

Inhibitory Effects of *Delonix regia*(Gulmohor) extract on Corrosion of Aluminium in Hydrochloric Acid

N. I. Prajapati and R. T. Vashi*

*Department of Chemistry, Navyug Science College, Rander Road, Surat-395005, Gujarat, India.

For correspondence: Email: 6haprajapati@gmail.com; Tel. No.: 09998572828.

Abstract

The inhibition of the corrosion of aluminium in hydrochloric acid solution by the leaves extract of *Delonix regia* has been studied using weight loss, Potentiodynamic Polarization, Electrochemical Impedance Spectroscopic (EIS) techniques. Corrosion rate increases with the increase in acid concentration and temperature. As inhibitor concentration increases corrosion rate decreases while percentage of inhibition efficiency (I.E.) increases. The value of free energy of adsorption (ΔG_{ads}^0), heat of adsorption (Q_{ads}), energy of activation (E_a), enthalpy of adsorption (ΔH_{ads}^0) and entropy of adsorption (ΔS_{ads}^0) were calculated. The inhibition effect is discussed in view of *Delonix regia* molecules adsorbed on the metal surface and it obeys Langmuir adsorption isotherm. Polarization curve indicates that inhibitor act as mixed type and the I.E. was found up to 93.48 %. The results obtained showed that the leaves extract could serve as an effective green inhibitor for corrosion of aluminium in hydrochloric acid.

Key words: Aluminium, Hydrochloric acid, *Delonix regia* , Corrosion, Inhibitor.

1. Introduction

Aluminium is one of the metals which is used in different human activities and many of important application. Aluminum is widely used in various industrial operations due to light weight, high thermal and electrical conductivity, relative high mechanical strength. Aluminium and its alloy are widely used in construction, vessels, pipes, machinery and packing. Aluminium is used in electronics due to it is super purity [1]. Hydrochloric acid solution is one of the most currently used acids in the pickling and electrochemical etching of aluminium capacitor foil [2].

In the recent years, there is an increasing awareness of environment and green chemistry. Therefore, many works were conducted to use the environmentally safe, readily available and cheap substances, as corrosion inhibitors, instead of the harmful synthetic chemicals [3-7]. *Delonix regia* contains large amount of terpenoids, polyphenolic compounds, tannins, cardiac glycosides and anthroquinones [8]. The aim of the present study is to investigate the corrosion inhibition effect of *Delonix regia* as a cheap and environment friendly corrosion inhibitor for aluminium in various concentration of HCl medium by weight loss, effect of temperature, polarization and EIS techniques.

2. Experimental section

2.1 Preparation of sample and solution

The aluminium specimens with a chemical composition of 99.54 % Al, 0.090 % Si, 0.320 % Fe, 0.0012 % Cu, 0.0034 % Mn, 0.0014 % Mg, 0.0042 % Cr, 0.0046 % Ni, 0.0020 % Zn, 0.0079 % Ti, 0.0005 % Pb, and 0.0026 % Sn were used in the present study. The metal sheet, test specimens of size 5.0 x 2.50 x 0.198cm having an effective area of 0.279 dm² were used. The specimens were cleaned by washing with distilled water, degreased by acetone, washed once more with doubled distilled water and finally dried and weighted by using electronic balance. Hydrochloric acid was used as corrosive solution having concentration of 0.75, 1.0 and 1.25 M prepared by diluting analytical grade of HCl purchased from Merck using double distilled water.

2.2 Preparation of extract

The extract of leaves of *Delonix regia* was prepared as follows: fresh leaves of *Delonix regia* were dried and ground into powder, then 0.2g of powder was put into a 200ml flat bottom flask containing 100ml of 2 M HCl. The resulting solution was boiled for 30 min and left overnight before filtering. The filtrate was diluted with the appropriate quantity of 2 M HCl to obtain 0.6, 0.8, 1.0 and 1.2g/L concentrations. The above process was repeated for the preparation of the acid leaves extract [5].

2.3 Weight loss measurements

For weight-loss measurement, the aluminium specimen having an area of 0.2797 dm² were each completely suspended in 230 ml of 0.75, 1.0 and 1.25 M HCl solution with and without different *Delonix regia* leaves extract concentrations using glass hooks at 301± 1 K for 24h. The coupons were retrieved after 24h, washed with distilled water, dried well and reweighed. From the weight loss data, corrosion rate (CR) was calculated.

