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Delonix Regia Leaf Extract as Environmental Friendly and Safe Corrosion Inhibitor for Carbon Steel in Aqueous Solutions

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Delonix regia leaf extract activity as a green corrosion inhibitor (environmental friendly) for carbon steel (CS) in 1M HCl has been studied using weight loss (WL), potentiodynamic polarization (PP), electrochemical frequency modulation (EFM) and electrochemical impedance spectroscopy (EIS). The weight loss results show that Delonix regia leaf extract is an excellent corrosion inhibitor. The inhibition efficiency (IE) increases with temperature from 25 to 45ºC, reaching a maximum value of 78.8 % at the highest concentration of 300 ppm at the temperature of 45ºC. Polarization measurements demonstrate that the Delonix regia leaf extract acts as a mixed type inhibitor. Nyquist plot illustrates that on increasing Delonix regia leaf extract dose, the charge transfer increases and the double layer capacitance decreases. The adsorption of Delonix regia leaf extract on CS obeys Temkin adsorption isotherm..

1. Introduction

Carbon steel is the extended metal used in industrial purpose, equipment, building and more in manufacturing of installations for petroleum, fertilizers and other industry. Protection of CS in aqueous solutions is universal request, conomical, environmental, and aesthetical important [1]. Use of inhibitor is more effective way to reduce corrosion. The organic compounds are widely used as corrosion inhibitors as such as O, N, P, S, and heavy metals. But the organic compounds are hazards and unfriendly environment inhibitors [2]. In spite of the broad spectrum of organic compounds available as corrosion inhibitors, there is increasing concern about the toxicity of most corrosion inhibitors because they are toxic to living organism and may also poison the earth. Organic corrosion inhibitor products are proven as effective, but they tend to be expensive also may be flammable, environmental risks and toxicity (inhalation and skin contact risk). There is accordingly a need for safer and more environmentally friendly corrosion inhibiting products that are also cost effective. These have prompted searches for green corrosion inhibitors. According to Eddy et al [3], green corrosion inhibitors have the advantage of being inexpensive, biodegradable, do not contain heavy metals (non-toxic compounds), and eco-friendly. The successful use of naturally occurring substances to inhibit the corrosion of metals in acidic and alkaline environment has been reported by some research groups [4]. Several researches have been carried out on the inhibition of

the corrosion of steel by some plant extracts. The successful use of naturally occurring substances to inhibit the corrosion of metals in both acidic and alkaline environments has been reported by some research groups [5-11] to mention but a few.

This research will discuss the "Delonix regia leaf extract" as green corrosion inhibitor which renewable source, friendly environmental acceptance, biodegradable, safer and cheaper than other green corrosion inhibitors for protecting CS in 1 M HCl. Hydrochloric acid is selected for present study, due to its wide industrial applications such as acid pickling, acid cleaning, water cooling and circulation or acid heat exchanger [12].

2. Experimental methods

2.1. Plant extract preparation

Delonix regia leaf was dried in the shade at room temperature and grind using electrical mill into fine powder. One g of powder Delonix regia leaf was dissolved in one liter bidistilled water by heating, cool at room temperature, then filtrate. 10 ml filtrate + 5 ml ethanol were put in condensate-collecting flask of rotary evaporator to determine concentration of dissolved substance in plant extract solution. The remained weight of dry substance in collection flask after evaporation the solvent equal the equivalent weight dissolved 10 ml filtrate. By this we can prepare stock plant extract solution with desired concentration [13].

2.2. Carbon steel samples

Chemical composition of CS was described below:

2.2. Test Solution

The corrosive media 1 M HCl solution were prepared for each experiment using analytical grade hydrochloric acid (37%) and diluted with bidistilled water.

The concentration range of Delonix regia leaf extract inhibitor was 50-300 ppm.

