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Full Paper

Comparing the Inhibition Efficiency of Two Bio-Inhibitors to Control the Corrosion Rate of Carbon Steel in Acidic Solutions

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Abstract- In this study, the inhibition efficiency of two bio-inhibitors to decrease the corrosion rate of carbon steel in acidic solutions is investigated. These green inhibitors contained the protein prepared from the *Hyalomma* tick and leech extract. Both potentiodynamic polarization and electrochemical impedance spectroscopy measurements were utilized for this research. When the inhibitor concentration increased, the inhibition efficiency increased for both inhibitors; however, adsorption isotherm mechanisms for these inhibitors were different. In addition, the inhibition efficiency for the leech extract was higher than the *Hyalomma* tick extract in corrosive solutions. This inhibition efficiency range in the H_2SO_4 solution was about 93-97% based on polarization test results. Changes in corrosion potential showed that molecules adsorption of both bio-inhibitors on steel substrates acted as mixed inhibitors. Calculated ΔG_{ads} values for corrosion reactions in the presence of both bio-inhibitors demonstrated that a physical adsorption event has happened on steel substrates. Based on the EIS test results, when the concentration of *Hyalomma* tick extract increased to 0.06 g/L, the suggested electrical circuits changed due to the formation of a layer of inhibitor molecules on the steel substrate in the HCl solution.

Keywords- Bio-inhibitor; *Hyalomma* tick extract; Leech extract; Corrosion resistance; Acidic solutions

1. INTRODUCTION

Most of the materials in various industries degrade when they are exposed to corrosive environments. Thus, several methods are investigated to decrease such attacks. The usage of inhibitors in corrosive solutions is one of these techniques [1-3]. Therefore, in recent years, many researchers tried to suggest new, cheap, environment-friendly, and biodegradable materials as inhibitors to control corrosion reactions [5,6]. Different plant extracts were suitable candidates for this purpose since most of these products contain complex constituents, such as nitrogen and oxygen base compounds, alkaloids, flavonoids, tannins, organic acid, anthraquinones, polyamides, and polyphenols. It is found that these compounds are effective to decrease the corrosion rates when they adsorb on steel substrates as protective layers [6-9]. In the following paragraph, some of the known plant extracts are reviewed.

Nypa fruticans Wurmb leaves [1], Nicotiana *tabacum* [4], *Mentha rotundifolia* [5], *Ginkgo* [6], *Sida acuta* [8], *Foeniculum vulgare* [9], Targetes erecta (marigold flower) extract [10], *Sargassum wightii* [11], coconut shell powder [12], *Salvia officinalis* [13], Chondroitin sulfate [14], *Kappaphycus alvarezii* [15], leaves of Hibiscus sabdariffa [16], Olive leaf extract [17], *bawang dayak* [18], Henna (Lawsonia inermis) [19], Kalanchoe Blossfeldina [20], coconut leaf extract [21], and *Ficus carica* [22] were found as green inhibitors to reduce corrosion attacks of various materials in acidic solutions. Most of these inhibitors were used for decreasing corrosion rates of metallic substrates such as carbon steel, aluminum, and copper alloys in various acidic or caustic corrosive solutions. The inhibition efficiency range was about 50-94%.

Thus, in our previous study, a solid powder of *Hyalomma* tick extract was used as a new inhibitor with high concentrations of 1-3 g/L [23]. However, this paper studies the use of protein, extracted from the leech and *Hyalomma* ticks, as bio-inhibitors for affecting corrosion reactions of carbon steel in acidic solutions in low concentrations of 0.05-0.06 g/L. It is noticeable that proteins are examples of naturally polyamide groups with macromolecule structure. Such materials contain N and O elements that can be adsorbed on metallic surfaces. Therefore, the adsorption mechanisms of both bio-inhibitors on the steel substrates in 1 M HCl and 1 M H_2SO_4 solutions were investigated. Both electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization techniques were employed for this purpose. In addition, the amount of utilized inhibitor concentration was low enough to be considered as cost-effective bio-inhibitors.

2. UTILIZED MATERIALS AND METHODS

The type of utilized substrates was carbon steel (ST37-2). Before the corrosion tests, substrates were abraded, cleaned in the ethanol solution, and dried in hot air. For the protein extraction process, ticks and leeches were collected in 70% ethanol solution, separately. They were washed in ethanol solution several times and homogenized in the sterile micro-tubes with the phosphate-buffered saline (PBS) solution. It contained 10 mmol/L NaH_2PO_4, 1.8 mmol/L KH_2PO_4, 140 mmol/L NaCl, and 2.7 mmol/L KCl. The pH was 7.4. Then, the homogenized liquid was placed in a laboratory centrifuge for 30 mins at a speed of 1000 rpm. At last, the top liquid was extracted as the protein. The purity of the protein was measured by a NanoDrop spectrophotometer with the method of A280. A similar process was found elsewhere [24].

