ejpmr, 2015,2(1),470-485

EUROPEAN JOURNAL OF PHARMACEUTICAL AND MEDICAL RESEARCH

www.ejpmr.com

Research Article

ISSN 2394-3211

EJPMR

HIBISCUS ROSA-SINENSIS (JASUD) LEAVES EXTRACTS USED AS CORROSION INHIBITORS FOR MILD STEEL IN HYDROCHLORIC ACID

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Article Received on 02/12/2014 **Article Revised on 24/12/2014** **Article Accepted on 16/01/2015**

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ABSTRACT

To investigate the efficiency of Jasud extract as a corrosion inhibitor for mild steel in Hydrochloric acid (HCl) and to assess the effect of temperature and the inhibition efficiency. The corrosion rates were determined using the gravimetric (weight loss) and Electrochemical techniques. The results obtained in the absence and presence of Jasud extracts were used to calculate the inhibition efficiency (%I), degree of surface coverage and to propose the mechanism of inhibition and type of adsorption. Results obtained showed that inhibition efficiency (%I) increased with the increase in concentration of Jasud extracts and with the increase in temperature. Phenomenon of chemical adsorption was proposed and inhibitor found to obey Langmuir, Flory-Huggins and Freundlich adsorption isotherms. The values of Ea, ΔH°, ΔS° and ΔG° obtained revealed that the adsorption process was spontaneous. Polarization study reveals that the inhibitors functions as a mixed inhibitor. This paper provides new information on the inhibiting characteristics of Jasud leaves extract under the specified conditions. This environmentally friendly inhibitor could find possible applications in metal surface anodising and surface coatings.

KEYWORDS*:* Corrosion; Mild Steel; Hydrochloric Acid; Jasud leaves extracts, adsorption.

INTRODUCTION

Mild steel is widely used as structural material in automobiles, pipes and chemical industries, constructional materials and machines due to its low cost and excellent mechanical properties despite of its tendency to corrode in aqueous solution, especially in acidic media (Badr, 2009;

Tao et al., 2012; Döner and Kardas, 2011; Pavithra, 2012). Acid solutions are often used in the industry for industrial acid cleaning, pickling, descaling, oil well acidizing and petrochemical processes which are normally accompanied by considerable dissolution of metal (Oguzie, 2008; Keles, 2011; Amin rt al., 2007; Bayol et al., 2008; Kardas, 2005). Thus, inhibitors are one of the most convenient methods for protection against corrosion, particularly in acid solutions to prevent unexpected metal dissolution and acid consumption (Sorkhabi,2008). Most of the well known acid inhibitors are organic compounds containing hetero atoms such as nitrogen, sulfur, oxygen. Synthetic dyes such as xylenol orange (Desai and Kapopara, 2014; Desai and Vashi, 2010), synthetic drugs such as Sulphathiazole (Desai and Vashi, 2011) and heterocyclic compounds are widely used as corrosion inhibitor. The majority of the reported inhibitors used in the industry is highly toxic, so they are very hazardous to the environment, expensive and non-environmentally friendly, due to these factors their applications are limited. Due to the toxicity of some corrosion inhibitors, there has been increasing search for green corrosion inhibitors (Al-Sehaibani, 2000). Natural products of plant origin containing different organic compounds (e.g., alkaloids, tannins, pigments, organic, and amino acids) are known to inhibit action (Epke et al., 1994; Abiola and Oforka, 2002). Inhibitors in this class are those that are environment friendly, less polluting, cheap, and easily available and are obtained from natural products such as plant extracts (Avwiri and Igho, 2003). Extracts of natural products like Murraya koenigii (Quraishi, 2010), Nypa fruticans wurmb (Orubite and Oforka, 2004), Emblica officinalis (Sartha and Vasudha, 2010), Phyllanthus amarus (Okafor et al., 2010), black pepper extract (Quraish et al., 2009) , khillah seeds (El-Etre, 2006), Ficus carica (Abdel-Gaber, 2008), piper guinensis (Ebenso et al., 2008), fenugreek seeds and leaves (Noor, 2008), Nyctanthes arbortristis (Sartha and Vasudha, 2009), Caffeic acid (de Souza and Spineli,2009), etc. have been tested as corrosion inhibitors for metals. These plant extracts are low cost, nontoxic, readily available, and ecofriendly substances. The species Hibiscus (Jasud) contains about 275 species in the tropics and sub-tropics (Dasuki, 2001). The herb Hibiscus rosa-sinensi is known as JAPA in Sanskrit and is a glabrous shrub broadly cultivated in the tropics as an attractive plant and has several forms with varying colours of flowers. Leaves and flowers of Hibiscus rosa-sinensis leaves are used as an antiseptic for boils and ulcers (Ali and Ansari, 1997; Kurup and Joshi, 1979; Nadkarni, 1954). Leaves and flowers of selected Hibiscus species are used in traditional medicine. Information on their antioxidant and antibacterial activities is meagre. The leaves and flowers are observed to be promoters of hair growth.

