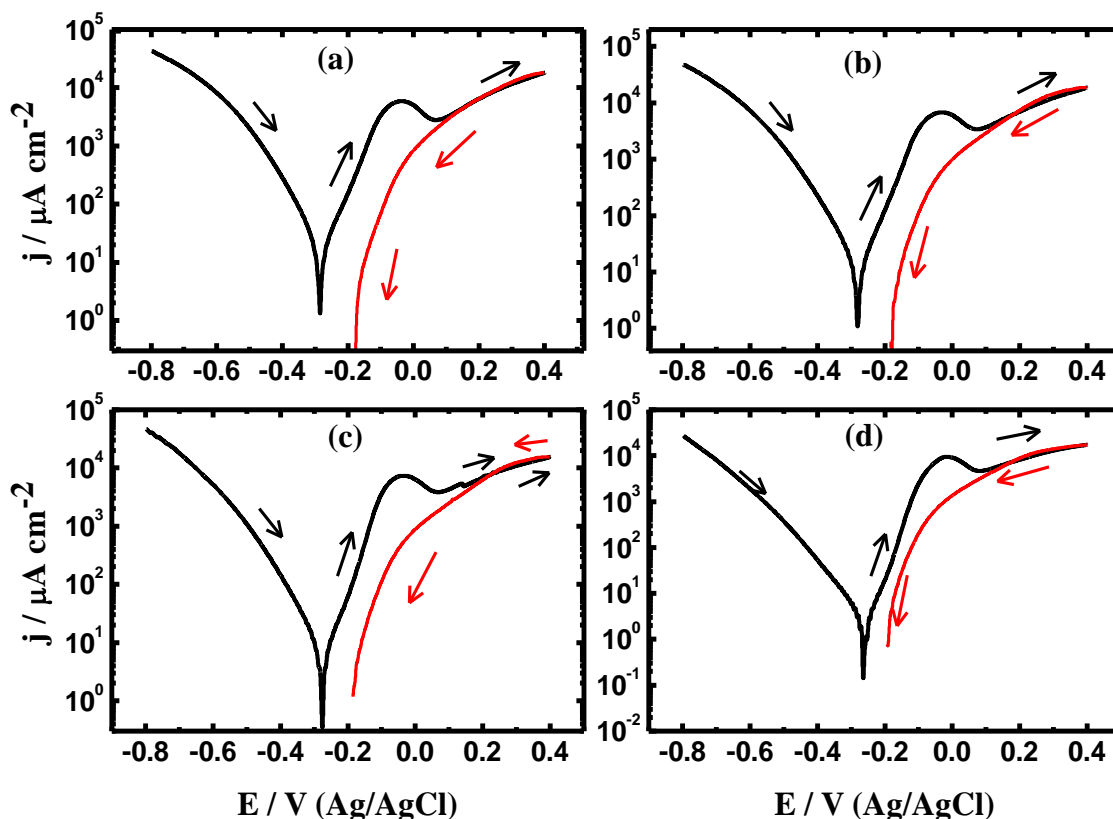
The values of the first CPEs,  $Q_1$ , with their  $n$  values are almost 1.0 represent double layer capacitors ( $C_{dl}$ ). As well as, the values of the second CPEs,  $Q_2$ , with their  $n$  values equal or close to unity and also represent double layer capacitors but with some porosity. The increase of the values of these CPEs also confirms that the corrosion of the Incoloy increased with increasing the temperature of the acid solution.

**Table 2.** Parameters obtained by fitting the EIS data presented in Fig. 4 with the equivalent circuit shown in Fig. 3 for the Incoloy in 1.0 M HCl solutions after different temperatures.

