

Research Article

Interesting Behavior of *Pachycormus discolor* Leaves Ethanol Extract as a Corrosion Inhibitor of Carbon Steel in 1 M HCl: A Preliminary Study

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The purpose of this paper is to evaluate the inhibitive action of an ethanol extract from the leaves of *Pachycormus discolor* (EEP) on the corrosion of carbon steel in 1 M HCl at different temperatures; gravimetric method and electrochemical tests were conducted. Potentiodynamic polarization was used at room temperature and resistance polarization at different temperatures. Thermodynamic adsorption parameters are shown in order to demonstrate the spontaneous adsorption. The inhibition of EEP was performed via adsorption of the extract species on the carbon steel surface. The ethanol extract obeys Langmuir adsorption isotherm. Potentiodynamic polarization results indicate that the ethanol extract acts as a mixed type inhibitor. The results revealed that the inhibition efficiency (IE) of EEP increases (when the concentration is increased). Phytochemical and FTIR analyses are also presented in this work. It was found that the IE increases with the temperature in a 2.0% v/v solution which showed 94.52% IE at 25°C and 97.89% at 75°C.

1. Introduction

Acid pickling is an accepted treatment of a metallic surface to remove impurities, stains, and steel rust with an acid solution, such as hydrochloric acid (HCl) and sulphuric acid (H₂SO₄), usually before subsequent processing such as extrusion, rolling, painting, galvanizing or plating with zinc, aluminum, and so forth. However, inhibitor application is required since the corrosive media impairs the metal used [1].

Another important treatment is the well acidizing that is achieved by pumping hydrochloric acid into the well which is useful to dissolve limestone, dolomite, and calcite cement between the sediment grains of the reservoir rocks. In order to protect the integrity of the already completed well, inhibitor additives are introduced to the well to prohibit the well steel casing corroding with the acid.

Today the use of corrosion inhibitors have been expanded, they range from rare earth elements [2–4] to organic

compounds [5–9] since they are ecofriendly and harmless to the environment.

There are natural antioxidants used as organic corrosion inhibitor or extract from aromatic herbs, species, and medicinal plants [10, 11].

The purpose of the present work is to show an alternative corrosion inhibitor based on endemic plant of Baja California, Mexico that will control the corrosion problem and will not damage the environment due to its biodegradable properties.

2. Experimental Procedure

2.1. Preparation of Carbon Steel Specimen. Specimens of carbon steel 1018 (AISI) were mechanically cut into coupons of size 5 cm × 1.2 cm × 0.2 cm and were provided with a hole of a uniform diameter to facilitate the suspension of

the coupons in the test solution for the application of the weight loss method. For electrochemical studies, cylindrical carbon steel 1018 were used as a working electrode, with a 1 cm² area exposed and the rest being covered. The chemical composition of this steel is 0.15–0.20% C, 0.60–0.90% Mn, 0.04 (max) P, 0.05 (max) S and the remainder is Fe. The working electrode was polished with sand papers from 100 to 600 grades and subsequently washed with methanol and then stored in a desiccator [12]. Accurate weight of the samples was obtained using an electronic balance METTLER TOLEDO.

2.2. Preparation of the Plant Extract. The leaves of the *Pachycormus discolor* were taken, dried in a desiccator SECADOR, and then well grounded into powder. Then 70 gr of this dry plant material remains in maceration with n-hexane, three times, at a rate of 20 volumes of solvent per 1 gr mass of plant material, kept stirred at room temperature for at least 4 hours each time. Filtering was met on the three portions of the extract. The distillation of solvents leads to a waxy mass, which was discarded for the study of corrosion inhibition.

Immediately after, the same plant material was kept under stirring three times for 6 hours at room temperature (25°C) at a rate of 10 volume of Ethanol (80% v/v) per 1 gr of dry and shattered leaves in order to extract the active components. The three portions were mixed, and then most of the solvent was eliminated by azeotropic distillation of ethanol at reduced pressure, prolonging the necessary distillation and/or increasing depression as possible. Do not exceed 45°C.

Concentrated solutions of corrosion inhibitor were prepared from the ethanol extract and were added to 1 M HCl at the range of 0.005% to 2.0% (v/v).

2.3. Infrared Analysis. IR analysis was carried out using a PERKIN-ELMER100 FTIR spectrometer to determine the functional groups of the ethanol extract.

2.4. Phytochemical Screening. Phytochemical screening was carried out on the ethanol extract of the leaves of *Pachycormus discolor* [13]. The plant extract were screened for various compounds.