2.4 Temperature effect

To study the effect of temperature on corrosion rate, aluminium coupons were completely immersed in 230 ml of 0.75M HCl solution without and with different concentrations of *Delonix regia* leaves extract at 313, 323 and 333K for 2h. From the data corrosion rate, inhibition efficiency (I.E.), activation energy (E_a) and heat of adsorption (Q_{ads}) and free energy of adsorption (ΔG^0_{ads}) were calculated.

2.5 Potentiodynamic polarization measurements

Both the potentiodynamic and EIS measurement were carried out using CHI608C –series, U.S. Model with CH- instrument. For polarization study, metal specimens were immersed with and without *Delonix regia* leaves extract in 0.75 M HCl solution. In the electrochemical cell aluminium specimens having an area of 1 cm² was used as a working electrode, Ag/AgCl electrode as a reference electrode and platinum electrode as an auxiliary electrode and allowed to establish a steady-state open

circuit potential (OCP) for approximately 65 min. The polarization curves were plotted with current Vs potential. An anodic and cathodic polarization curve gives corresponding anodic and cathodic Tafel lines. The intersect point of Tafel lines gives the corrosion potential (E_{corr}) and corrosion current (i_{corr}) [9].

2.6 Electrochemical Impedance Spectroscopy (EIS) measurements

EIS measurements were made at corrosion potentials over a frequency range of 1 to 10^5 Hz by a sine wave with potential perturbation amplitude of 5 mV. A graph was drawn by plotting real impedance (Z') versus imaginary impedance ($-Z''$). From the Nyquist plots of Z' Vs $-Z''$ the charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were calculated. An experiment was carried out in absence and presence of inhibitor.

3. Results and discussion

3.1 Weight loss experiments

The weight loss experiments were carried out in 0.75, 1.0 and 1.25 M HCl solution containing 0.6, 0.8, 1.0 and 1.2 g/L concentration of *Delonix regia* leaves extract at 301 ± 1 K for an exposure period of 24 h was investigated. Corrosion rate (C.R.) was calculated using following equation:

$$\text{C.R. (mg/dm}^2\text{d)} = \text{Weight loss (gm.)} \times 1000 / \text{Area in dm}^2 \times \text{day} \quad (1)$$

Inhibition efficiency (I.E.) was calculated by using following equation,

$$\text{I. E. (\%)} = \left\{ \frac{(W_u - W_i)}{W_u} \right\} \times 100 \quad (2)$$

Where: W_u = Weight loss in absence of inhibitor, W_i = Weight loss in presence of inhibitor.

The degree of surface coverage (θ) of the aluminium specimen for different concentration of HCl solution have been evaluated by weight loss experiments using the following equation,

$$\theta = \frac{(W_u - W_i)}{W_i} \quad (3)$$

Results showed in Table-1 indicate that as the acid concentration increases corrosion rate increase while I.E. decreases. Corrosion rate was 2195.20, 6163.74 and 8283.87 mg/dm²d corresponding to 0.75, 1.0 and 1.25 M HCl concentrations respectively for an immersion period of 24 h at 301 ± 1 K (Figure-1). At constant acid concentration, as the inhibitor concentration increases corrosion rate decreases while I.E. increases. For example, in 0.75 M HCl solution, the I.E. was found to be 56.02, 66.61, 83.55 and 93.48 % corresponding to 0.6, 0.8, 1.0 and 1.2 g/L inhibitor concentration respectively (Table-1 and Figure-2).

Table-1. Effect of HCl concentration on corrosion rate (C.R.) and inhibition efficiency (IE) of aluminium having different concentration of *Delonix regia* leaves extract.

Inhibitor concentration (g/L)	Acid concentration					
	0.75 M		1.0 M		1.25 M	
	CR (mg/dm ² d)	I.E. (%)	CR (mg/dm ² d)	I.E. (%)	CR (mg/dm ² d)	I.E. (%)
Blank	2195.20	-	6163.74	-	8283.87	-
0.6	965.31	56.02	2931.71	52.43	4290.31	48.20
0.8	732.92	66.61	2398.99	61.07	3578.83	56.79
1.0	361.10	83.55	1337.14	78.30	2506.25	69.74
1.2	143.01	93.48	829.46	86.54	1433.67	82.69

Table-2: Inhibition efficiency (I.E.), Corrosion rate (ρ) and Surface coverage (θ) of *Delonix regia* leaves extract on aluminium in 0.75 M HCl for an immersion period of 24h at 301 ± 1 K.

