

# Green Approach to Corrosion Inhibition of Copper by the Extract of *Calligonum comosum* in Strong Acidic Medium

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The corrosion inhibition effect of *Calligonum comosum* (CC) extract on copper (Cu) in 2 mol L<sup>-1</sup> HCl medium has been investigated by potentiodynamic polarization and electrochemical impedance spectroscopy methods. The polarization studies showed that CC extract acts as mixed type protection and maximum inhibition efficiency. 80.06 pct in 2 mol L<sup>-1</sup> HCl is obtained at 0.8 g L<sup>-1</sup> CC extract. The Nyquist plots showed an increase of the charge-transfer resistance with increasing of the inhibitor concentration and decrease double-layer capacitance. The inhibitory action of the extract was discussed in view of the Langmuir adsorption isotherm.

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## I. INTRODUCTION

COPPER is a metal with a wide range of applications such as using in the cooling systems of nuclear installations, automobiles, power plants, hotels, oil refineries, and sugar factories<sup>[1-3]</sup> owing to its high electrical and thermal conductivity, mechanical workability, and malleability. However, it is an active metal that does not resist well to corrosion, particularly in the presence of an aggressive acid environment.<sup>[4]</sup>

Some methods and compounds have been used for corrosion protection of metal in a corrosive solution such as organic compounds, especially those with N, O, and S atoms<sup>[5,6]</sup> and organic self-assembled monolayer.<sup>[7,8]</sup> Nevertheless, most of these compounds are not only expensive but also toxic for living beings. Recently, the development of novel corrosion inhibitors of plant extracts has attracted some attention as safe to the environment corrosion inhibitor.<sup>[9]</sup> Plant extracts are an incredibly rich source of natural chemical compounds that can be extracted by simple procedures with low cost and are biodegradable, readily available, and nontoxic in nature.

Environmental green inhibitors have attracted several researchers for corrosion inhibition of mild steel, 304, and 316 stainless steels by plant extracts in acidic medium. There are numerous naturally occurring substances like *Azardica indica*,<sup>[10]</sup> *Ferula gummosa*,<sup>[11]</sup> *Black pepper*,<sup>[12]</sup> *Chamomile* and *Halfabar*,<sup>[13]</sup> *Strychnos nux vomica*,<sup>[14]</sup> *Ferula assa-foetida* and *Dorema ammoniacum*<sup>[15]</sup> were studied as corrosion inhibitors in HCl and H<sub>2</sub>SO<sub>4</sub> media, which have been reported to have an anti-corrosion effect. One of the compounds extracted from plants that can be used as anti-corrosion is CC extract compound.

Most compounds of CC extract, especially phenolic compounds, contain heteroatom oxygen and aromatic rings.<sup>[16]</sup> Thus, it seems that CC extract can be a useful corrosion inhibitor. Presently, to the best of our knowledge, there is no reported work on inhibiting effects of the CC extract on Cu in hydrochloric acid solution. Therefore, in the present work, the effect of the CC extract on Cu in HCl solution has been studied.

The purpose of this work is to investigate the protection efficiency of the CC extract on Cu corrosion by Tafel polarization, electrochemical impedance spectroscopy (EIS), and the temperature effect [without and with the inhibitor was studied in the temperature range from 298 K to 338 K (25 °C to 65 °C)] in 2 mol L<sup>-1</sup> HCl.

## II. EXPERIMENTAL

### A. Materials

The Cu was used with purity of ≥99.9 wt pct in the present study. The metal sheet (Cu) was cut into rectangular samples of 1 cm × 1 cm × 0.1 cm soldered with Cu wire for an electrical connection and mounted onto the epoxy resin to offer only one active flat surface exposed to the corrosive environment. All other chemicals were purchased from Merck. Corrosion tests were carried out to using a μ-AUTOLAB potentiostat/galvanostat model μIII AUTO 71174 connected to a Pentium IV personal computer through a USB electrochemical interface. A polished Cu electrode was used as working electrode in the conventional three-electrode cell. The polarization and impedance obtained data were analyzed using Nova 1.6 electrochemical software. The temperature test was adjusted to 298 K to 338 K (25 °C to 65 °C) using TAMSON model T1000 thermostat.