2.5. Weight Loss Method. The polished and preweighed carbon steel coupons of uniform size were tied with threads and suspended in 50 mL test solutions (in duplicate), with and without the inhibitor at 1% and 2% (v/v) concentrations for various time intervals. The coupons were washed, dried, and weighed. Then the weight loss was calculated.

The parameters used in this study are the following:

- (1) Time: 1.5 h, 4.5 h, 6.5 h, 21 h, 48 h, 109 h.
- (2) Inhibitor concentration: 1%, 2% (v/v).

The corrosion rate of the coupon of carbon steel 1018 was determined according to the loss in weight as a function of time.

$$\rho_{\text{corr}} = \frac{m_1 - m_2}{At}, \quad (1)$$

Where, m_1 and m_2 are the weight before and after the corrosion, respectively. A is the total area of the coupon and t the corrosion time. From this data, inhibition efficiency (IE) was calculated.

The inhibition efficiency is given by the following:

$$\text{IE}(\%) = \left(\frac{\rho_{\text{corr}}^{\circ} - \rho_{\text{corr}}}{\rho_{\text{corr}}^{\circ}} \right) \times 100, \quad (2)$$

where, $\rho_{\text{corr}}^{\circ}$ is the corrosion rate without inhibitor. ρ_{corr} is the corrosion rate with inhibitor.

2.6. Electrochemical Measurements. The electrochemical analysis was performed using a three electrode system [14]. The carbon steel working electrode, saturated calomel (SCE) reference electrode, and high purity graphite counter electrodes were used in the corrosion cell. Measurements were performed using a Gamry Instrument Potentiostat/Galvanostat/ZRA PC 4/300 and DC 105 Corrosion Technique software.

The working electrode was polished with 120 to 600 grit sand paper, washed with methanol, and dried. Instead of a salt bridge, a Luggin capillary arrangement was used to keep SCE on the working electrode to avoid the ohmic contribution. The polarization plots were obtained 10 minutes after the working electrode was immersed in the solution using Tafel Technique at room temperature (23°C). Applying a scan rate of 1 mV/s in a range from –250 mV to +250 mV versus corrosion potential (E_{corr}) of the working electrode measured against SCE.

The inhibition efficiency was calculated using the formula [15]

$$\text{IE}(\%) = \left(\frac{I_{\text{corr}} - I_{\text{corr}}^*}{I_{\text{corr}}} \right) \times 100, \quad (3)$$

where I_{corr} is corrosion current without inhibitor. I_{corr}^* is corrosion current with inhibitor.

Values of linear polarization resistance (R_p) were obtained using the polarization resistance technique at 23°C, 40°C, 60°C, and 75°C. The scan rate was 0.125 mV/s in a range from –0.20 to 0.20 mV versus corrosion potential (E_{corr}) of the working electrode measured against SCE.

The inhibition efficiency was calculated using the formula [16]

$$\text{IE}(\%) = \left(\frac{Rp_{\text{inh}} - Rp_{\text{blank}}}{Rp_{\text{inh}}} \right) \times 100, \quad (4)$$

where Rp_{inh} is the polarization resistance in the presence of inhibitor at each temperature. Rp_{blank} is the polarization resistance in the absence of inhibitor at each temperature.

The gravimetric and electrochemical corrosion tests were conducted in accordance with the practices recommended in ASTM standards G3 and G31 [17, 18].

3. Results and Discussion

3.1. Weight Loss Studies. The inhibitor was tested at two different concentrations 1% and 2% (v/v); their corresponding

TABLE 1: Corrosion rates as a function of immersion time with and without *Pachycormus discolor* ethanol extract.

Inhibitor concentration % (v/v)	Immersion time (hr)					
	Corrosion rates ($\text{mg} \cdot \text{cm}^{-2} \cdot \text{hr}^{-1}$)					
	1.5	4.5	6.5	21	48	109
Blank	0.78	0.51	0.57	0.57	0.48	0.57
1.0	0.47	0.17	0.14	0.08	0.06	0.05
2.0	0.18	0.12	0.09	0.04	0.03	0.03

TABLE 2: Inhibition efficiency as a function of immersion time and concentration of *Pachycormus discolor* ethanol extract.

Inhibitor concentration % (v/v)	Immersion time (hr)					
	Inhibition efficiency (%)					
	1.5	4.5	6.5	21	48	109
1.0	39.59	66.09	74.34	84.61	86.59	89.88
2.0	76.07	77.40	83.79	92.47	93.50	93.50

corrosion rates and inhibition efficiencies are presented in Tables 1 and 2, respectively. The corrosion rate decreases as the inhibitor concentration increases.