Flowers have been found to be effective in the treatment of arterial hypertension (Dwivedi et al., 1977) and to have significant antifertility effect (Sethi et al., 1986 ; Singh et al., 1982).

Many researcher works used *hibiscus rosa-sinensis* as corrosion inhibitor for metal in acid solutions (Rajendran et al., 2009 ;Anuradha et al., 2007). Therefore, The present work was established to study the corrosion inhibition of mild steel in hcl solution by employing well known plant hibiscus rosa-sinensis leaves extract as a potential corrosion inhibitor using potentiodynamic polarization measurements and gravimetric method. The thermodynamic parameters were also obtained and discussed.

EXPERIMENTAL

Materials preparation

Mild steel contains Fe = 99.746; Mn = 0.100; C = 0.058; Al = 0.033; Si = 0.010; Cr = 0.008; Cu = 0.004; Ni = 0.0029; Mo = 0.002% were used in this study. Each sheet, which was 0.12 cm in thickness, was mechanically press-cut into coupons of dimension 3.5 x 3 cm with small hole of about 5 mm diameter near the upper edge. These coupons were used in the “as cut” condition, inhibition efficiency without further polishing, but were de-greased in absolute ethanol, dried in acetone, weighed and stored in a moisture-free desiccator prior to use.

25 gms of green leaves of Jasud crused with 25ml of water and then filter it. This filtrate was used as inhibitor with the concentration 0.25 - 1.25%.

All chemicals and reagents used were of analytical grade and used as source without further purification. The aggressive media were, respectively, 1, 2 and 3 M HCl solution. Extract of Jasud leaves were used inhibitor in the concentration range 0.25 to 1.25%,

Weight loss method

The test specimens were immersed in 1, 2 and 3 M HCl solution with and without inhibitors. Only one specimen was suspended by a glass hook, in each beaker containing 230 ml of the test solution and was open to air at room temperature for 24 h duration. After the immersion, the surface of specimen was cleaned by double distilled water followed by rinsing with acetone and sample was weighed again in order to calculate inhibiton efficiency (η %) and the corrosion rate (CR). Triplicate experiments were performed in each case and the mean values of the weight loss data are calculated.

To study the effect of temperature on corrosion rate, the test specimen was immersed in 230 ml in 1 M HCl, with extract of Jasud leaves as inhibitor concentration 0.25, 0.50, 0.75, 1.0 and 1.25% at solution temperatures of 313, 323 and 333 K for a period of 3 h. To study the effect of temperature, thermostat assembly with an accuracy of ± 0.5°C was used. Wesley (1956) and ASTM (1950) pointed out that thermostatic controls to within ± 1°C usually are considered satisfactory. Inhibition efficiency (η %), corrosion rate (C_R), energy of activation (E_a), heat of adsorption (Q_{ads}) free energy of adsorption (ΔG^0_{ads}), enthalpy of adsorption (ΔH^0_{ads}) and entropy of adsorption (ΔS^0_{ads}) were calculated and shown in Table-1.

Polarization

For polarization study, metal specimens of rectangular design having an area of 0.0932 dm^2 were exposed to corrosive solutions. Mild steel metal was used as a working electrode, SCE was used as reference electrode and the auxiliary graphite electrode was placed in a 230 ml corrosive media through which external current was supplied automatically from the computerized polarization instrument. The change in potential was measured by potentiostate / galvanostate (Gamry-Make, USA) on the potentiostate mode with 5 mg/ sec scan rate. Polarization has been taken with and without inhibitors in 1 M HCl. The curves show polarization of both the anodes and cathodes.

RESULTS AND DISCUSSION

The results are presented in Tables 1 to 2 and Figs. 1 to 8. To assess the effect of corrosion of Mild steel in HCl, ark of Jasud leaves was used as inhibitors.