Inhibitor	Inhibitor Concentration (g/L)	C.R. (ρ) (mg/dm ² d)	log ρ	I.E. (%)	Surface coverage θ	C/ θ
Blank	-	2195.20	3.34	-	-	-
<i>Delonix regia</i> (Gulmohor)	0.6	965.31	2.98	56.02	0.56	1.07
	0.8	732.92	2.86	66.61	0.66	1.21
	1.0	361.10	2.55	83.55	0.83	1.20
	1.2	143.01	2.15	93.48	0.93	1.29

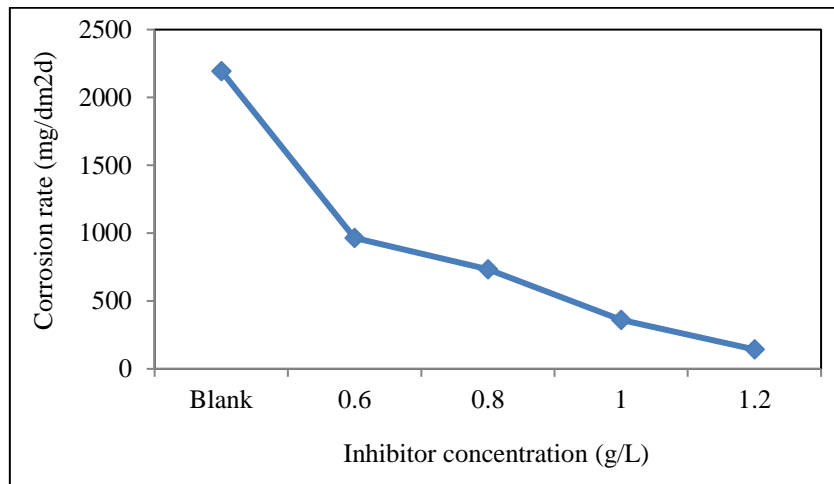


Fig.1: Corrosion rate of aluminium in 0.75 M HCl solution in absence and presence of different concentration *Delonix regia* leaves extract for 24 h.

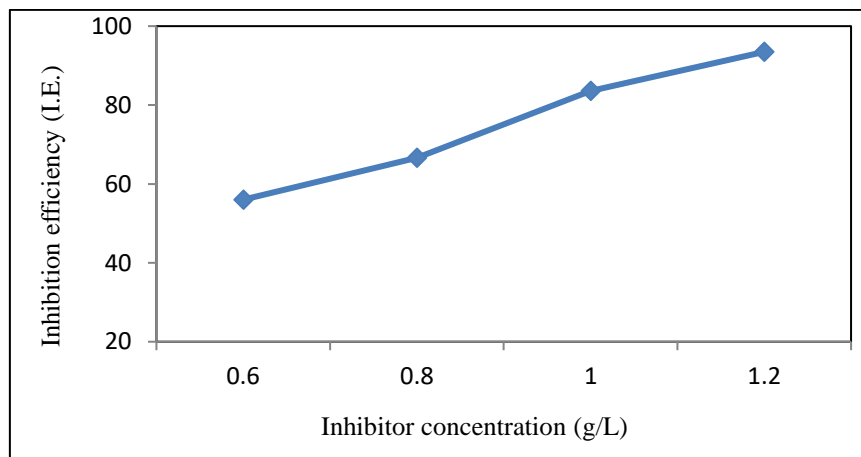


Fig.2: Inhibition efficiency of aluminium corrosion in 0.75 M HCl solution in presence of different concentration *Delonix regia* extract for an immersion period of 24h.