The corrosion rate diminishes 6 to 9 times after about 109 hours of immersion time, and the data are shown in Table 1.

The addition of inhibitors increases the IE, irrespective of the time of immersion. This may be due to the adsorption of phytochemical constituents of the extracts on the metal surface. The results related with the effect of time of different inhibitor concentrations for carbon steel 1018 immersed in 1 M HCl are also shown in Table 2. The inhibition efficiency is higher when the immersion time is increasing. The highest efficiency (93.50%) was obtained with the 2% v/v of inhibitor concentration after 109 hr testing.

3.2. Polarization Studies. The effect of the inhibitor concentration is shown in Figure 1 which presents the anodic and cathodic potentiodynamic polarization measurements of carbon steel 1018 in 1 M HCl solution at room temperature with EEPD. The electrochemical corrosion parameters: current density (i_{corr}), cathodic and anodic Tafel slopes (β_c and β_a), and the percentage efficiency (IE) for the corrosion inhibition of carbon steel 1018 in 1 M HCl at room temperature at different concentrations of the plant extract are given in Table 3.

Polarization studies revealed that the corrosion current density (i_{corr}) decreased noticeably with the addition of the extract. Further, there was an anodic shift of the E_{corr} value from -471 mV (blank) to -432 mV at 2% v/v indicating that the *Pachycormus discolor* leaves extracts acted as an anodic inhibitor [19] for carbon steel 1018 in 1 M HCl, which was supported by the gradual and significant decrease of the anodic Tafel slope, β_a is 113.9 mV/decade of blank to 71.0 mV/decade at 2% v/v. In lower inhibitor concentrations, the values of the cathodic Tafel slopes were decreasing too, though not markedly. This means that the extract must have acted by blocking anodic sites and also cathodic site to some extent, and the extract contained the active molecules which behaved as mixed type of inhibition.

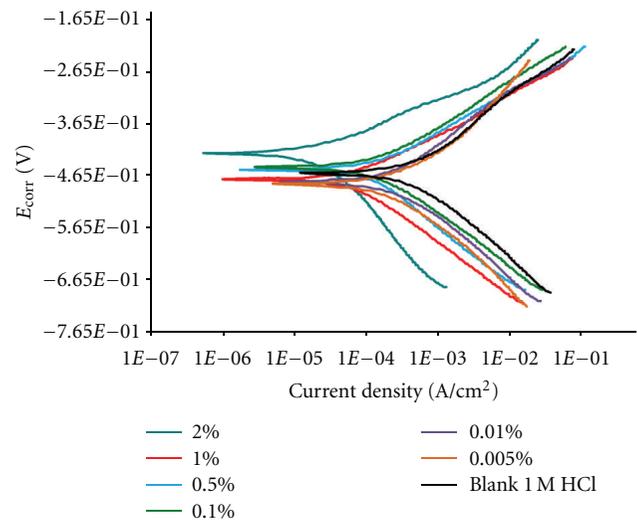
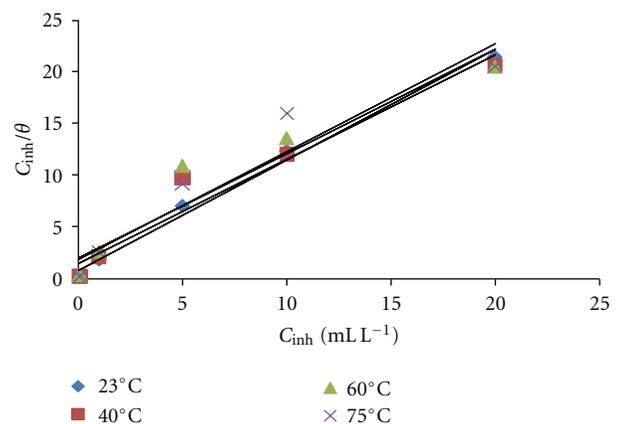
FIGURE 1: Anodic and cathodic plots of carbon steel 1018 in 1 M HCl solution in the absence and presence of various concentrations of *Pachycormus discolor* ethanol extract.

FIGURE 2: Langmuir adsorption isotherm of EEPD on carbon steel 1018 surface in 1 M HCl.

TABLE 3: Polarization parameters for carbon steel in 1 M HCl at room temperature containing various concentrations of *Pachycormus discolor* ethanol extract.