The *Hyalomma* tick and leech exist in almost all parts of Iran. The image of the *Hyalomma* tick is represented in Figure 1(a). The main material of the *Hyalomma* tick was chitin. Chitin has a large molecule with the formulas of $(C_8H_{13}O_5N)_n$ [25], as shown in Figure 1(b). In addition, Hirudin as the main material of leech (illustrated in Figure 1(c)) exhibits a large molecule with the structure shown in Figure 1(d). Totally, both bio-materials contained O and N elements in circular structures that could be effective as a corrosion inhibitor for metallic materials.

The *Hyalomma* tick and leech extracts were used as bio-inhibitors in HCl and H_2SO_4 solutions, respectively. The concentration of both acidic solutions was 1 M. The added concentration of inhibitors in corrosive solutions were 0.04, 0.05, and 0.06 g/L. Details of utilized specimens were reported in Table 1. Electrochemical tests were done using a potentiostat device (model AUTO LAB-OGF500) monitored by z-view software.

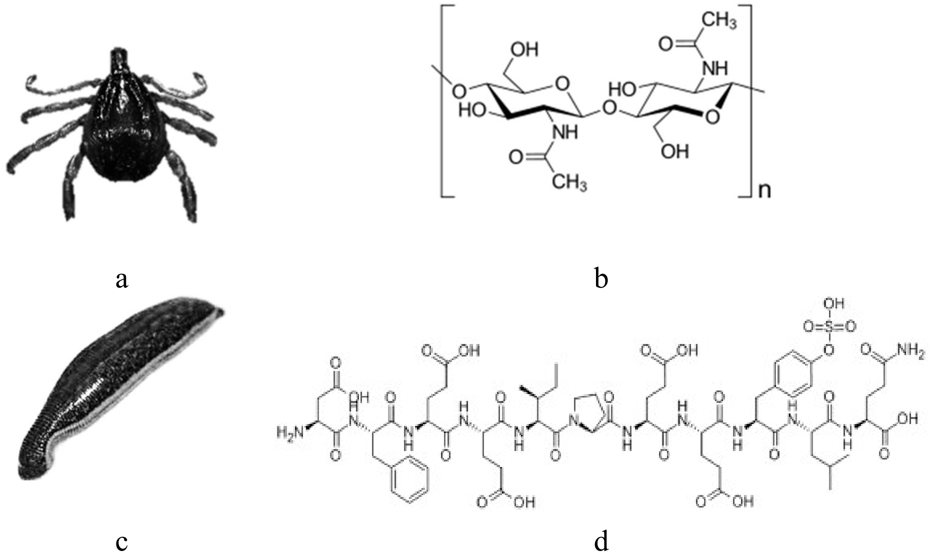


Figure 1. (a) *Hyalomma* tick image, (b) the schematic structure of chitin, (c) leech image, and (d) the schematic structure of Hirudin

For these tests, a conventional set of three electrodes were employed containing a steel substrate with the area of 1 cm^2 (as a working electrode), a platinum sheet (as an auxiliary electrode), and a saturated calomel electrode (as a reference electrode). The immersion time before the electrochemical measurement was about 30 mins. The frequency range for EIS tests was between 10 mHz and 100 kHz. It was noticeable that the degree of surface coverage by the occupation of inhibitor molecules (θ) was measured based on both polarization and EIS test results found in other research [23]. More details of electrochemical test details were reported in previous studies [26,27]. All corrosion tests were performed at a temperature of 25 ºC. In addition, to investigate the corroded surfaces, field emission scanning electron microscopy (FE-SEM) images were utilized.