Temperature	Kinetic EIS parameters						
	$R_S / \Omega \text{ cm}^2$	$Q_1$		$R_{P1} / \Omega \text{ cm}^2$	$Q_2$		$R_{P2} / \Omega \text{ cm}^2$
		$Y_{Q1} / \text{F cm}^{-2}$	$n$		$Y_{Q2} / \text{F cm}^{-2}$	$n$	
20 °C	12.63	0.000708	1.0	33.75	0.000146	0.84	976.8
30 °C	11.77	0.000881	0.96	28.65	0.000161	1.0	730.6
40 °C	10.39	0.001324	1.0	22.33	0.000190	0.96	532.0
50 °C	9.66	0.002133	1.0	18.28	0.001143	0.86	391.4
60 °C	9.13	0.002717	0.95	14.46	0.001495	0.79	178.2

3.2. Cyclic potentiodynamic polarization (CPP) measurements

CPP method is one of the most used techniques in understanding the uniform and localized corrosion in harsh environments. In order to study the effect of immersion time on the corrosion behavior of the Incoloy® 800, the CPP curves were obtained after (a) 0.5 h, (b) 2.0 h, (c) 4.0 h, and (d) 24 h of immersion in 1.0 M HCl solutions at room temperature as shown respectively in Fig. 5. The polarization parameters obtained from the curves shown in Fig. 5 are listed in Table 3.



**Figure 5.** Cyclic potentiodynamic polarization curves obtained for Incoloy electrode after (a) 0.5 h, (b) 2.0 h, (c) 4 h, and (d) 24 h immersion in 1.0 M HCl solutions at room temperature.

These parameters include the values of cathodic Tafel slope ( $\beta_c$ ), corrosion potential ( $E_{Corr}$ ), corrosion current densities ( $j_{Corr}$ ), anodic Tafel slope ( $\beta_a$ ), polarization resistance ( $R_p$ ), and corrosion rate ( $R_{Corr}$ ). The values of  $\beta_c$  and  $\beta_a$  were determined according to our previous study [16,17]. The values of the  $j_{Corr}$  and  $E_{Corr}$  were obtained from the extrapolation of anodic and cathodic Tafel lines located next to the linearized current regions. From the slope analysis of the linear polarization curves in the vicinity of  $E_{Corr}$ , the values of  $R_p$  in the acid solution were obtained [18]. Also, the values of  $R_{Corr}$  (mills per year, mpy) were calculated using the equation [19]:

$$R_{Corr} = \frac{j_{Corr} k E_W}{d A} \tag{2}$$

Where, k is a constant that defines the units for the corrosion rate ( $k = 3272$ ),  $E_W$  the equivalent weight in grams/equivalent of Incoloy® 800 ( $E_W$  calculated = 24.21), d is the density in  $g\ cm^{-3}$  ( $d =$



7.95), and A the area of electrode in  $\text{cm}^2$ . It is well known that the cathodic reaction of metals and alloys in acidic solutions is the evolution of hydrogen according to the following equation;



On the other side, the anodic reaction that consumes the produced electrons from the cathodic reaction for the Incoloy is the dissolution of iron, which is the base metal in the alloy, as follows [20];