Concentration (v/v) %	E_{corr} (mV/SCE)	β_c (mV/dec)	β_a (mV/dec)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	IE %
Blank	-471.0	125.8	113.9	442.0	—
0.005	-495.0	136.7	129.1	284.0	35.7
0.001	-490.0	116.9	111.0	237.0	46
0.1 0	-492.0	108.3	94	139	68.55
0.5 0	-465.0	119.2	80.9	114.0	74.20
1.0	-484.0	108.8	78.4	69.20	84.34
2.0	-432.0	167.5	71.0	24.20	94.52

TABLE 4: Polarization resistance values for carbon steel in 1 M HCl at different temperatures obtained by electrochemical measurements in the absence and presence of EEPD in various concentrations.

Inhibitor concentration (%) v/v	Temperature ($^{\circ}\text{C}$)	Polarization resistance (Ω)	Inhibition efficiency (%)
Blank	23	43.865	
	40	17	
	60	5.16	
	75	2.067	
0.005	23	68.22	35.70
	40	21.03	19.14
	60	6.008	14.11
	75	2.989	30.86
0.01	23	85.60	48.70
	40	31.07	45.28
	60	7.346	29.75
	75	3.174	34.87
0.1	23	99.88	56.08
	40	32.19	47.18
	60	8.353	38.22
	75	3.395	39.11
0.5	23	153.6	71.44
	40	34.86	51.23
	60	9.527	45.83
	75	4.591	54.97
1.0	23	236.2	81.42
	40	101.4	83.23
	60	19.54	73.59
	75	5.56	62.82
2.0	23	620.2	92.95
	40	574.4	97.00
	60	148.4	96.52
	75	115.5	97.89

3.3. *Effect of Temperature.* The effect of the temperature on the corrosion rate of carbon steel 1018 in the absence and in the presence of various inhibitors concentrations was studied at a temperature range from 23 $^{\circ}\text{C}$ to 75 $^{\circ}\text{C}$, using the polarization resistance electrochemical measurement (Table 4). It was found that the polarization resistance in

the absence and in the presence of inhibitor decreases with the increase in temperature, but the polarization resistance value is much greater for an inhibited acid solution than the uninhibited acid solution.

Regarding the 2% v/v inhibitor concentration, it shows an interesting behavior since the values of polarization

TABLE 5: Adsorption parameters for EEPD in 1 M HCl obtained from Langmuir adsorption isotherms at different temperatures.

Temperature T ($^{\circ}\text{C}$)	Adsorption parameters		
	slope	K (mL^{-1}L)	r^2
23	1.04	1.46	0.9930
40	1.01	0.72	0.9539
60	1.01	0.51	0.9230
75	1.05	0.58	0.9230

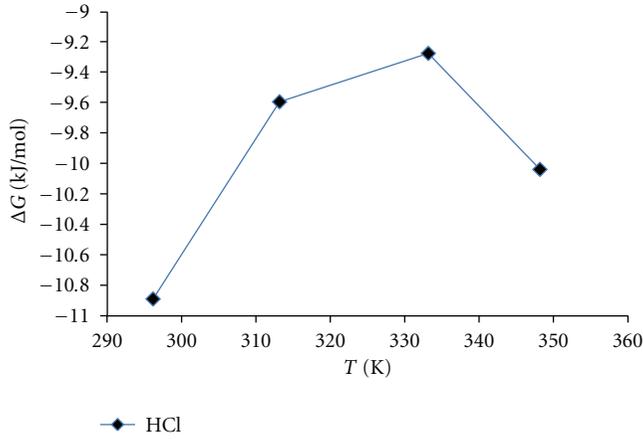


FIGURE 3: The variation of ΔG_{ads} with T .

resistance decrease with an increase in the temperature, though, it is not remarkable; actually the IE increased because the polarization resistance values are much higher for the uninhibited solution at 2% v/v than the uninhibited acid solution. The IE at 75 $^{\circ}\text{C}$ is 97.89%.

The effect of temperature on the inhibited acid metal reaction is highly complex because many changes occur on the metal surface such as rapid etching and desorption of inhibitor and the inhibitor itself may undergo decomposition and/or rearrangement. However, it was found that the few inhibitors with acid metal systems have specific reactions which are as effective at high temperature as they are at low temperature [20–23].