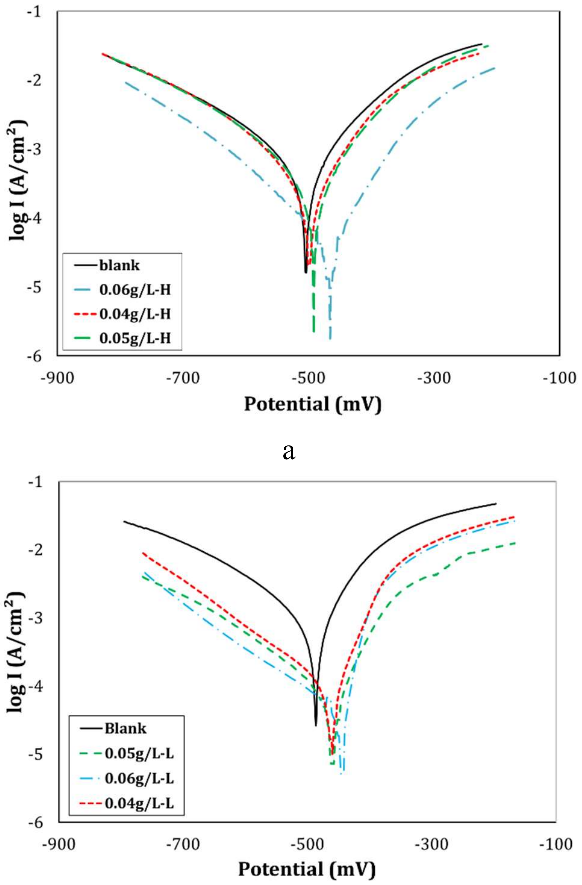
Table 1. Details of utilized specimens

Sample name	Inhibitor concentration (g/L)	Extracted protein type as bio-inhibitor	Corrosive solution type (1 M)
Blank	0	-	H_2SO_4, HCl
0.04 g/L-H	0.04	*Hyalomma* tick	HCl
0.05 g/L-H	0.05	*Hyalomma* tick	HCl
0.06 g/L-H	0.06	*Hyalomma* tick	HCl
0.04 g/L-L	0.04	leech	H_2SO_4
0.05 g/L-L	0.05	leech	H_2SO_4
0.06 g/L-L	0.06	leech	H_2SO_4

3. RESULTS AND DISCUSSION

Polarization curves were employed to investigate the corrosion rate of carbon steel substrates in corrosive solutions in the presence of bio-inhibitors and are shown in Figure 2. Besides, Table 2 represents data extracted from polarization results containing corrosion potentials (E_{corr}), corrosion current densities or corrosion rates (i_{corr}), cathodic Tafel slopes (β_{c}), and anodic Tafel slopes (β_{a}). In the presence of both bio-inhibitors, the values of E_{corr} reduced insignificantly compared to blank specimens. Since this reduction was less than 80 mv, both bio-inhibitors could act as a mixed inhibitor. Similar reports were shown by other research [12,23]. In this situation, rates of both anodic and cathodic corrosion reactions were affected. It was noticeable that the iron atoms dissolution was the anodic reaction and the hydrogen evolution was the cathodic reaction. It was found that when the value of E_{corr} was less negative the thermo-dynamical tendency to corrosion reactions was low [13,22]. Thus, the tendency of carbon steel substrates to corrosion reaction in acidic solutions decreased thermodynamically in the presence of both bio-inhibitors.

In addition, both cathodic and anodic Tafel slopes values were reduced in the presence of bio-inhibitors in utilized corrosive solutions. This suggested that the inhibitor molecules adsorbed on steel surfaces and could act as barriers to reach corrosive ions toward substrates. It was reported that O and N elements in the chemical composition of bio-inhibitors were the most important adsorption center to adsorb and interact with the metal surface [12]. In corrosive solutions, corrosion current densities of steel carbon substrates decreased when bio-inhibitors were added to solutions. When the corrosion rates decreased by the presence of inhibitors it showed that the attack of corrosive ions on the steel substrates was hindered [12].



a

b

Figure 2. Polarization curves in (a) the HCl solution, (b) the H_2SO_4 without and with the presence of bio-inhibitors

Figure 3 illustrates the inhibition efficiency (θ) versus the inhibitor concentration for both utilized bio-inhibitors. As shown in Figure 3, when the *Hyalomma* tick extract concentration in the HCl solution increased from 0.04 to 0.06 g/L the value of θ increased from 0.31-0.88. However, in the H_2SO_4 solution, the increase in θ values was more obvious in the presence of leech extract. The range of the θ values was about 0.93-0.97. The value of E_{corr} in the HCl solution was more negative than the H_2SO_4 solution for all specimens. Moreover, the corrosion rates of steel substrates in the HCl solution were higher than the other solution. Consequently, the inhibition efficiency of leech extract was more obvious than the other utilized bio-inhibitor in acidic solution; however, changes in the leech extract concentration had not a huge effect on the changes of inhibition efficiency.

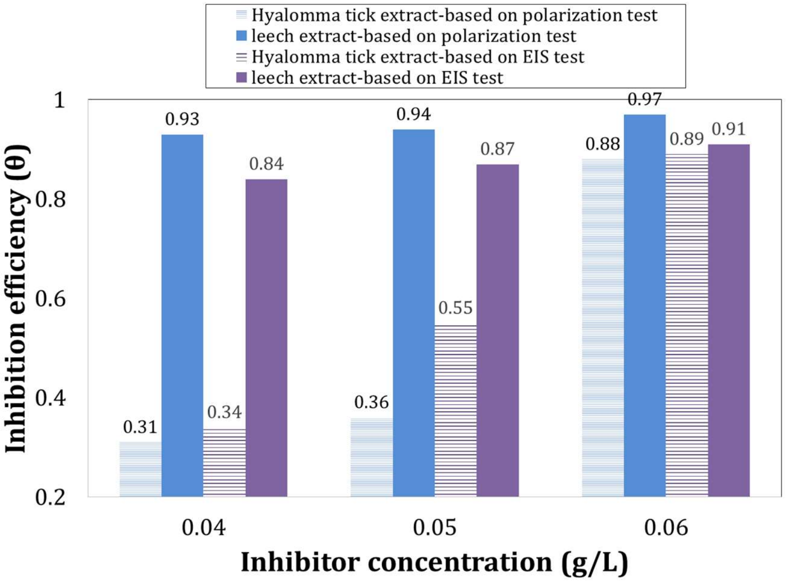


Figure 3. The plot of the inhibition efficiency (θ) versus the inhibitor concentration based on polarization and EIS tests