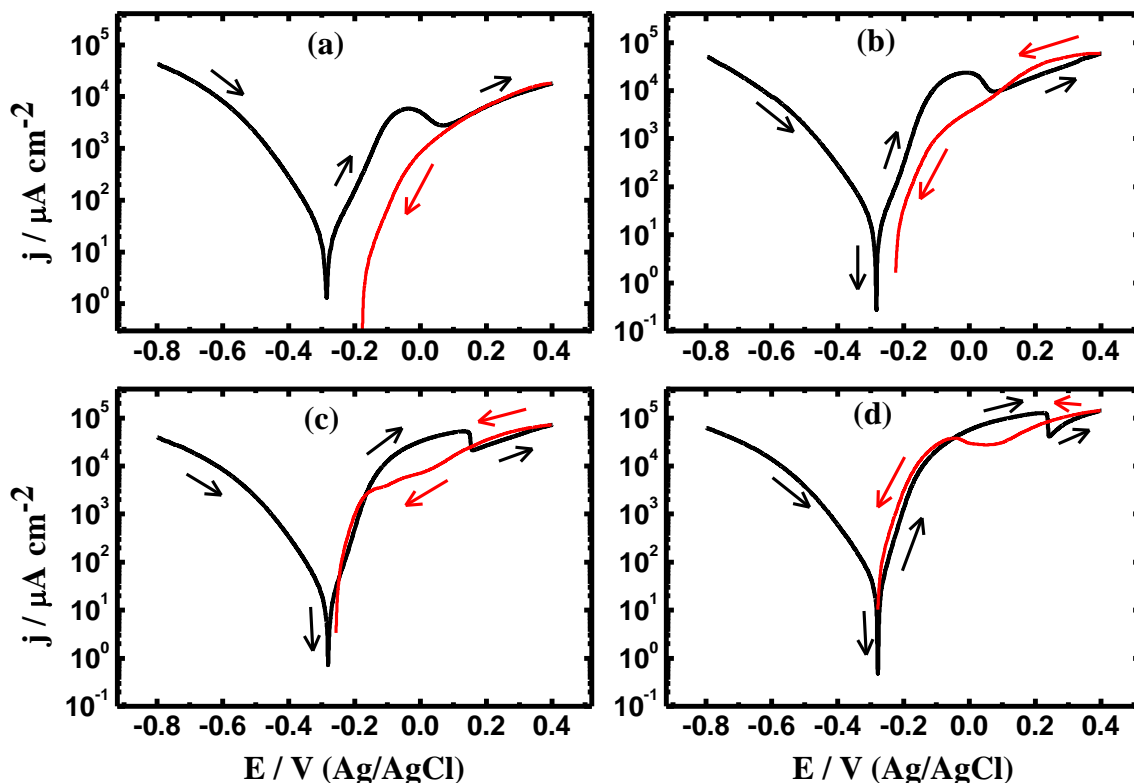
It is clearly seen from Fig. 5 that the anodic branch of the Incoloy shows an active-passive behavior for all immersion periods of time. The active region is defined by the rapid increases of current with increasing the potential in the first 100 mV from the OCP value. The increase of current here was due to the corrosion of the Incoloy through the dissolution of the iron presented in the alloy into ferrous cations,  $\text{Fe}^{2+}$ , as depicted in equation (4). Also, The produced  $\text{Fe}^{2+}$  further oxidized to ferric cations,  $\text{Fe}^{3+}$ , under the influence of the corrosive attack of the hydrochloric acid solution and with increasing the applied potential in the less negative direction as follows;



On the other hand, the passive region is noticed just after the active one, where the current stays steady and then decreases, which is due to the formation of an oxide film or as a result of the enrichment for the percent of Cr on the alloy surface and makes it more passivated [18,21,22]. The parameters shown in Table 3 indicate that increasing the exposure period of time shifted the  $E_{\text{Corr}}$  toward the less negative values and decreased the corrosion of the Incoloy through increasing the values of  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$  as well as increasing the corresponding  $R_p$  values. It is worth to mention also that the CPP behavior for all immersion periods of time did not show any pitting corrosion takes place for the Incoloy in 1.0 M HCl solution. Where, the values of the current in the backward recorded almost the same values in the forward direction at the more positive potential values. Further increment in the backward potential values toward the more negative direction provided lower current values compared to the currents recorded in the forward direction. This confirms that the Incoloy did not suffer pitting attack but only uniform corrosion that decreases with increasing its exposure period in 1.0 M HCl solutions from 5.0 h to 2.0 h and further to 4.0 h and the lowest  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$  with the highest  $R_p$  values were recorded for the electrode after 24 h immersion before measurement.

**Table 3.** Corrosion parameters obtained from the potentiodynamic polarization measurements for the Incoloy electrode that was immersed for different periods of time in 1.0 M HCl solutions.