2.3. WL measurements

Similar CS coupons with dimensions (2×2×0.2) cm were abraded with emery paper (grade from 320 to 1200 grit sizes) and then washed with bidistilled water and acetone. After accurate weighting, the coupons were immersed in a beaker which contained 100 ml of HCl with and without addition of different concentrations of Delonix regia Leaf extract. Through period time of 30 min coupons were removed, washed, dried and re-weighted accurately through 3 hours at various temperatures from 25 to 45°C. The average WL of CS coupons could be obtained. %IE and the degree of surface coverage θ of Delonix regia Leaf for the corrosion of CS can be obtained from equation (1).

$$\%IE = \theta \times 100 = \left[1 - \frac{w}{w^\circ}\right] \times 100 \qquad (1)$$

Where W° and W are the values of the average weight losses without and with addition of the extract, respectively

2.4. Electrochemical measurement

Electrochemical cell with three electrodes was used. The working electrode was CS of surface

Elements	C	Cr	Ni	Si	Mn	P	S	Fe
Composition (weight %)	0.14	0.1	0.01	0.024	0.11	0.05	0.05	Rest

area of 1 cm². All potentials were given with reference to the saturated calomel electrode (SCE). The counter electrode was a platinum plate of surface area of 1 cm². The working electrode was immersed in the test solution for 30 min to reach the open circuit potential (the CS used in the polarization measurements was identical to that used in the WL measurements). Before each experiment, the electrode was abraded using emery papers as before. After this, the electrode was cleaned with ethyl alcohol or acetone and washed by bidistilled water.

Tafel polarization curves were determined by polarizing to ± 250 mV with respect to the free corrosion potential (vs. SCE) at a scan rate of 0.5 mV/s. Stern-Geary method used for the determination of corrosion current is performed by extrapolation of anodic and cathodic Tafel lines to a point which gives log i_{corr} and the corresponding corrosion potential (E_{corr}) were obtained. Then i_{corr} was used for calculation of % IE and θ as in following equation (2):

$$\% \text{ IE} = \theta \times 100 = [1 - (i_{corr(inh)} / i_{corr(free)})] \times 100$$

Where i_{corr} $_{(free)}$ and i_{corr} $_{(inh)}$ are the corrosion current densities in the absence and presence of inhibitor, respectively.

EIS measurements were carried out in frequency range from 0.3 Hz to 100 KHz with amplitude of 5 mV peak to peak using AC signals at open potential. The experimental impedance was analyzed and interpreted based on the equivalent circuit. The main parameters deduced from the analysis of Nyquist diagram are the charge transfer resistance R_{ct} (diameter of high-frequency loop) and the double layer capacity C_{dl}. The IE and the θ obtained from the EIS measurements are calculated from the following equation:

$$\% \text{IE} = \theta \text{x} 100 = [1 - (R°_{ct}/R_{ct})] \times 100 \qquad (3)$$

where $R°_{ct}$ and R_{ct} are the charge transfer resistances without and with extract, respectively.

Electrochemical frequency modulation (EFM) was carried out using two frequencies 2 and 5 Hz. The base frequency was 0.1 Hz, so the waveform repeats after 1 s. The higher frequency must be at least two times the lower one. The higher frequency must also be sufficiently slow that the charging of the double layer does not contribute to the current response. Often, 10 Hz is a reasonable limit. The Intermodulation spectra contain current responses assigned for harmonical and intermodulation current peaks. The large peaks were used to calculate the corrosion current density (i_{corr}), the Tafel slopes (β_a and β_c) and the causality factors CF-2, CF-3 [14].

All measurements were carried out using a using Gamry Potentiostat/Galvanostat/ZRA (model PCI4/750) with a Gamry framework system based on ESA400 measurements; computer was used for collecting data. Echem Analyst 6.03 Software was used for plotting, graphing and fitting data.

3. Results and Discussion

3.1 WL measurements

WL of CS was determined at various times interval in the absence and presence of different doses of Delonix regia leaf extract are shown in Fig. 1. The IE % values calculated are listed in Table 1, it is noted that the IE % increases steadily with increasing the concentration of extract and θ were calculated by equation (1). The observed IE of the Delonix regia leaf could be attributed to the adsorption of its components on CS surface forming protective layer, this layer isolates the metal surface from the corrosive medium and hence

decreasing the corrosion rate, with increasing efficiency as extract doses increase [15].