### 1. Plant material

Flowering aerial parts of *Calligonum comosum* were collected during spring and summer 2013 from Kashan area (Isfahan Province, Iran) at an altitude of 850 m. Stem and flowers were separated, dried in the shade, and

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ground (80 mesh). An authenticated specimen of the plant was also deposited in the herbarium of the Kashan Research Botanical Garden, Research Institute of Forests and Rangelands, Kashan, Iran.

### 2. Preparation of methanol extracts

Ten grams of the powdered stem and flower was Soxhlet extracted with 500 mL of methanol for 8 hours. Solvent was removed by rotary evaporation (Buchi, Flawil, Switzerland) and the residue was dried in a vacuum oven at 323 K (50 °C). The CC extract was from the 0.1 to 0.8 g L<sup>-1</sup> in 2 M HCl solution and was prepared by dilution of 37 pct HCl (Merck) using distilled water.

### 3. Preparation of electrodes

Before each experiment, the working electrode was abraded with a sequence of emery papers of different grades (320 to 2000 grain size) and then electrode substrates were washed with distilled water, thoroughly degreased with ethanol and washed with distilled water again and dried with soft paper, then immediately inserted into the glass cell containing test solution.

## B. Methods

### 1. Test solutions

The experimental measurements were carried out in 2 mol L<sup>-1</sup> HCl solution in the absence and presence of various concentrations of CC extract solution. All the solutions were prepared using HCl and CC extract in distilled water. All the experiments were done at 298 K (25 °C) in solutions open to the atmosphere under unstirred conditions. A freshly prepared solution was used for each experiment.

### 2. Corrosion tests

Electrochemical experiments performed in conventional three-electrode electrochemical cell with the Cu as a working electrode, a platinum electrode as a counter electrode, and saturated (3 mol L<sup>-1</sup>) Ag/AgCl electrode as a reference electrode. Before polarization and EIS measurements, the working electrodes were immersed into the test solution and left for 1600 s at the open circuit potential (OCP). A scan rate of potential was 0.005 V s<sup>-1</sup>, and potential was scanned in the range of -200 to +200 mV relative to the corrosion potential. The corrosion potential ( $E_{\text{corr}}$ ) and the corrosion current ( $I_{\text{corr}}$ ) were obtained from the Tafel plots using the NOVA 1.6 software. In the case of EIS, a.c. signals of 10 mV amplitude and various frequencies from 100 kHz to 0.01 Hz at open circuit potentials were impressed while the Cu (working electrode) surface area was 1 cm<sup>2</sup>.

## III. RESULTS AND DISCUSSION

### A. Tafel Polarization Measurements

The potentiodynamic polarization behavior of Cu in 2 mol L<sup>-1</sup> HCl in the presence of various concentrations of CC extract is shown in Figure 1. The corrosion parameters

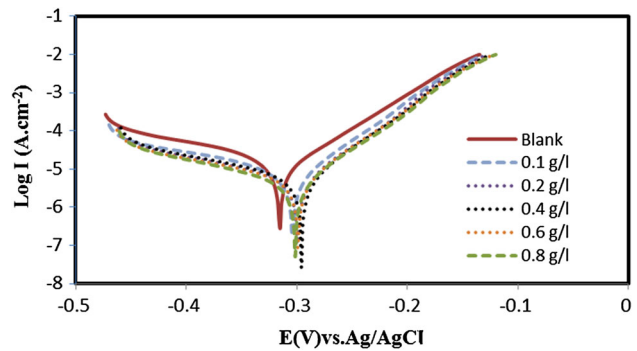


Fig. 1—Polarization curves of Cu in 2 mol L<sup>-1</sup> HCl containing various concentrations of CC extract.

such as corrosion potential ( $E_{\text{corr}}$ ), corrosion current density ( $I_{\text{corr}}$ ), anodic, and cathodic Tafel slopes ( $b_a$  and  $b_c$ ) were derived from these curves (Table I). The inhibition efficiency (IE) is calculated by the following expression<sup>[17]</sup>:

$$\text{IE pct} = \frac{i_{\text{corr}} - i_{\text{corr}}(c)}{i_{\text{corr}}} \times 100, \quad [1]$$

where  $I_{\text{corr}}(c)$  and  $I_{\text{corr}}$  are the corrosion current density of Cu with and without CC extract in HCl solutions, respectively.