Table 2. Polarization data for (a) the HCl solution, (b) the H_2SO_4 solution without and with the presence bio-inhibitors

a

Sample name	i_{corr} (mA/cm^2)	E_{corr} (mV)	β_a (mV/dec)	β_c (mV/dec)
Blank	0.8659	-506.8	139.6	-213.9
0.04 g/L-H	0.5958	-501.0	129.0	-185.8
0.05 g/L-H	0.5650	-494.5	126.8	-190.9
0.06 g/L-H	0.1037	-468.4	87.1	-147.0

b

Sample name	i_{corr} (mA/cm^2)	E_{corr} (mV)	β_a (mV/dec)	β_c (mV/dec)
Blank	0.4201	-489.3	114.8	-221.8
0.04 g/L-L	0.1005	-463.2	54.0	-159.4
0.05 g/L-L	0.0758	-461.6	66.3	-155.1
0.06 g/L-L	0.0451	-447.6	30.6	-174.3

Figure 4 depicts the plot of corrosion rates versus the concentration of inhibitor for utilized bio-inhibitors in both acidic solutions. The decrease in the corrosion rate for carbon steel in the presence of *Hyalomma* tick extract obeyed a polynomial equation; however, when the leech extract concentration increased the corrosion rates of steel substrates in the H_2SO_4 solution reduced based on a logarithmic equation. This behavior showed that the adsorption mechanism of both inhibitors was different from each other.

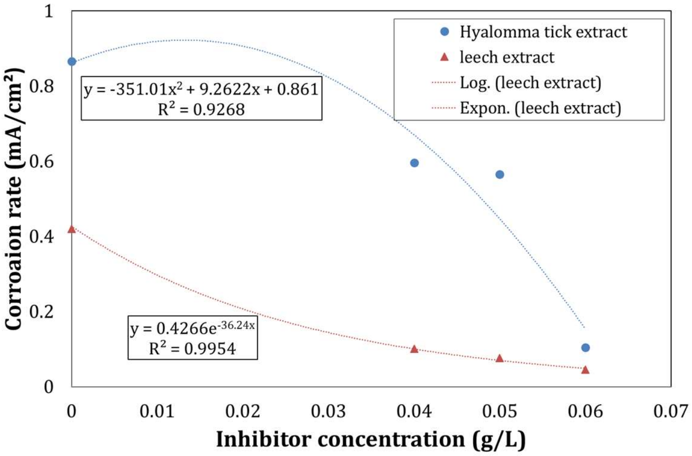


Figure 4. The plot of corrosion rates versus the inhibitor concentration for utilized bio-inhibitors