3.2 Temperature effect

To investigate the influence of temperature on corrosion of aluminium, the weight loss experiments were also carried out at 313, 323 and 333K in 0.75 M HNO₃ in absence and presence of *Delonix regia* for an immersion period of 2h. The results in Table-3 shows that corrosion rate increases with rise in temperature. Corrosion rate was 21451.44, 26642.76 and 43417.92 mg/dm²d corresponding to 313, 323 and 333K respectively. The value of energy of activation (E_a) has been calculated with the help of Arrhenius equation [10].

$$\log \frac{\rho_2}{\rho_1} = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad (4)$$

Where ρ_1 and ρ_2 are the corrosion rate at temperature T_1 and T_2 respectively.

Table-3. Temperature effect on corrosion rate (CR), activation energy (E_a) and heat of adsorption (Q_{ads}) for aluminium in 0.75 M HCl in absence and presence of *Delonix regia* extract for an immersion period of 2 h.

Inhibitor concentration (g/L)	Temperature						Mean (E _a) From Equation (4) (kJ/mol)	Q _{ads} (kJ/ mol)	
	313 K		323 K		333 K			313-323 K	323-333 K
	CR mg/dm ² d	I. E. (%)	CR mg/dm ² d	I. E. (%)	CR mg/dm ² d	I. E. (%)			
Blank	21451.44	-	26642.76	-	43417.92	-	30.92	-	-
0.6	3560.88	83.4	6950.28	73.9	18362.52	57.70	71.54	-48.16	-65.36
0.8	2874.48	86.6	6006.36	77.4	16646.40	61.66	76.54	-53.15	-67.85
1.0	2102.16	90.2	5148.36	80.6	12484.80	71.24	77.25	-66.49	-46.64
1.2	943.80	95.6	4247.40	84.0	8237.28	81.02	92.83	-119.10	-18.84

Results given in Table-3, indicates that the values of E_a were higher in inhibited acid ranging from 71.54 to 92.83 kJmol⁻¹ than E_a value for uninhibited acid (30.92 kJ/mol) which indicates physical adsorption of the inhibitor on metal surface and the adsorption of inhibitor causes an increase in the E_a value of the process [11]. Results of Table-3 indicates that as temperature increases, rate of corrosion increase while percentage of I.E. decreases. The value of E_a were also calculated from the slope of the Arrhenius plot of $\log \rho$ versus $1/T \times 1000$ (Figure-3) shows good agreement with the calculated values.

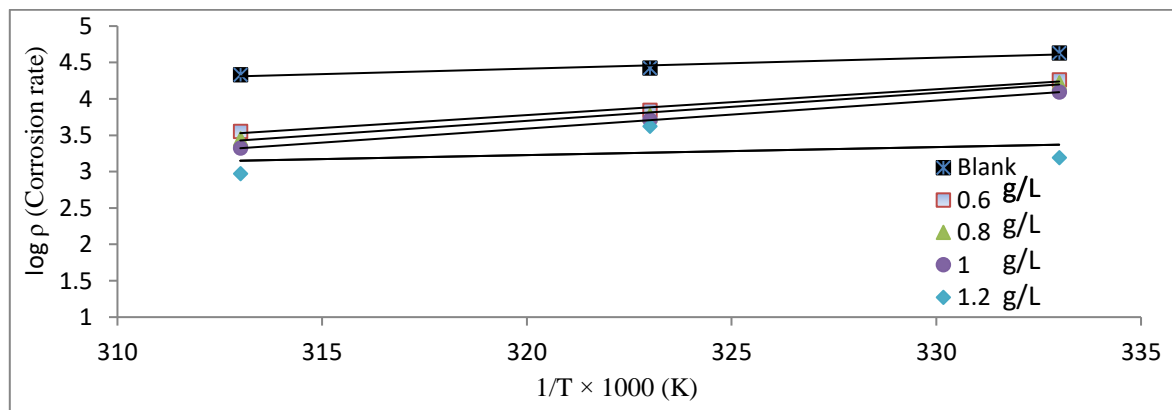


Fig.3 : Arrhenius plots for aluminium in 0.75 M HCl in absence and presence of the different concentration of *Delonix regia* extract.