3.2.1. EIS measurements

The corrosion behavior of CS in 1 M HCl solution in the presence of Delonix regia leaf was investigated by EIS at 25°C after 30 min of immersion. Figure 2 shows the results of EIS experiments in the Nyquist representation. It is observed that
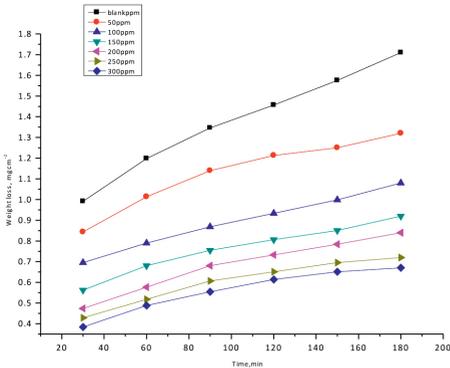
the curves approximated by a single capacitive semi-circles, showing that the corrosion process was mainly charge transfer controlled [16]. The general shape of the curves is very similar for all samples; the shape is maintained throughout
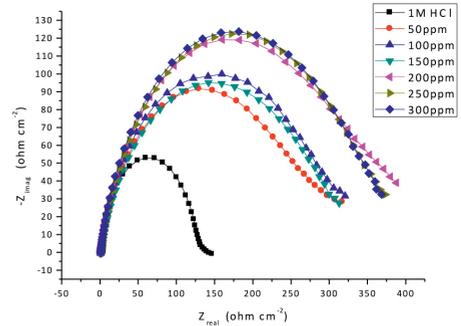

Fig. 2: Nyquist plots for CS in 1M HCl at different doses of Delonix regia leaf at 25°C
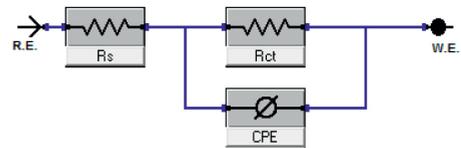

Fig. 3: Electrical equivalent circuit used to fit the EIS data

Fig. 1: WL-time curves for the corrosion of CS in 1 M HCl in the absence and presence of different doses of Delonix regia leaf at 25°C

Tab. 1: Corrosion rate (C.R.), surface coverage (θ) and IE obtained from WL measurements for CS in 1 M HCl solutions without and with various doses of Delonix regia leaf extract at 25°C

concentration ppm	Weight loss mg cm^{-2}	C.R., mg cm^{-2} min^{-1}	θ	%IE,
Blank	1.71	0.009	---	----
50	1.32	0.0073	0.228	22.8
100	1.08	0.006	0.368	36.8
150	0.92	0.0051	0.462	46.2
200	0.84	0.0047	0.509	50.9
250	0.72	0.004	0.579	57.9
300	0.67	0.0037	0.608	60.8

the whole concentrations, indicating that almost no change in the corrosion mechanism occurred due to the inhibitor addition [17].The diameter of Nyquist plots increases on increasing the Delonix regia leaf concentration due to inhibition behavior of Delonix regia leaf extract. The Nyquist plots are analyzed in terms of the equivalent circuit (Figure 3) composed with classic parallel capacitor and resistor. The impedance of a CPE is described by the Equation 4 [18]:

$$Z_{CPE} = Y_0^{-1} (j\omega)^{-n} \tag{4}$$

Where Y_0 is the magnitude of the CPE, j is an imaginary number, ω is the angular frequency at which the imaginary component of the impedance reaches its maximum values and n is the deviation of the CPE ($-1 \leq n \leq 1$). The values of the interfacial capacitance Cdl can be calculated from CPE parameter values Y_0 and n using Equation 5:

$$C_{dl} = Y (\omega_{max})^{n-1} \tag{5}$$

The impedance parameters including the resistance R_{ct}, double layer capacitance C_{dl} and IE are given in Table 2. Also, Bode plots for the CS in 1 M HCl solution are shown in Figure 4. In the high frequency limit to the electrolyte resistance represents (ohmic resistance),R_s, while the resistance at low frequency represents the sum of $(R_s + R_{ct})$, where R_{ct} is in the first approximation determined by both electrolytic conductance of the oxide film and the polarization resistance of the dissolution and repassivation process.