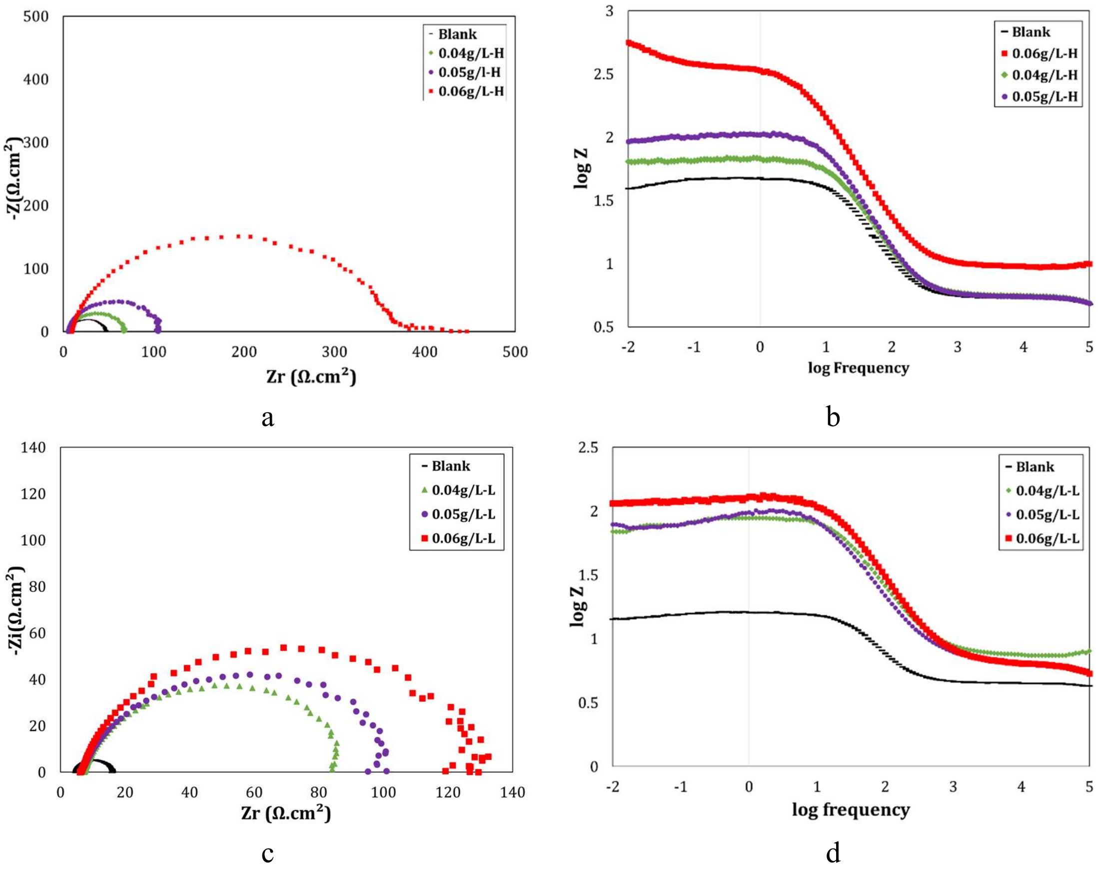
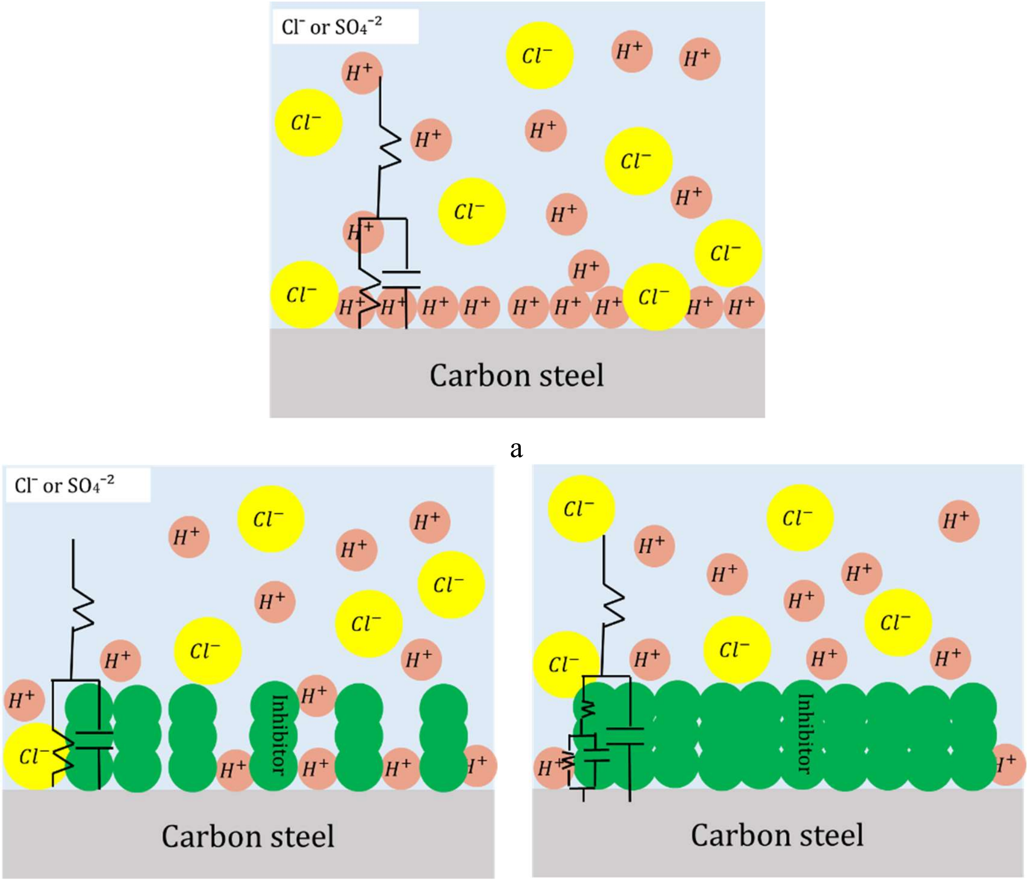


Figure 5. (a) Nyquist plot, and (b) Bode plot in the HCl solution (c) Nyquist plot, and (d) Bode plot in the H_2SO_4 solution

For EIS measurements, both Nyquist and Bode plots for various specimens are shown in Figure 5. For the HCl solution, when the *Hyalomma* tick extract concentration was 0.06g/L the shape of Nyquist and Bode plots changed compared to other conditions. However, the presence of leech extract as a bio-inhibitor with various concentrations did not change the shape of Nyquist and Bode plots. Totally, such utilized bio-inhibitors increased the impedance significantly in acidic solutions. For more details, the suggested electrical circuits were designed by Z-view software. The related circuits are presented in Figure 5 and the extracted data are reported in Table 3.