The addition of CC extract in the concentration range of 0.1 to 0.8 g L<sup>-1</sup> decreases the  $I_{\text{corr}}$  value from 21.9 (blank) to 4.3  $\mu\text{A cm}^2$  (0.8 g L<sup>-1</sup>) resulting in 80.06 pct of IE pct. Regarding the potentiodynamic polarization curves, it can be clearly seen that the  $E_{\text{corr}}$  values shifted to positive potentials in the presence of CC extract concentration, although there was not a specific relation between  $E_{\text{corr}}$  and inhibitor concentration. It is also worth noting that the values of  $b_a$  and  $b_c$  changes dramatically with the addition of CC extract. This indicates that CC extract acts as a mixed type of inhibitor. The polarization resistance ( $R_p$ ) is an important parameter, that ability of inhibitor in the prevention of electron exchange in a corrosive environment. The  $R_p$  was calculated using the following equation<sup>[18]</sup>:

$$R_p = \frac{b_a \times b_c}{A \times i_{\text{corr}} \times (b_a + b_c)}, \quad [2]$$

where  $b_a$  and  $b_c$  are the anodic and cathodic Tafel slopes, respectively,  $I_{\text{corr}}$  the corrosion current density values, and A is electrode surface that obtained from the polarization studies. As can be seen from Table I,  $R_p$  increases from  $3.0 \times 10^2 \Omega \text{ cm}^2$  for blank to  $9.1 \times 10^3 \Omega \text{ cm}^2$  for 0.8 g L<sup>-1</sup> (optimal condition), and this shows that the CC extract provides the high polarization resistance. This effect may be related to the adsorption of the CC extract compound at the active sites of the electrode surface, retarding the corrosion reaction.<sup>[19]</sup>

### B. Electrochemical Impedance Measurements (EIS)

Figure 2 shows Nyquist plots obtained from impedance measurements for Cu in 2 mol L<sup>-1</sup> HCl in the presence of different concentrations of the CC extract

**Table I. Polarization Parameters for Cu in 2 mol L<sup>-1</sup> HCl in the Presence and Absence of CC Extract**

$C_{\text{extract}}$ (g L <sup>-1</sup> )	$E_{\text{corr}}$ (mV)	$R_p$ ( $\Omega$ cm <sup>2</sup> )	$b_c$ (mV dec <sup>-1</sup> )	$b_a$ (mV dec <sup>-1</sup> )	$I_{\text{corr}}$ ( $\mu$ A cm <sup>-2</sup> )	IE ( pct)
Blank	315	$3.0 \times 10^2$	105.37	174.99	21.92	—
0.1	303	$5.7 \times 10^3$	61.89	133.17	7.36	66.40
0.2	299	$6.7 \times 10^3$	58.85	137.68	6.11	72.10
0.4	295	$7.0 \times 10^3$	55.03	142.38	5.61	74.38
0.6	298	$7.5 \times 10^3$	54.83	140.76	5.21	76.20
0.8	301	$9.1 \times 10^3$	56.69	134.71	4.37	80.02

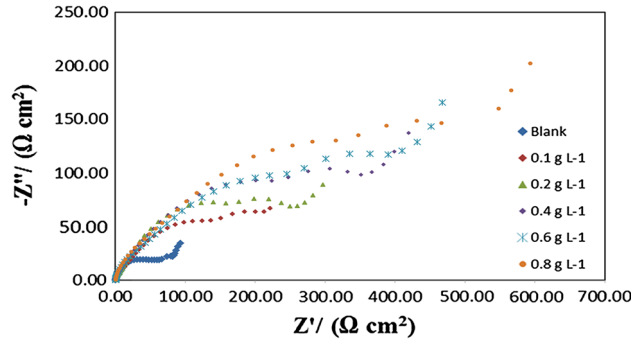


Fig. 2—Nyquist plots for Cu in 2 mol L<sup>-1</sup> HCl containing CC extract with different concentrations.