The values of heat of adsorption (Q_{ads}) were calculated by using the following equation [12].

$$Q_{ads} = 2.303R \left[\log \left(\frac{\theta_2}{1-\theta_2} \right) - \log \left(\frac{\theta_1}{1-\theta_1} \right) \right] \left[\frac{T_1 T_2}{T_2 - T_1} \right] \quad (5)$$

Where, θ_1 and θ_2 are the fraction of the metal surface covered by the inhibitor at temperature T_1 and T_2 respectively. It is evident that in all cases, the value of Q_{ads} were negative and ranging from -18.84 to -119.10 kJ/mol. The negative values shows that the adsorption and hence the I.E. decreases with rise in temperature.

The surface coverage ' θ ' value was calculated by using equation- 3. A plot of inhibitor concentration C_{inh} versus C_{inh}/θ was presented in Figure-3 which gives straight line with slope values equal to unity indicates that the system follows Langmuir adsorption isotherm [13]. This isotherm can be represented as,

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (6)$$

Where, K_{ads} is the equilibrium constant and C_{inh} is the inhibitor concentration.

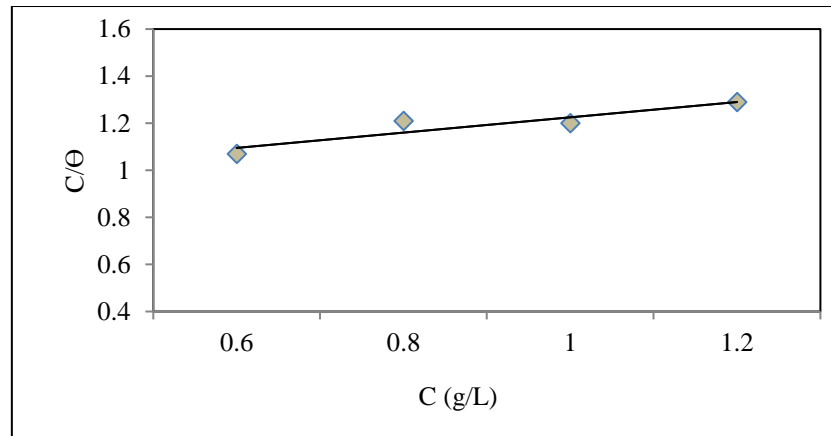


Fig.4: Langmuir adsorption isotherm plot for aluminium in 0.75 M HCl containing *Delonix regia* as green inhibitor at 301 K.

Free energy of adsorption (ΔG°_{ads}) was determined by the Langmuir isotherm was given by a plot of C/θ versus C (Figure-4) [14]. From the intercepts of the straight line on the C/θ axis, K_{ads} can be calculated which was related to ΔG°_{ads} as given by the following equation [15,16].

$$\Delta G^{\circ}_{ads} = -RT \ln (55.5 K_{ads}) \quad (7)$$

Where, R is the gas constant, T is the concentration of water in solution in Molar [17], K_{ads} is the equilibrium constant of the adsorption/desorption process. The mean ΔG°_{ads} value was negative (-13.82 kJ/mol) indicating that the adsorption mechanism of *Delonix regia* on aluminium in 0.75 M HCl at the studied temperatures is physisorption with adsorptive layer having electrostatic character [18]. This is concluded on the fact that the values of ΔG°_{ads} -20 kJ/mol are consistent with physisorption, while those around -40 kJ/mol or higher are associated with chemisorption [19].

The enthalpy of adsorption (ΔH_a°) and entropy of adsorption (ΔS_a°) were calculated using the equations (8) and (9),

$$\Delta H_a^\circ = E_a - RT \quad (8)$$

$$\Delta S_a^\circ = \Delta H_a^\circ - \Delta G_a^\circ / T \quad (9)$$

Results indicates that values of ΔH_a° were positive and increase in presence of inhibitor indicating a higher degree of surface coverage and higher protection efficiency attained due to raising the energy barrier for the aluminium corrosion reaction. The enthalpy change ΔH_a° was positive and ranging between 56.18 to 126.41 kJ/mol indicating the endothermic nature of the reaction suggests that higher temperature favours the corrosion process. Positive values of ΔS_a° ranging from 0.225 to 0.450 kJ/mol indicates that corrosion process is entropically favourable.

3.3 Potentiodynamic polarization study

Figure-5 represents the potentiodynamic polarization curves of aluminium in 0.75 M HCl in absence and presence of *Delonix regia* leaves extract. Electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), anodic Tafel slope (β_a), cathodic Tafel slope (β_c) and percentage inhibition efficiency (I.E.) were given in Table-4.