As shown in Figure 6(a), when the inhibitor molecules were not in the solution, the corrosive ions of hydrogen attacked the metallic surface without any obstacles for diffusion and adsorption on the steel surface. In this situation, the impedance for steel corrosion was low. Thus, the suggested electrical circuit contained the electrolyte solution (R_s), the constant phase angle element of double layers (CPE_{dl}), and the charge transfer resistance (R_{ct}). It was reported that the surface roughness affected the values of resistance and capacitance of double layers [28]. Similar behavior was also reported for carbon steel substrates in other corrosive solutions [29].



a

b

c

Figure 6. Suggested equivalent circuits for (a) blank specimen, (b) one-time constant in the presence of inhibitor and (b) two-time constant circuit in the presence of 0.06 g/L *Hyalomma* tick extract

When the bio-inhibitors were added to acidic solutions, molecules of inhibitors adsorbed on the steel surfaces and acted as physical obstacles for the diffusion of hydrogen ions. Therefore, as shown in Figure 6(b), the lower content of hydrogen ions could participate in the cathodic reaction. In addition, when the concentration of inhibitor increased in the electrolyte, such obstacles and the charge transfer resistance increased. This showed that bio-inhibitor molecules physically adsorbed on the steel surfaces [30]. However, when the *Hyalomma* tick extract concentration was 0.06g/L, a film of inhibitor with a continuous form could form on the steel substrate and acted as a barrier film. In this situation, other elements added to the suggested circuit, as illustrated in Figure 6(c). These elements consisted of a constant phase angle element of the formed film (CPE_f) and the film resistance (R_f).

By utilizing the Z-view software values of different elements in electrical circuits were extracted and reported in Table 3.

The presence of an inhibitor in acidic solutions had no significant effect on the solution resistance. However, the values of R_{ct} increased obviously in the presence of bio-inhibitors. Thus, as shown in Figure 3, inhibition efficiencies were about 0.34-0.89 in the HCl solution and were about 0.84-0.91 in the H_2SO_4 solution.

Table 3. Impedance parameters in (a) the HCl solution, and (b) the H_2SO_4 solution with and without inhibitor

a

Sample name	R_s (Ωcm^2)	R_{ct} (Ωcm^2)	CPE_{dl} ($mFcm^{-2}$)	n	R_f (Ωcm^2)	CPE_f ($mFcm^{-2}$)	n
Blank	5.21	40.51	0.29	0.91	-	-	-
0.04 g/L-H	5.47	61.58	0.22	0.93	-	-	-
0.05 g/L-H	5.44	97.67	0.10	0.93	-	-	-
0.06 g/L-H	6.48	383.7	0.15	0.95	33.8	22.4	0.89

b

Sample name	R_s (Ωcm^2)	R_{ct} (Ωcm^2)	CPE_{dl} ($mFcm^{-2}$)	n
Blank	4.50	11.20	0.45	0.91
0.04 g/L-L	7.44	71.02	0.11	0.92
0.05 g/L-L	6.41	83.47	0.10	0.91
0.06 g/L-L	6.29	118.6	0.01	0.94

Consequently, similar to polarization test results, the inhibition performance of leech extract was higher than the *Hyalomma* tick extract. It was noticeable that changes in the leech extract concentration had no significant alternation in increasing inhibition performance, compared to the *Hyalomma* tick extract as an inhibitor. In the presence of bio-inhibitor molecules in the corrosive solution the value of C_{dl} decreased and this event depicted the better corrosion resistance of steel substrates. Besides, by increasing the concentration of inhibitor, the reduction in values of CPE_{dl} increased. It was reported that the C_{dl} affected by various parameters such as the chemical composition and structure of inhibitor molecules, the temperature, the surface condition of metallic surfaces, and the potential of metal/solution interface [19,31]. Totally, the replacement of inhibitor molecules could decrease the dielectric constant of double layers and values of CPE_{dl}. In addition, an increase in the thickness of the double layers lowered the values of CPE_{dl} [18].

The nature of interactions between the surface of metals and inhibitor molecules would be explained with adsorption isotherms. Thus, there are various known adsorption isotherms such as Frumkin, Freundlich, Langmuir, Langmuir-Freundlich, Temkin, Flory-Huggins, Brunauer–Emmett–Teller (BET), El-Awady, and Bockris-Swinkels [2,11,32]. These isotherms would be changed when temperature, electrolyte type, adsorption mode, chemical characteristics, and electronic properties of utilized inhibitor, type of applied substrates, and metal surface charge were different [12].

Figure 7 shows the adsorption mechanism of utilized bio-inhibitors. These mechanisms were the Frumkin and the Langmuir adsorption isotherm for *Hyalomma* tick extract and leech extract, respectively. It was noticeable that θ values were obtained from both polarization and EIS test results. As depicted in Figure 7, the value of the correlation coefficient (R^2) for all plots was closed to 1.