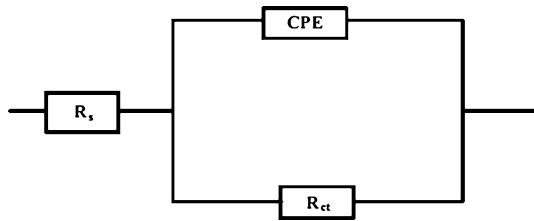


Fig. 3—The equivalent circuit model.

inhibitor. These impedance plots were modeled by the equivalent circuit as depicted in Figure 3. The Nyquist plot contains a depressed semicircle with the center under the real axis, such behavior is characteristic for solid electrode which is attributed to surface roughness and inhomogeneities of metal electrodes. The existence of a single semicircle shows the presence of single charge transfer process during dissolution, which is unaffected by the presence of an inhibitor molecule. The charge-transfer resistance ( $R_{ct}$ ) and the interfacial double layer ( $C_{dl}$ ) values are derived from these curves (Table II). The IE obtained from the charge-transfer resistance is calculated by the following relation<sup>[1]</sup>:

$$IE \text{ pct} = \frac{R_{ct}^{\text{inh}} - R_{ct}^{\text{bare}}}{R_{ct}^{\text{inh}}} \times 100, \quad [3]$$

where  $R_{ct}^{\text{inh}}$  and  $R_{ct}^{\text{bare}}$  are the charge-transfer resistances in the presence and absence of the CC extract. The values of IE pct are in quite good agreement with the results obtained previously from polarization measurements. For the description of a frequency independent phase shift between an applied alternating potential

**Table II. Impedance Data of Cu in 2 mol L<sup>-1</sup> HCl With and Without CC Extract**

$C_{\text{extract}}$ (g L <sup>-1</sup> )	$R_s$ ( $\Omega$ cm <sup>-2</sup> )	$R_{ct}$ ( $\Omega$ cm <sup>-2</sup> )	$C_{dl}$ ( $\mu$ F cm <sup>-2</sup> )	$n$	IE pct
Blank	0.80	77	1620	0.76	—
0.1	0.66	257	1596	0.60	70.04
0.2	0.76	323	1270	0.58	76.16
0.4	0.58	381	1136	0.61	79.79
0.6	0.79	391	886	0.60	80.31
0.8	0.68	444	549	0.64	82.65

and its current response, a constant phase element (CPE) is used instead of capacitance (C). The CPE is defined by the mathematical expression<sup>[20,21]</sup>:

$$Z_{\text{CPE}} = 1/Y_0(jw)^n,$$

where  $Z_{\text{CPE}}$ , impedance of CPE;  $Y_0$ , a proportional factor;  $w$ , angular frequency;  $j$ ,  $(-1)^{1/2}$ ; and  $n$ , the exponential term, which has many different explanations; it can be associated with the roughness of electrode surface,<sup>[22]</sup> a distribution of reaction rates,<sup>[21]</sup> non-uniform current distribution,<sup>[23,24]</sup> etc.

Simulation of Nyquist plots with a model containing constant phase element (CPE) instead of capacitance and charge-transfer resistance ( $R_{ct}$ ) showed excellent agreement with experimental data. The main parameters deduced from the analysis of the Nyquist diagram for 2 mol L<sup>-1</sup> HCl containing various concentrations of CC extract are given in Table II. With increasing CC extract concentration, the  $R_{ct}$  increased and capacitance ( $Y_0$ ) decreased, indicating that increasing CC extract concentration decreased the corrosion rate. This fact suggests that the inhibitor molecules acted by adsorption at the metal/solution interface.<sup>[25]</sup> The lower value of  $n$  for 2 mol L<sup>-1</sup> HCl medium indicated surface inhomogeneity resulted from roughening of metal surface due to corrosion. Addition of plant extract (0.8 g L<sup>-1</sup>) was caused to decrease  $n$  value from 0.76 to 0.64, indicating reduction of surface inhomogeneity due to the adsorption of plant extract molecules.