The main parameter deduced from the analysis of Nyquist diagram, resistance of charge transfer R_{ct} (diameter of high frequency loop). The capacity of double layer C_{dl} is defined as:

$$C_{dl} = \frac{1}{2\pi R_{ct} f_{max}} \tag{6}$$

Where f_{max} is the maximum frequency at which the Z_{imag} of the impedance is a maximum. The inhibition efficiency (IE) can be calculated from Equation 7:

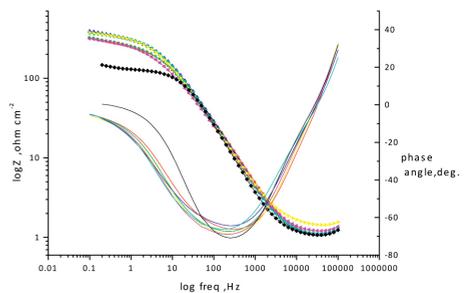
Fig.4: Bode plots for CS in 1M HCl solutions in the absence and presence of various Delonix regia leaf doses at 25ºC

Tab. 2: EIS data of CS in 1 M HCl in the presence of different doses of Delonix regia leaf extract at 25° C

Inh. conc., ppm	R_{ct}, Ωcm^2	C_{dl}, x10^{-4} F cm^{-2}	θ	%IE,
Blank	129.4	0.689	-----	-----
50	271.2	9.890	0.523	52.3
100	283.0	1.292	0.543	54.3
150	286.1	1.064	0.548	54.8
200	342.7	0.990	0.622	62.2
250	342.9	0.969	0.623	62.3
300	350.1	0.938	0.630	63.0

$$IE\% = \left(1 - \frac{R_{ct}^{\circ}}{R_{ct}}\right) \times 100 \qquad (7)$$

Where R°_{ct} and R_{ct} are the charge transfer resistance without and with investigated extract, respectively. With increasing of extract dose R_{ct} value increases, CR decreases, C_{dl} decreases that indicating a decrease in the local dielectric constant and/or an increase in the thickness of the electrical double layer, suggesting the extract molecules function by the formation of the protective layer at the metal surface. [19, 20].

3.2.2. PP measurements

In presence of Delonix regia leaf extract both anodic and cathodic polarization curves shifted to less current density values due to inhibitive action of extract and with increasing of extract dose the extent of shift increase (Figure 5). The values of corrosion current density (i_{corr}), corrosion potential (E_{corr}), anodic Tafel constants (β_a) and cathodic Tafel constant (β_c) from polarization curves are given in Table 3. From Table 3, by increasing dose of Delonix regia leaf extract, corrosion potential (E_{corr}) is largely shifted to less negative values corrosion current density (i_{corr}) decreased and The % IE increased. Both the anodic and cathodic Tafel constants decrease. these results indicate that
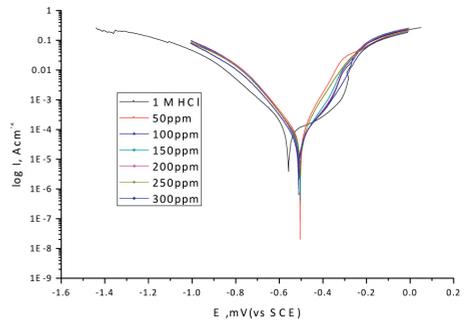

Fig. 5: PP for corrosion of CS in 1 M HCl in the absence and presence of different doses of Delonix regia leaf at 25°C

the Delonix regia leaf acts as mixed inhibitor [21] .This means that the Delonix regia leaf molecules are adsorbed on both the anodic and cathodic sites resulting in an inhibition of both anodic dissolution and cathodic reduction reactions. The values of %IE obtained by polarization technique are similar to those obtained by WL measurements and EIS.

3.2.3. EFM measurements

Electrochemical frequency modulation (EFM) technique has advantage that is used to determine the corrosion current value without knowledge of Tafel

Tab. 3 : PP data of CS in 1 M HCl in the presence of different doses of Delonix regia leaf extract at 25°C

Conc., ppm	i_{corr}, µA cm^{-2}	-E_{corr}, mV vs SCE	β_a, mV dec^{-1}	β_c, mV dec^{-1}	CR, mmy	θ	%IE
0 (1M HCl)	119.0	559	466	108.4	54.56	---	---
50	70.2	504	74	117	32.06	0.41	41.0
100	64.2	509	137	125	29.32	0.46	46.0
150	57.9	504	105	120	26.45	0.51	51.0
200	56.3	507	115	118	25.74	0.53	53.0
250	53.2	503	116	118	24.29	0.55	55.0
300	40.1	511	92	99	18.30	0.66	66.0