3.4. Adsorption Isotherms. The primary step in the mechanism of action of the inhibitors in acid solutions is generally the adsorption on the metal surface, which is usually oxide free in acid solutions. Bockris states that [24] the adsorption of an organic substance onto the metal surface can be expressed in the following exchanger reaction:



where n is the number of water molecules displaced of the metal surface by one molecule of the adsorbed inhibitor; the value of n depends on the transversal section of the organic molecule area with respect to the water molecule. The adsorption of the organic molecules occurs because the interaction between the energy on the metal surface and the inhibitor is greater than the interaction of the energy on the metal

surface and the water molecules. When the equilibrium of the process described in (5) is reached, it is possible to obtain diverse expressions of the adsorption isotherm plots, and thus, the degree of surface coverage ($\theta = \text{IE}(\%)/100$) can be plotted as a function of the inhibitor concentration under test.

From Table 4, it can be concluded that θ increases with the inhibitor concentration; this is attributed to more adsorption of inhibitor molecules onto the carbon steel surface. The surface coverage values (θ) for different concentrations of the inhibitor in an acid medium have been evaluated from the polarization resistance data. The data was plotted graphically to obtain a suitable adsorption isotherm (Figure 2). The Langmuir adsorption isotherm [25] was applied to analyze its mechanism by the following:

$$\frac{C}{\theta} = \frac{1}{K} + C, \quad (6)$$

where C is the inhibitor concentration in mL L^{-1} in the electrolyte, and K (mL^{-1}L) is the equilibrium constant for the adsorption/desorption process.

The adsorption parameters from Langmuir adsorption isotherms are estimated and given in Table 5. It indicates that the inhibitor obeys the Langmuir model since the experimental data presents adequate curve fittings for the applied adsorption isotherms; the correlation coefficients (r^2) were in the range: $0.993 \geq r^2 \geq 0.923$ and also the slopes tend to be close to a unit.

The K value demonstrates that it is affected by the temperature; when the temperature increases from 23 $^{\circ}\text{C}$ to 40 $^{\circ}\text{C}$, K decreases as a result, on the other hand, when the temperature increased from 65 to 75, the value of K increased as well; however, the value K that is obtained at room temperature (23 $^{\circ}\text{C}$) is much greater than the values at other temperatures. This means that the strength of adsorption is higher.

3.5. Thermodynamic Adsorption Isotherms. Thermodynamic adsorption data, such as change in free energy (ΔG_{ads}), enthalpy of adsorption (ΔH_{ads}), and the entropy of adsorption (ΔS_{ads}), can be calculated depending on the estimated values of K from adsorption isotherms [26] at different temperatures as follows.

The ΔG_{ads} values at all studied temperatures can be calculated from the following [27]:

$$K = \frac{1}{C_{\text{H}_2\text{O}}} \exp\left(\frac{\Delta G_{\text{ads}}}{RT}\right), \quad (7)$$

where $C_{\text{H}_2\text{O}}$ is the concentration of water molecules (in mL L^{-1}) at the metal/solution interface. Then, the ΔG_{ads} obtained values are plotted versus T (Figure 3) in accordance with the basic equation [28]:

$$\Delta G_{\text{ads}} = \Delta H_{\text{ads}} - T\Delta S_{\text{ads}}. \quad (8)$$

A straight line of interception represents the ΔH_{ads} values. By introducing the values in (8), the ΔS_{ads} values are calculated at all studied temperatures. As it can be observed in Figure 3,

TABLE 6: Thermodynamic adsorption parameters for EEPD on carbon steel 1018 in 1 M HCl at different temperatures.

T ($^{\circ}\text{C}$)	Thermodynamics adsorption parameters				
	ΔG (kJ mol^{-1})	ΔH (kJ mol^{-1})		ΔS (kJ mol^{-1})	
23	-10.8896	-36.649		-0.1595	
40	-9.5931	-36.649	-14.576	-0.1477	-0.07722
60	-9.2747	-14.576	7.6593	-0.0716	-0.00485
75	-10.0375	7.6593		-0.0068	

TABLE 7: Phytochemical screening of EEPD.

Compounds of functional group	Presence	Compounds of functional group	Presence
Alkaloids	+	Reducing sugars	+
Steroidal saponins	+	Quinones and Lactones	+
Phenols and or/tannins	+	Amines	-

a segmented straight line of three opposite slopes was obtained in 1 M HCl indicating the existence of three sets of adsorption sites with different energetic enthalpies of adsorption, leading to the occurrence of a comprehensive adsorption.