From Table II, it is found that  $R_{ct}$  values increase with an increase in the inhibitor concentration, while  $CPE_{dl}$  values decrease, which results in maximum IE pct (82.6 pct) at high concentration.

The protective action of organic substances during metal corrosion is based on the adsorption ability of their molecules, where the resulting adsorption film isolates the metal surface from the corrosive medium.

### C. Effect of Temperature

Temperature has a great effect on the corrosion phenomenon. Generally the corrosion rate increases with the rise of the temperature. For this aim, the polarization experiment (Figure 4), in the range of temperature 308 K to 338 K (35 °C to 65 °C), is performed in the absence and presence of 0.1 g L<sup>-1</sup> concentration of CC extract. The corresponding data are shown in Table III.

It seems that increasing in the inhibitor concentration increases the number of molecules adsorbed over the Cu surface, blocking the active sites of acid attack and thereby protecting the metal from corrosion.<sup>[17]</sup> It is clear from Table III that by increasing the temperature in the range 308 K to 338 K (35 °C to 65 °C), the IE increased from 61.8 to 86.74 pct. Increasing IE with increasing temperature can be attributed to strong adsorption of the coating to the surface. The IE level also increased as the temperature increased (Table III). The fact that IE pct increased with temperature is explained by Bouyanzer *et al.*,<sup>[26]</sup> and de Souza *et al.*,<sup>[27]</sup> as the likely specific interaction between the metal surface and the inhibitor. Ivanov<sup>[28]</sup> considers the increase of IE pct with temperature increases as the change in the nature of the adsorption mode. The inhibitor is being physically adsorbed at lower temperatures, while chemisorption is favored as temperature increases. Noor *et al.*<sup>[29]</sup> suggested increasing temperature, when chemical changes occur in the inhibitor

molecules, leading to an increase in the electron density at the adsorption centers of the molecule, which causes an improvement in IE.

Arrhenius Eq. [4] and transition state Eq. [5] were used to calculate activation parameters for the corrosion process<sup>[30,31]</sup>:

$$W = k \exp\left(-\frac{E_a}{RT}\right), \quad [4]$$

$$W = \frac{RT}{Nh} \exp\left(\frac{\Delta S^*}{R}\right) \exp\left(-\frac{\Delta H^*}{RT}\right), \quad [5]$$

where  $W$  is the corrosion rate obtained from gravimetric measurements,  $K$  the Arrhenius pre-exponential factor,  $T$  absolute temperature,  $E_a$  the activation corrosion energy in the corrosion process,  $h$  Planck's constant,  $N$  Avogadro's number,  $\Delta S^*$  the entropy of activation, and

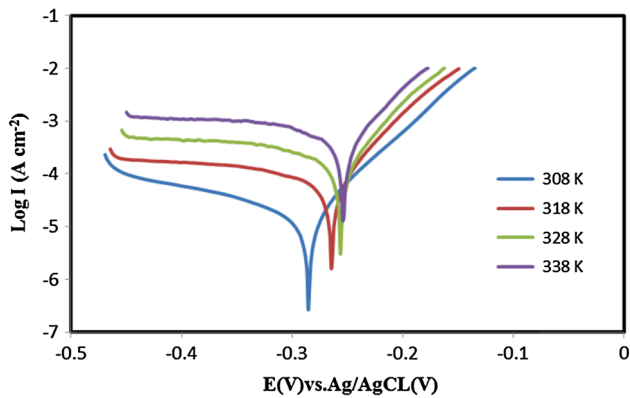


Fig. 4—Effect of temperature on polarization curves of Cu corrosion rate in free and inhibited acid solutions.