Table -4. Potentiodynamic polarization data and Inhibition efficiency (I.E.) of *Delonix regia* leaves extract as green inhibitor for aluminium in 0.75 M HCl.

System	E_{corr} (V)	I_{corr} ($\mu A / cm^2$)	Tafel slope (mV / decade)			IE(%) Calculated from	
			Anodic ($+\beta_a$)	Cathodic ($-\beta_c$)	β (mV)	Polarization method	Weight loss method
Blank	-0.895	9156	6.706	5.621	1.329	-	-
<i>Delonix regia</i> (Gulmohor)	-0.891	1085	8.884	6.080	1.569	88.14	93.48

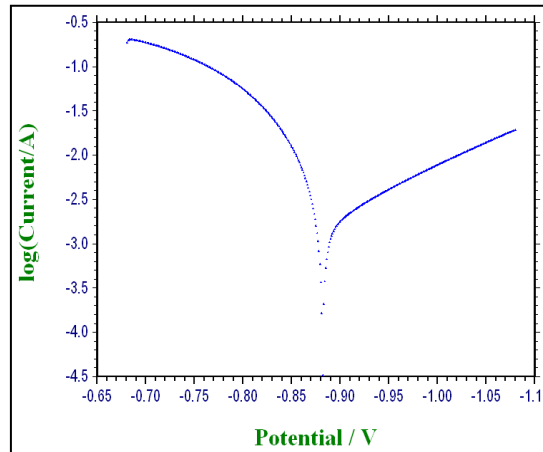


Fig.5(a):Polarization curve for corrosion of aluminium in 0.75 M HCl in absence of inhibitor.

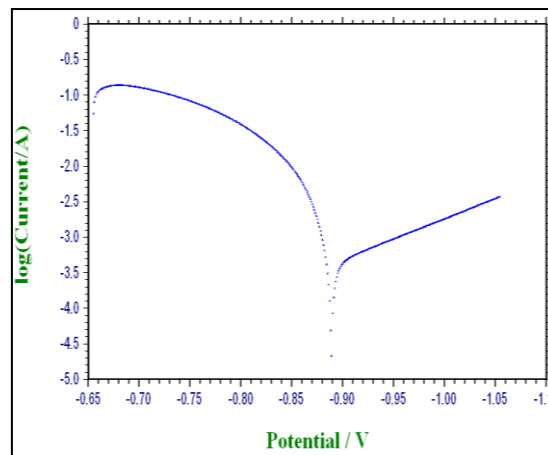


Fig.5(b): Polarization curves for aluminium in 0.75 M HCl in presence of 1.2 g/L *Delonix regia* extract.

From Table-4, it was observed that the addition of *Delonix regia* leaves extract in acid solution indicates the significant decrease in corrosion current density (i_{corr}) and decrease in corrosion rate with respect to blank. There is significant change in the anodic and cathodic slopes after the addition of the inhibitor. This Tafel curves indicate that *Delonix regia* function as a mixed type inhibitor with the predominant cathode effectiveness (Figure-5 and Figure-6).

Inhibition efficiency (I.E.) from polarization study was calculated using following equation [20].

$$\text{I. E. (\%)} = \frac{i_{corr(uninh)} - i_{corr(inh)}}{i_{corr(uninh)}} \quad (10)$$

3.4 Electrochemical impedance spectroscopy (EIS) measurements

Nyquist plots for the corrosion of aluminium in 0.75M HCl solution in absence and presence of *Delonix regia* leaves extract was examined by EIS method at room temperature was shown in Figure-7 and EIS parameters in Table-5.

Table-5. EIS parameters for corrosion of Aluminium in 0.75 M HCl containing *Delonix regia* leaves extract.