The negative values of ΔG_{ads} indicate that the adsorption of EEPD on the carbon steel 1018 surface is an spontaneous process [29, 30].

The most negative value of ΔG_{ads} occurs at room temperature (23°C); furthermore, the negative value of ΔH_{ads} indicates that the adsorption of the inhibitor's molecules is an exothermic reaction.

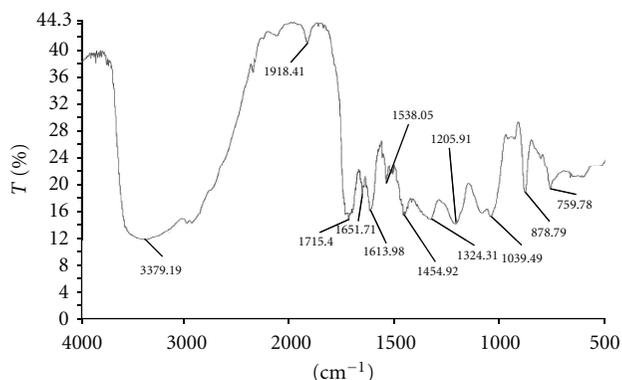
In addition, ΔG_{ads} decreases (becomes more negative) with an increasing temperature in the range from 60°C to 75°C ; it also shows the positive value of ΔH_{ads} , indicating the occurrence of an endothermic process at which increasing the temperature facilitates the adsorption of the molecules.

The negative values of ΔS_{ads} (Table 6) are accompanied with the exothermic adsorption process, which agrees with the expected event; when the adsorption is an exothermic process, it must be accompanied by a decrease (becomes more negative) in the entropy change [31].

Moreover, the value of the adsorption heat (ΔH_{ads}) gives valuable information about the mechanism of the inhibitor adsorption. According to the listed values of ΔH_{ads} (Table 6) it can be explained by the following.

Both endothermic ($\Delta H_{\text{ads}} = 7.6593 \text{ kJ mol}^{-1}$) and exothermic ($\Delta H_{\text{ad}} = -36.649$) adsorption behaviors were determined depending on the exact range of the applied temperatures. The average of ΔH_{ads} is about $-22.15 \text{ kJ mol}^{-1}$ which is larger than the common physical adsorption heat, but smaller than the common chemical adsorption heat [32]; it probably indicates that both physical and chemical adsorption took place.

3.6. Infrared Analysis. The IR spectra (Figure 4) of the extract of *Pachycormus discolor* contain bands which correspond to: phenolic hydroxyl groups (3379.39 cm^{-1}); carbonyl groups (1715 cm^{-1}), aromatic C=C bending (1613 cm^{-1}), =C-H out of plane bending (878.79 and 750.78 cm^{-1}), two bands

FIGURE 4: IR spectra of ethanol extract of *Pachycormus discolor*.

appear for the C-O stretching vibrations in esters in 1324 and 1205 cm^{-1} .

3.7. Phytochemical Screening. Phytochemical screening was carried out on the ethanol extract of the leaves of *Pachycormus discolor*. The plant extracts were screened for various compounds (Table 7).

Several papers that have been published show that compounds such as alkaloids may be responsible for the corrosion inhibitive effect in a hydrochloric acid medium [33, 34]. Other corrosion inhibitors [35, 36] contain lactones, saponins, polyphenols, and they are also effective.

In the present case, the mixture of the different compounds can be responsible for the corrosion inhibitive effect. There is no data about the main compound on the ethanol extract of the leaves of *Pachycormus discolor*.

4. Conclusions

- (i) The Ethanol extract of the leaves of *Pachycormus discolor* acts as a mixed type inhibitor for the corrosion of carbon steel 1018 in 1 M HCl.

- (ii) Weight loss studies, polarization studies, electrochemical and polarization resistance measurements are in good agreement.
- (iii) The effect of immersion time of EEPD showed maximum efficiency in 109 hours at room temperature at 2% v/v inhibitor concentration.
- (iv) The polarization resistance increases with the amount of the inhibitor.
- (v) The adsorption of *Pachycormus discolor* on the carbon steel 1018 surface in hydrochloric acid obeys the Langmuir adsorption isotherm model.
- (vi) The effect of temperature revealed that both physical and chemical adsorption take place.
- (vii) The negative value of free energy change of adsorption indicates that the adsorption of the inhibitor on carbon steel 1018 surface was spontaneous.
- (viii) The inhibition efficiency of EEPD at room temperature is greater than at higher temperatures, except at 2% v/v inhibitor concentration.

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