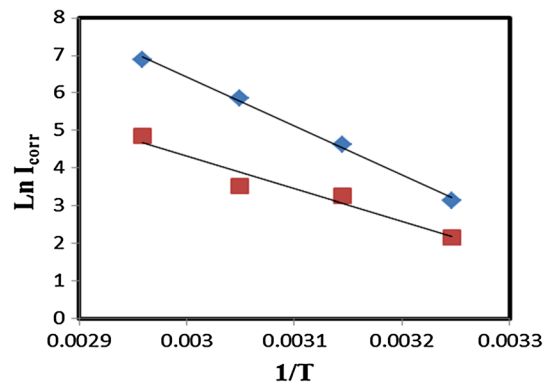


Fig. 5—Arrhenius plots of  $\ln(I_{\text{corr}})$  vs  $1/T$  in the absence and presence of 0.1 g L<sup>-1</sup> of CC extract.

Table IV. Activation Parameters,  $E_a$ ,  $\Delta H$ ,  $\Delta S$ , of the Dissolution of Cu in 2 mol L<sup>-1</sup> HCl in the Absence and Presence of Various Concentrations of CC Extract

Method	$K_{\text{ads}}$ (L g <sup>-1</sup> )	$\Delta G_{\text{ads}}$ (kJ mol <sup>-1</sup> )	$\Delta H_{\text{ads}}$ (kJ mol <sup>-1</sup> )	$\Delta S_{\text{ads}}$ (J mol <sup>-1</sup> )	$E_a$ (kJ mol <sup>-1</sup> )
Polarization	27.5	-25.32	47.11	-158.16	71.87
EIS	37.7	-26.10	—	—	—
Blank	—	—	—	—	108.33

Table III. Polarization Parameters for the Corrosion of the Cu in 2 mol L<sup>-1</sup> HCl Without and With Addition of Various Concentrations of CC Extract at Different Temperatures

Temperature [K (°C)]	CC (g L <sup>-1</sup> )	$E_{\text{corr}}$ (mV)	$I_{\text{corr}}$ ( $\mu\text{A cm}^{-2}$ )	$b_c$ (mV dec <sup>-1</sup> )	$b_a$ (mV dec <sup>-1</sup> )	IE pct
308 (35)	blank	285	23.00	57.660	380.13	61.81
	0.1	294	8.78	48.480	88.93	
318 (45)	blank	263	101.02	58.812	598.07	73.84
	0.1	298	26.41	62.071	603.75	
328 (55)	blank	256	350.64	80.220	889.09	90.36
	0.1	312	33.78	59.682	507.43	
338 (65)	blank	253	978.72	100.420	378.28	86.74
	0.1	290	129.74	66.375	2240.20	

$\Delta H^*$  is the enthalpy of activation.<sup>[17]</sup> The corrosion activation energy ( $E_a$ ) for different concentrations of CC extract is calculated by linear regression between  $I_{\text{corr}}$  and  $1/T$  (Figure 5), and the result is shown in Table IV. All the linear regression coefficients are close to 1, indicating that the Cu corrosion in HCl can be elucidated using the kinetic model.

The positive sign of the  $\Delta H^*$  reflects the endothermic nature of the Cu dissolution process (Table IV). It was suggested that the large and negative values of  $\Delta S^*$  refer to the activated complex in the rate-determining step and represents an association rather than a dissociation step. This finding therefore suggests that a decrease in disordering takes place on going from reactants to the activated complex.<sup>[32]</sup>

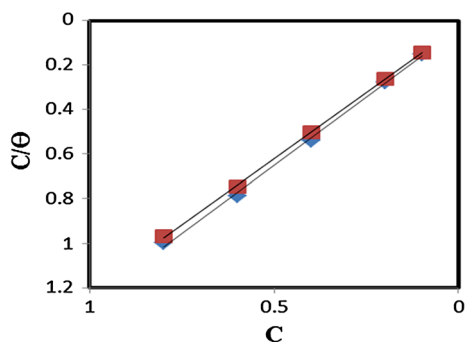


Fig. 6—Variation of  $C/\theta$  with different concentrations of CC extract obtained from polarization and EIS method.