It was found that for the Frumkin isotherm, the plot of log (θ/ (1-θ) C) versus θ should be linear [2,16] and the related equation is reported as follows

$$\log [(\theta/1-\theta)C]= \log K_{\mathrm{ads}} +2a\theta \tag{1}$$

where a is the coefficient of interaction, C is the inhibitor concentration, and K_{ads} is the equilibrium constant for the desorption-adsorption process. It was found that when the value of K_{ads} was high the adsorption of molecules to the surface has occurred more than the desorption process [2]. In addition, when a is positive it shows the attraction process and when the repulsion of interaction is dominant a is negative. Besides, it is zero when there is no interaction between the adsorbent species [16]. Thus, based on Figure 7(a), the value of a was positive and indicated that there was an attraction between the *Hyalomma* tick extract molecules. The scheme behavior of molecules for the Frumkin adsorption isotherm is shown in Figure 8(a). Similar to the obtained result of this paper, it was found that the adsorption of

Imidazole derivatives molecules on the aluminum surface in 0.5 M HCl solution was also Frumkin isotherm [33].

For the Langmuir adsorption isotherm, equation (2) is obtained [12]

$$\theta /1\text{-}\theta = K_{\text{ads}}C \qquad (2)$$

Due to Figure 7(b), the adsorption isotherm for leech extract molecules obeyed the Langmuir mechanism. In this isotherm, it is supposed that each site of the metal surface would be occupied by one molecule of adsorbent [2,4]. Thus, a monolayer of adsorbent would be formed on the surface of the metal, as shown in Figure 8(b). This mechanism is known as an ideal behavior for inhibitor molecules [35]. In addition, there was no interaction between the adsorbent and the metal surface. It was reported that the adsorption of *Marigold* flower extract compound on the mild steel in 0.5 M H_2SO_4 solution was the Longmuir isotherm [10].

The values of K_{ads} would be calculated through equations (1) and (2). Table 4 shows such measurements based on both electrochemical test results. The values of K_{ads} for the *Hyalomma* tick extract were higher than the leech extract. In addition, the free energy of standard adsorption ($\Delta G^{o}_{\text{ads}}$) would be measured by equations (3) [12,23]:

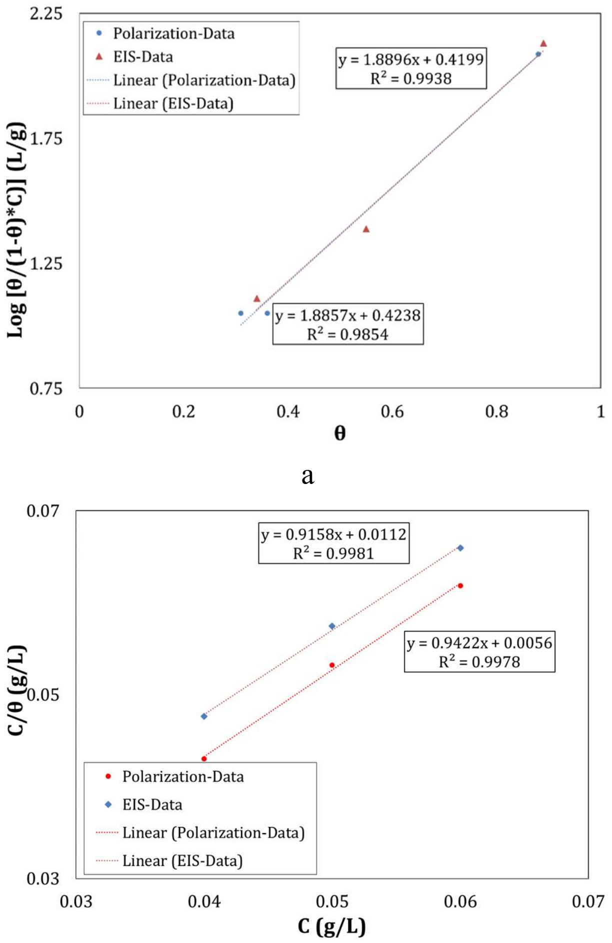
$$\Delta G^{o}_{\text{ads}} = -RT\ln\,[1000\ K_{\text{ads}}] \qquad (3)$$

Where R is the universal gas constant, and T is the absolute temperature. Measured values are reported in Table 4. The values of $\Delta G^{o}_{\text{ads}}$ for the adsorption of both bio-inhibitors were negative. This event showed a spontaneous reaction. Since these values were lower than -40 kJ /mole, it can be concluded that both bio-inhibitor molecules had the physical adsorption on carbon steel substrates. It was found that the lower value of ΔG^{o}_{ads} illustrated a physical adsorption event. In this situation, an electrostatic interaction has occurred between charged centers of inhibitor molecules and the charged metal surface [6,34]. Therefore, the formed layer of adsorption molecules of inhibitor had a weak bond (e.g. Van der Waals bonds) to the metal surfaces [2]. In addition, the value of $\Delta G^{o}_{\text{ads}}$ for the *Hyalomma* tick extract molecules was more negative than values for the leech extract. Thus, the value of ΔG^{o}_{ads} for the Frumkin adsorption isotherm was more negative than the Langmuir isotherm.