slopes [22]. The great strength of the EFM is the causality factors which serve as an internal check on the validity of EFM measurement. The causality factors CF-2 and CF-3 are calculated from the frequency spectrum of the current responses. The experimental EFM data were treated using two different models: complete diffusion control of the cathodic reaction and the "activation" model. For the latter, a set of three non-linear equations had been solved, assuming that the corrosion potential does not change due to the polarization of the working electrode [23]. The larger peaks were used to calculate the corrosion current density (i_{corr}), the Tafel slopes (β_c and β_a) and the causality factors (CF-2 and CF-3) as shown in Table 4, indicating that this extract inhibit the corrosion of CS in 1 M HCl through adsorption [24]. By increasing concentration of Delonix regia leaf extract the IE_{EFM} % increase, IE_{EFM} % calculated as follows:

$$IE\%_{EFM=}\left(1-\frac{i_{corr}}{i_{corr}^{\circ}}\right)\times100 \qquad (8)$$

Where i°_{corr} and i_{corr} are C.R. without and with Delonix regia leaf extract, respectively.

3.3.1. Adsorption isotherms

Information on the interaction between inhibitor and metal surface can be provided using the adsorption isotherms [25].The adsorption of an organic molecules at metal–solution interface can occur as a result of adsorption process between organic molecules present in the aqueous solution (Org) and the water molecules previously adsorbed on the metallic surface (H_2O) [26]:

$$Org_{sol} + xH_2O_{ads} = Org_{ads} + xH_2O_{sol} \qquad (9)$$
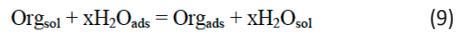
Where Org $_{(sol)}$ and Org $_{(ads)}$ are the organic species in the bulk solution and adsorbed one on the metallic surface, respectively H_2O is the water molecule adsorbed on the metallic surface and x is the size ratio representing the number of water molecules replaced by one organic molecule. The surface coverage values (θ) were evaluated using corrosion rate values obtained from the weight loss method. The values for different inhibitor concentrations were tested by fitting to various isotherms. The best fit was obtained with Temkin adsorption isotherm (Fig. 6), given by Equation 10:

$$\theta = 1/f \ln K_{ads} C \qquad (10)$$

Where f is the factor of energetic homogeneity, of the metal surface describing the molecular interactions in the adsorption layer [27]. If f > 0, mutual repulsion of molecules occurs and if f <

Tab. 4: Electrochemical kinetic parameters obtained by EFM technique for CS in the absence and presence of various doses of the Delonix regia leaf extract in 1 M HCl at 25^{5}C

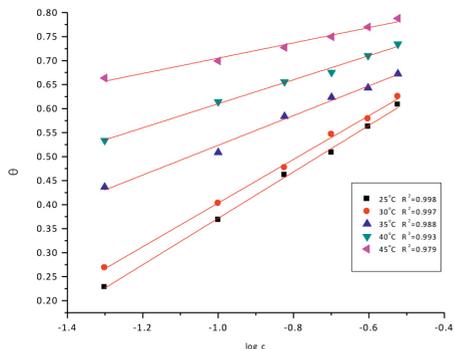| Conc., ppm	i_{corr}, µA cm^{-2}	β_a mV dec^{-1}	β_c mV dec^{-1}	C.R. mpy	CF2	CF3	%IE	θ
blank	171.6	14.3	33.2	78.41	1.9	2.2	----	---
250	52.6	52.6	37.16	3.5	1.8	2.5	81.33	0.8133
200	54.6	54.6	35.61	3.07	2.0	2.7	77.93	0.7793
150	55.7	55.7	34.73	3.23	2.1	2.8	76.00	0.7600
100	55.9	55.9	34.61	2.37	2.0	2.7	75.74	0.7574
250	57.5	57.5	33.35	2.5	1.9	2.5	73.00	0.7300
50	58.4	58.4	32.61	3.4	2.0	2.7	71.36	0.7136

Fig. 6: Temkin adsorption plots for CS in 1 M HCl containing various doses of Delonix regia leaf at 25°C

0 attraction takes place [28]. From Figure 8 it can be observe that, both the linear correlation coefficient (R^2) and the slope of straight line are close to 1, so suggests that the adsorption of Delonix regia leaf on CS surface obeys the Temkin model and exhibit single-layer adsorption characteristic. The equilibrium constant of the adsorption process (K_{ads}) can be calculated from the intercept of the straight line. Relation between K_{ads} and $\Delta G°_{ads}$ is given by the following equation [29]:

$$K_{ads}=1/55.5 \exp \Delta G^0_{ads}/RT \qquad (11)$$

The value 55.5 is the concentration of water in solution in mol l^{-1}. The calculated ΔG^0_{ads} values, using Equation (11), were also given in Table 5.