#### D. Adsorption Isotherm

In order to gain more information about the mode of adsorption of these compounds on the surface of Cu, the experimental data have been tested with several adsorption isotherms including Langmuir, Frumkin, Freundlich and Temkin isotherms. However, the best fit is obtained from the Langmuir isotherm.<sup>[33]</sup> These adsorption isotherms were tested for consistency with adsorption behavior of the CC extract on Cu surface in 2 mol L<sup>-1</sup> HCl solution.

The fit to the Langmuir isotherm was determined by plotting the ratio of the extract concentration over the degree of surface coverage ( $\frac{C}{\theta}$ ) vs the extract concentration ( $C$ ), in order to check how consistent the data are with the following equation<sup>[33]</sup>:

$$\frac{C}{\theta} = \frac{1}{K_{\text{ads}}} + C, \quad [6]$$

where  $K_{\text{ads}}$  is the adsorption equilibrium constant. The correlation coefficient,  $R^2$ , was used to choose the isotherm that it is the best fit experimental data. The degree of surface coverage  $\theta$  ( $\theta = \text{IE}/100$ ) was evaluated from the polarization curves:

$$\theta = \frac{i_{\text{corr}} - i_{\text{corr}(c)}}{i_{\text{corr}}}. \quad [7]$$

The relation between  $C/\theta$  and  $C$  is shown in Figure 6. These plots are linear with a slope equal to unity ( $R^2 > 0.99$ ), while for the Temkin and Frumkin