System	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F} / \text{cm}^2$)	IE(%)	
			Calculated from	
			EIS method	Weight loss method
Blank	84	45.130	-	-
<i>Delonix regia</i> (Gulmohor)	240	5.529	87.74	93.48

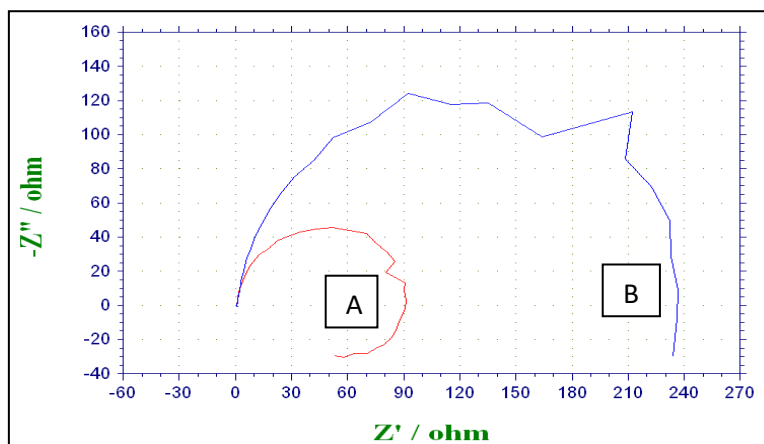


Fig.6 : Nyquist plot for aluminium in (A) in 0.75 M HCl (Blank) (B) in 0.75 M HCl in presence of 1.2 g/L *Delonix regia* extract.

It is observed from Figure-6 that the impedance diagram is almost semi circular. The difference has been attributed to frequency dispersion. The semi circular nature of the plots indicates that the corrosion of aluminium is mainly controlled by charge transfer process.

The diameter of capacitive loop in the presence of inhibitor is bigger than that in the absence of inhibitor. The high frequency capacitive loop is related to the charge transfer resistance (R_{ct}). To calculate the double layer capacitance (C_{dl}), the frequency at which the imaginary component of the impedance is maximum was found as presented in the following equation [21].

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad (11)$$

Where' f' is the frequency at the maximum height of the semicircle on the imaginary axis and R_{ct} is the charge transfer resistance [22].

Inhibition efficiency (I.E.) from EIS method was calculated using following equation:

$$I.E. (\%) = \frac{C_{dl}(uninhi.) - C_{dl}(inhi.)}{C_{dl}(uninhi.)} \times 100 \quad (12)$$

The addition of inhibitor increase R_{ct} value while decreases in C_{dl} values which is due to the adsorption of inhibitor on the metal surface. The results suggest that the inhibitor acts by the formation of a physical protective layer on the surface that retards the charge transfer process and therefore inhibit the corrosion reaction, leading to increase in R_{ct} values. Moreover, the adsorbed inhibitor species decrease the electrical capacity of electrical double layer values at the electrode/solution interface and therefore decrease the value of C_{dl} [23].

3.5 Mechanism of corrosion

Generally, aluminium dissolves in acid solutions due to hydrogen evolution type of attack, the reactions taking place at the microelectrode of the corrosion cell being represented as [24,25]:



Followed by the reactions:



The following secondary reactons can also take place in acid solutions [26]:



3.6 Mechanism of inhibition

Chou and Leu [27] noted that *Delonix regia* leaves extract contains some phytochemicals with hetero atoms (N,S,O) having higher molecular weight such as 4-hydroxybenzoic acid, gallic acid, 3,4-dihydroxycinnunmic acid, 3,5-dinitrobenzoic acid, alkaloids, L-azetidine-2-carboxylic acid, amine oxide base and 3,4-dihydroxybenzaldehyde etc. Structure of some of these compounds are shown in Figure-7.