Tab. 5: Values of adsorption isotherm parameters for CS in 1 M HCl contain different doses of Delonix regia leaf extract.

Temp., K	Adsorption isotherm	K_{ads}, $g^{-1}L$	$-\Delta G°_{ads}$, kJ mol^{-1}
298		58.7	20.0
303		77.1	21.1
308	Temkin	491.0	26.1
313		2740.4	31.1
318		2750.3	33.7

Negative values of $\Delta G°_{ads}$ indicate that the adsorption process is spontaneous. If the absolute value of ΔG^0_{ads} was of the order of -20 kJ mol^{-1} or lower, the adsorption process is physisorption and in this process the inhibition occurs by the electrostatic interaction between the organic molecules and iron atom. If the absolute value of $\Delta G°_{ads}$ is of the order of -40 kJ mol^{-1} or higher, the adsorption process is chemisorption and in this process the covalent bond is formed by the charge sharing or transferring from the inhibitor molecules to the metal surface [30, 31]. Calculated $\Delta G°_{ads}$ values from Table 6 indicate that the adsorption mechanism of Delonix regia leaf extract on CS in 1 M HCl solution is typically chemisorption and Delonix regia leaf extract is strongly adsorbed on the CS surface.

3.3.2. Effect of temperature

By using of WL measurements that listed in Table 6, we can study the effect of temperature (range from 25 to 45°C) on the corrosion rate of CS in 1M HCl in absence and presence of different doses of Delonix regia leaf extract. As the temperature and concentration of Delonix regia leaf extract increase, the CR decreases and hence the IE% increases. So the inhibition behavior was observed for chemisorption of extract on metal surfaces.

In examining the effect of temperature on the corrosion process in the presence of the Delonix regia leaf, the Arrhenius equation below was used:

$$\text{Log } C.R. = \log A - \frac{E_a}{2.303 \cdot R \cdot T} \qquad (12)$$

Where C.R. is the corrosion rate, E_a is the apparent activation energy, and A is the frequency factor.

Figure 7 represents Arrhenius plot (as log C.R. versus 1/T) for CS corrosion in 1M HCl in the absence and presence of different doses of Delonix regia leaf extract. Straight lines were obtained with slope equals to E_a / 2.303 R. Table 7 contains values of Ea for the corrosion reaction in the absence and presence of Delonix regia leaf extract. Table 8 shows the decrease of activation Energy (E_a) and corrosion rate of CS. The values of E_a of the corrosion process of CS protected by extract are lower than the unprotected CS in 1 M HCl solution. The large decrease in the Ea of the corrosion process in the presence of the extract indicates the higher IE of the extract due to the adsorption of extract molecules on the metal surface to form stable metal-inhibitor complex (M Inh) [32].

The other thermodynamic parameters (ΔS^* and ΔH^*) were calculated from the linear regression of transition state (Fig. 8) using Equation (13)

$$C.R. = (RT/Nh)\, exp^{(\Delta S^*/R)}\, exp^{(-\Delta H^*/RT)} \tag{13}$$

Where h is Planck's constant, N is Avogadro number, ΔS^* is the entropy of activation and ΔH^* is the enthalpy of activation. A plot of log (C.R./T) vs. 1/T (Fig. 8) should give a straight line, with a slope of (ΔH^*/2.303R) and an intercept of [log (R/Nh)+ΔS^*/2.303R]. Examination of the kinetic values shows that the increase of inhibitor concentration leads to decrease of

Tab. 6: Data of WL measurements for CS in 1 M HCl solution in the absence and presence of different doses of Delonix regia leaf extract at different temperatures