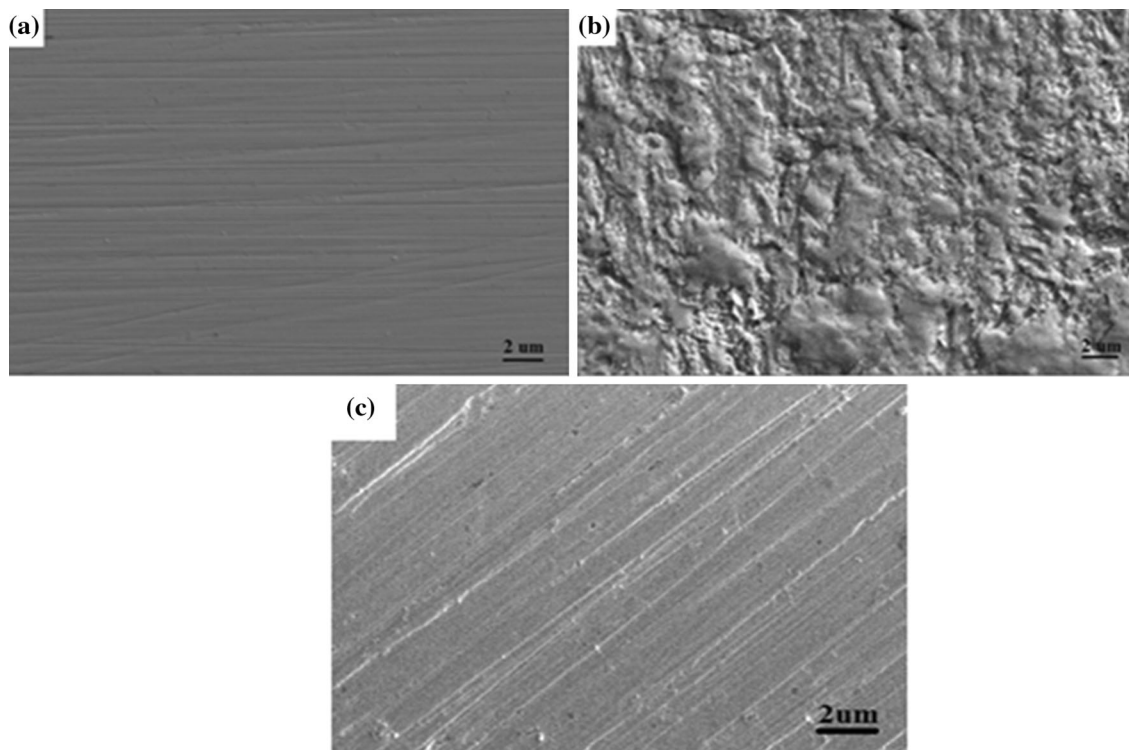


Fig. 7—SEM micrographs of a polished Cu specimen prior to testing (a), and the surface of Cu specimens after immersion in 2 mol L<sup>-1</sup> HCl solutions for 3 h: without CC extract (b), containing 0.8 g L<sup>-1</sup> CC extract (c).

isotherms, the  $R^2$  values obtained were 0.97 and 0.95, respectively. This result suggests that the adsorption of the CC extract on metal surface followed the Langmuir adsorption isotherm. This isotherm assumes that the adsorbed molecules occupy only one site, and there are no interactions with other adsorbed species.<sup>[34]</sup> The equilibrium constant for adsorption process which is related to the free energy of adsorption  $\Delta G_{\text{ads}}^0$  is expressed by the following equation<sup>[33]</sup>:

$$K_{\text{ads}} = \frac{1}{999} \exp\left(-\frac{\Delta G_{\text{ads}}^0}{RT}\right), \quad [8]$$

where  $R$  is the universal gas constant ( $8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ ),  $T$  the thermodynamic temperature, and 999 is the concentration of water in the solution expressed in  $\text{g L}^{-1}$ .

The energy of adsorption could not have been calculated due to the unknown molecular mass of the CC extract. The free energy of adsorption values  $\Delta G_{\text{ads}}^0$  was obtained for CC extract, and results indicate that the values of  $\Delta G_{\text{ads}}^0$  are  $-25.32$  for CC extract in  $2 \text{ mol L}^{-1} \text{ HCl}$ . The negative values of  $\Delta G_{\text{ads}}^0$  mean that adsorption of CC extract on Cu surface is a spontaneous process, and furthermore, the values of  $\Delta G_{\text{ads}}^0$  also show the electrostatic interactions of the inhibitor molecules and Cu surface.<sup>[33]</sup>

#### E. Scanning Electron Microscopy

Figure 7(a) shows the typical SEM images of abraded Cu. SEM micrographs obtained from Cu surface specimen immersion in  $2 \text{ mol L}^{-1} \text{ HCl}$  solutions for 3 hours in the absence of CC extract are shown in Figure 7(b). It can be observed that the Cu surface was strongly damaged in the absence of the inhibitor. However, Figure 7(c) shows that there was much less damage on the surface of the Cu and good protective film adsorbed on a specimen surface which is responsible for the inhibition of corrosion with CC extract.

## IV. CONCLUSIONS

The obtained results indicate that the CC extract can act as an efficient inhibitor of corrosion of Cu in  $2 \text{ mol L}^{-1} \text{ HCl}$  medium. The obtained IE of CC extract from polarization curves and impedance method is in reasonably good agreement. The Tafel behavior of CC extract indicates that it is a mixed type inhibitor. The results of impedance measurements indicate that the charge-transfer resistance ( $R_{\text{ct}}$ ) increased with increasing concentration of inhibitor, and also it slightly increased with rising temperature. The adsorption of the inhibitor molecules on the Cu surface in acid media obeys the Langmuir isotherm. The obtained values of  $\Delta G_{\text{ads}}$  indicate that the inhibitor is adsorbed on the Cu surface through electrostatic interactions. So, CC extract act as a good corrosion inhibitor for Cu in  $2 \text{ mol L}^{-1} \text{ HCl}$  medium. The results show the high performance of this inhibitor for protection of Cu corrosion which is

commonly used in heating and cooling systems in the industry. Finally the CC extract is a product that can be used as an alternative for toxic chemical inhibitors in acidization and acid pickling of Cu.

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