Table 4. Mesurments of kinetical parameters

Based on EIS test results			
Hyalomma tick extract		Leech extract	
K_{ad} (L/g)	$\Delta G^{o}_{\text{ads}}$(kJ/mol)	K_{ad} (L/g)	$\Delta G^{o}_{\text{ads}}$(kJ/mol)
2.63	-19.51	1.09	-17.33
Based on polarization test results			
Hyalomma tick extract		Leech extract	
K_{ad} (L/g)	$\Delta G^{o}_{\text{ads}}$(kJ/mol)	K_{ad} (L/g)	$\Delta G^{o}_{\text{ads}}$(kJ/mol)
2.65	-19.53	1.06	-17.26

Figure 9 shows FE-SEM images of corroded surfaces after 12 h immersion for some specimens. There were pores and cracks on all corroded surfaces of specimens which were the results of corrosion reactions. In the presence of inhibitor in acidic solutions, the number of defects and discontinuities decreased on surfaces of specimens. In addition, the size of pores lowered significantly. A similar trend was also observed in other research [23].



a

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Figure 7. (a) The Frumkin adsorption isotherm of *Hyalomma* tick extract, and (b) the Langmuir adsorption isotherm of leech extract

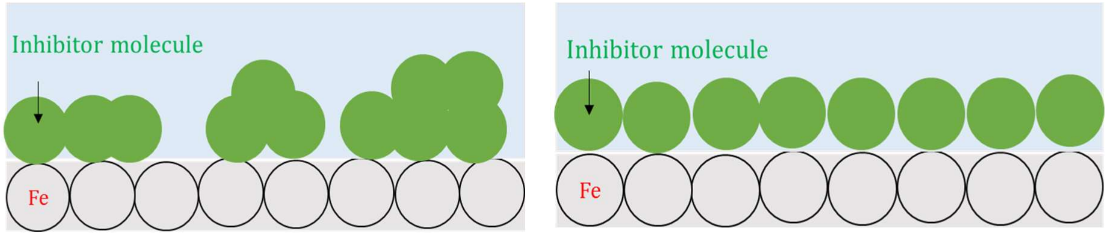

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Figure 8. The scheme behavior of molecules for (a) the Frumkin, and (b) the Langmuir adsorption isotherm

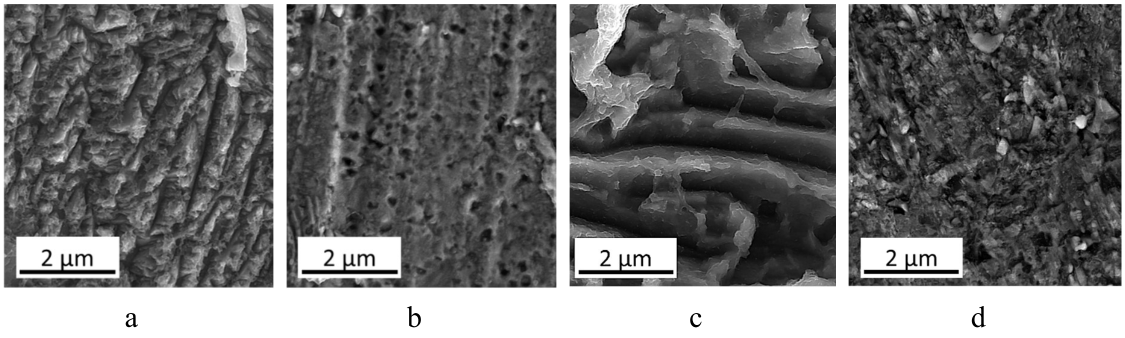

Figure 9. FE-SEM images of corroded surfaces after 12 h immersion for (a) Blank, (b) 0.06 g/L-H in HCl solution and (c) Blank, and (b) 0.06 g/L-L in H_2SO_4 solution

4. CONCLUSIONS

The inhibitive effect of the protein extracted from the *Hyalomma* tick and leech as bio-inhibitors with low concentrations in acidic solutions was studied. The obtained results were summarized as follows:

- Both electrochemical test results demonstrated that the selected bio-inhibitor exhibited an effective role to reduce significantly the corrosion rates of carbon steel in acidic solutions. Both bio-inhibitor acted as a mixed-type inhibitor for steel substrates. Besides, the values of ΔG_{ads} showed that both inhibitors adsorbed physically on the steel substrates in acidic solutions.
- Based on polarization test results for the leech extract, the inhibition efficiency was about 0.93-0.97 at low concentrations of inhibitor (0.04-0.06 g/L) in the H_2SO_4 solution. Moreover, the adsorption mechanism was the Langmuir model.
- When the concentration of the utilized inhibitor (the *Hyalomma* tick extract) was changed from 0.04 to 0.06 g/L, the inhibition efficiency increased from 0.34 to 0.88 in the HCl solution. In addition, the Frumkin adsorption isotherm was observed for this bio-inhibitor.

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