Exposure period	Parameter					
	$\beta_c$ $\text{Vdec}^{-1}$	$E_{\text{Corr}}$ V	$j_{\text{Corr}}$ $\mu\text{A cm}^{-2}$	$\beta_a$ $\text{Vdec}^{-1}$	$R_p$ $\Omega \text{cm}^2$	$R_{\text{Corr}}$ $\text{mmy}^{-1}$
0.5 hour	0.090	-0.285	20	0.078	908	0.20
2.0 hour	0.095	-0.280	17	0.078	1095	0.17
4.0 hour	0.105	-0.275	12	0.073	1560	0.12
24 hour	0.105	-0.270	1.8	0.070	10145	0.02



**Figure 6.** Potentiodynamic polarization curves obtained for Incoloy electrode that was immersed in 1.0 M HCl solutions for 5.0 h at (a) 30 °C, (b) 40 °C, (c) 50 °C, and (d) 60 °C, respectively.

**Table 4.** Parameters obtained from the potentiodynamic polarization measurements shown in Fig. 6 for the Incoloy electrode in 1.0 M HCl solutions at different temperatures.

Temperature	Parameter					
	$\beta_c$ Vdec <sup>-1</sup>	$E_{Corr}$ V	$j_{Corr}$ $\mu A\ cm^{-2}$	$\beta_a$ Vdec <sup>-1</sup>	$R_p$ $\Omega\ cm^2$	$R_{Corr}$ $mmy^{-1}$
20 °C	0.090	-285	20	0.078	908	0.20
30 °C	0.083	-0.275	23	0.070	718	0.23
40 °C	0.080	-0.270	26	0.060	573	0.26
50 °C	0.080	-0.268	30	0.058	487	0.30
60 °C	0.085	-0.265	60	0.055	242	0.60

In order to confirm the effect of increasing temperature on the corrosion behavior of the Incoloy® alloy 800, we carried out polarization measurements at (a) 30 °C, (b) 40 °C, (c) 50 °C, and (d) 60 °C and the curves are shown respectively in Fig. 6. The corresponding corrosion parameters obtained from Fig. 6 are listed in Table 4. These parameters were also obtained as previously

mentioned for the Incoloy after different exposure periods of time. The CPP measurements shown in Fig. 6 indicate that the increase of temperature increased the active region and decreased the passive region, where the passive region almost disappeared at 60 °C, Fig. 6(d). This effect increased the corrosion of the Incoloy in 1.0 M HCl also by the significant increase in the values of  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$ , which reflects on decreasing the values of  $R_p$  as can be seen from Table 4. The CPP results thus agree with the data obtained from the EIS measurements that the corrosion of Incoloy® 800 increases with increasing the time of immersion as well as with the increase of the acid temperature.

#### 4. CONCLUSIONS

The effects of immersion time and solution temperature on the corrosion of Incoloy® alloy 800 (Ni32.5/Cr21/Fe) in 1.0 M HCl pickling solutions were reported. The study was carried out by using open-circuit potential, electrochemical impedance spectroscopy (EIS) and cyclic potentiodynamic polarization (CPP) measurements. EIS measurements indicated that increasing the immersion time was found to decrease the corrosion of the Incoloy alloy in 1.0 M HCl solutions due to increasing the polarization resistance with time. CPP results revealed that the increase of immersion time shifted the values of  $E_{\text{Corr}}$  for the alloy towards the less negative direction, decreased the values of  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$  as well as increased the values of  $R_p$ . It has been also found that the increase of temperature of the hydrochloric acid solution remarkably increased the corrosion of the Incoloy alloy due to the decrease of the polarization resistance and the increase of its cathodic and anodic currents,  $j_{\text{Corr}}$  and  $R_{\text{Corr}}$ . Results together confirmed that the increase of exposure period of time decreased the corrosion of the Incoloy® alloy 800, while the increase of the temperature of the HCl solution increases that corrosion.

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