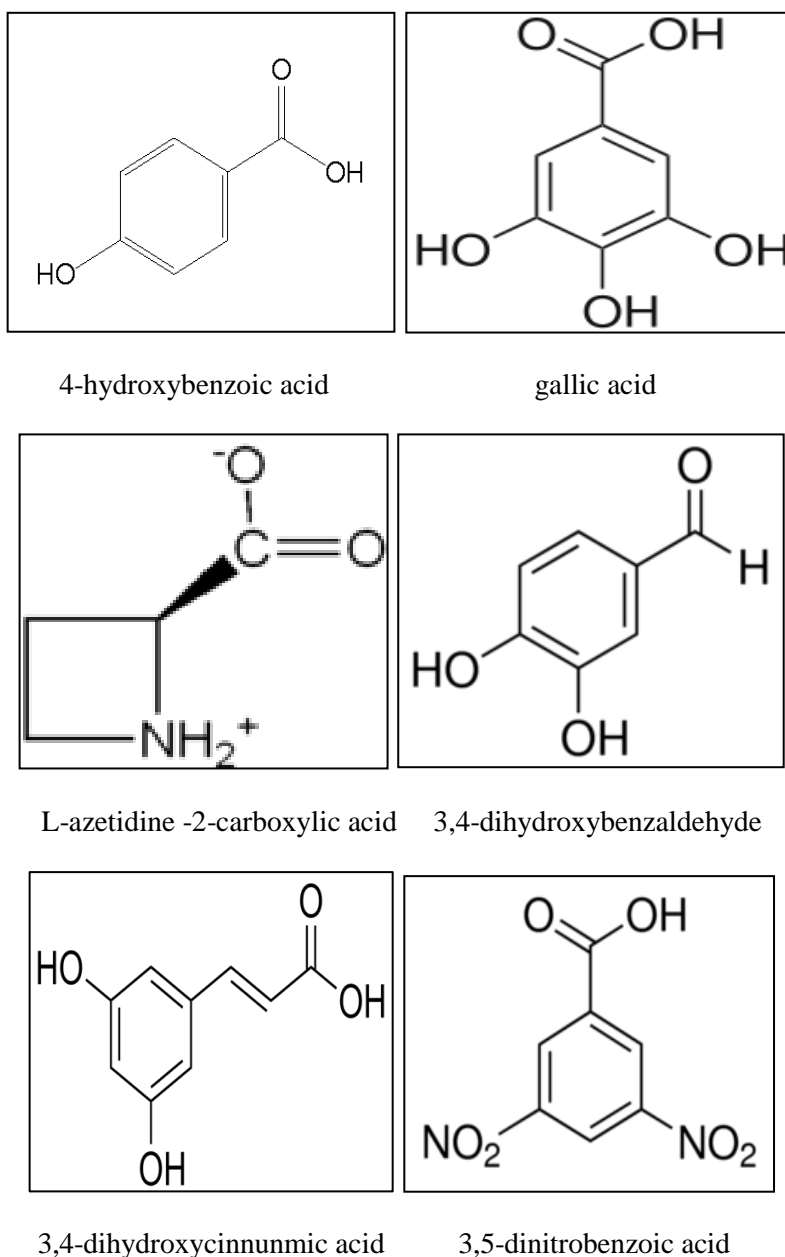


Fig.7: Structure of phytochemicals

These phytochemical substances (N organic compounds) due to their ability to form soluble amine salts with HCl [28]. This translates to high concentrations of N organic compounds in acid extract of leaves. The inhibitive properties of *Delonix regia* may be due to the presence of phytochemicals having functional groups –OR, -NR, and or –SR in the extract. This extract may be able to inhibit by being adsorbed on the metal surface through the donation of the nitrogen or oxygen or sulphur or by being adsorbed on the metal surface through their pi electrons [5].

From Table-3, it can also be shown that the I.E. decreases with an increase in temperature. Such behaviour can be interpreted in terms of the fact that the phytochemical molecules are physically

adsorbed on the surface of metal. Since, a physisorption mechanism is indicative of a weak adsorption bond, it may be possible that at higher temperatures, the decrease in I.E. may be a result of increased agitation of the solution resulting from higher rates of hydrogen evolution, thereby reducing the ability of the molecules to be adsorbed on the surface of the metal.

4. Conclusions

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

1. As acid concentration increases corrosion rate increases while I.E. decreases.
2. At constant acid concentration, as inhibitor concentration increases corrosion rate decreases while I.E. increases.
3. As temperature increase corrosion rate increases while I.E. decreases.
4. The leaves extract of *Delonix regia* showed maximum I.E. of 93.48% at an optimum concentration of 1.2 g/L.
5. The values of E_a obtained in the presence of the extract were higher compared to the blank acid solution which indicates that inhibitor was more effective at lower temperature.
6. The values of ΔG_{ads}° were negative, which reveals the spontaneous adsorption of inhibitor onto metal surface.
7. Plot of C/θ versus C shows straight line with almost unit slope, which suggest that the inhibitor cover both anodic and cathodic regions through general adsorption following Langmuir isotherm.
8. Polarization curves indicates that the *Delonix regia* extract act as mixed type of inhibitor.

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