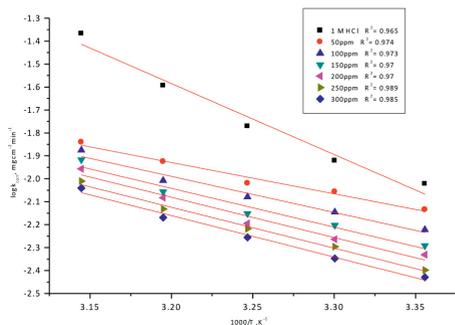| Conc., ppm	Temp., °C	C.R., mg cm^{-2} min^{-1}	θ	%IE
50	25	0.0073	0.228	22.8
	30	0.0088	0.269	26.9
	35	0.0096	0.436	43.6
	40	0.0119	0.534	53.4
	45	0.0144	0.664	66.4
100	25	0.006	0.368	36.8
	30	0.0072	0.403	40.3
	35	0.0083	0.509	50.9
	40	0.0098	0.614	61.4
	45	0.0133	0.69	69.0
150	25	0.0051	0.462	46.2
	30	0.0063	0.477	47.7
	35	0.0071	0.584	58.4
	40	0.0088	0.656	65.6
	45	0.0121	0.718	71.8
200	25	0.0047	0.509	50.9
	30	0.0054	0.546	54.6
	35	0.0064	0.623	62.3
	40	0.0083	0.675	67.5
	45	0.0111	0.743	74.3
250	25	0.004	0.579	57.9
	30	0.0051	0.579	75.9
	35	0.0061	0.643	64.3
	40	0.0074	0.71	71.0
	45	0.0098	0.722	72.2
300	25	0.0037	0.608	60.8
	30	0.0045	0.625	62.5
	35	0.0056	0.672	67.2
	40	0.0068	0.734	73.4
		0.0091	0.788	78.8

Fig. 7: Arrhenius plots, Log C.R. vs 1/T curves for CS in 1 M HCl in the absence and presence of different doses of Delonix regia leaf extract.

Tab. 7: Activation parameters for corrosion of CS in the absence and presence of different doses of Delonix regia leaf extract in 1 M HCl at different temperatures

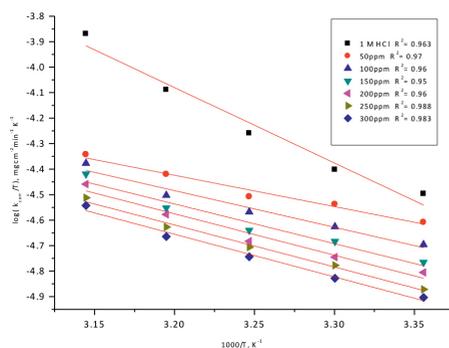| Conc., ppm	E_a kJ mol^{-1}	ΔH^*, kJ mol^{-1}	$-\Delta S^*$, J mol^{-1} K^{-1}
0 (1M HCl)	59.3	24.6	94.2
50	34.6	13.9	206.9
100	34.1	13.7	195.4
150	33.7	13.5	189.0
200	32.4	13.0	185.6
250	30.1	12.0	185.0
300	26.1	10.2	184.1

Fig. 8: Transition state plots Log C.R./T vs. 1/T curves for CS in 1 M HCl in the absence and presence of different doses of Delonix regia leaf extract

all parameters of corrosion process (Table 7). The positive value of the enthalpy ΔH* means that the process is endothermic and it needs more energy to achieve the activated state or equilibrium. The negative value of ΔS* (Table 8) indicates that activated complex in the rate determining step represents an association rather than a dissociation step, meaning that a decrease in disorder takes place during the course of transition from reactant to the activated complex [33].

4. Conclusions

On the basis of this study the following conclusions can be drawn:

1. Delonix regia leaf extract acts as corrosion inhibitor for carbon steel in acidic medium.

2. The inhibition efficiency of Delonix regia leaf extract increases with increase of the extract concentration and also with temperature.

3. Delonix regia leaf was found to obey Temkin adsorption isotherm from the fit of the experimental data at all the doses studied.

4. The values of E^*_a obtained in the presence of the Delonix regia leaf extract were lower compared to the blank acid solution which further support the chemical adsorption proposed.

5. The values of ΔG^o_{ads} obtained are low and negative, which reveals the spontaneity of the adsorption process

6. Also the results indicate that the Delonix regia leaf extract acts as mixed type inhibitor.

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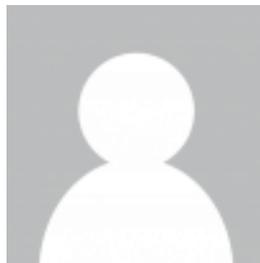
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