The corrosion rate of mild steel in HCl was increased with the acid concentration which was shown in figure-1. The inhibition efficiency (η %) and degree of surface coverage (θ) at each concentration of ark of Jasud leaves were calculated by comparing the corrosion rate in absence (W_u) and presence of inhibitor (W_i) using the relationships:

$$\eta\% = \left(\frac{Wu - Wi}{Wi}\right) X100 \qquad (1)$$

$$\theta = \left(\frac{Wu - Wi}{Wi}\right) \qquad (2)$$

The corrosion rate of mild steel was calculated as using the relation:

$$CR\ (mpy) = \frac{534W}{DAt} \qquad (3)$$

Where, 'W' is the weight loss of mild steel in grams, 'A' is the surface area of specimen in inches square, 'D' is the density of mild steel and 't' is the time in hours.

As a constant inhibitor concentration, the inhibition efficiency decreases with the increase in acid concentration. At 1.25 % inhibitor concentration, the inhibition efficiency of ark of Jasud is 93.70, 86.63 and 78.43 % with respect to 1, 2 and 3 M acid concentration respectively. At a constant acid concentration, the inhibition efficiency of the ark of Jasud leaves increases with the inhibitor concentration, e.g. in 1 M HCl the inhibition efficiency was found to be 86.38, 86.59, 87.60, 88.82 and 93.70 % with respect to 0.25, 0.50, 0.75, 1.0 and 1.25 % inhibitor concentration respectively (Figure 1).

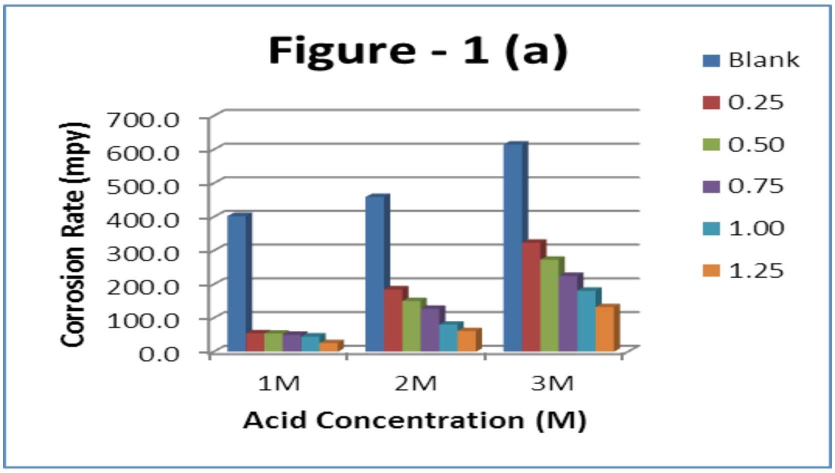


Figure:1(a) Corrosion rate of Jasud leaf extract for mild steel at different acid and inhibitor concentrations for 24 h at 301 K.

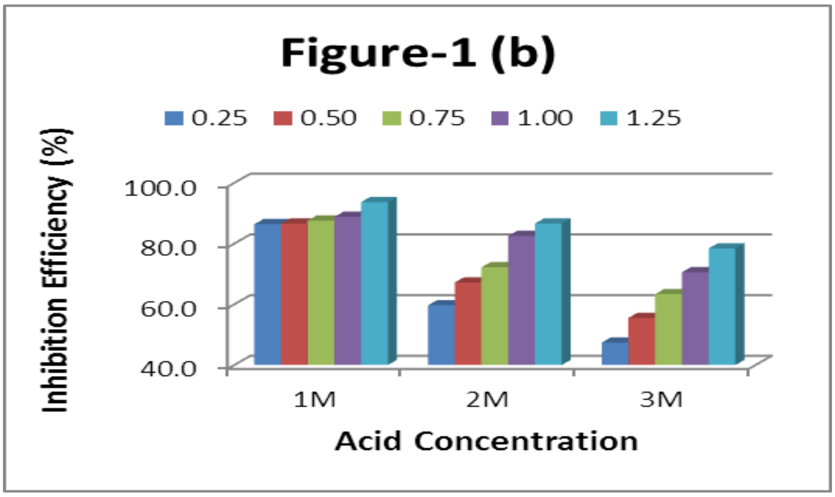


Figure:1(b) Effect of inhibition efficiency of Jasud leaf extract for mild steel at different acid and inhibitor concentrations for 24 h at 301 K.

The temperature has significant influence on a metal corrosion rates. The effect of change in temperature on the corrosion rates of mild steel in 1 M HCl, the corrosion of Ark of Jasud leaves was increased with rising temperatures. Corrosion rate was measured in 1 M HCl containing 0.25, 0.50, 0.75, 1.0 and 1.25 % inhibitor concentration at a solution temperature of 313, 323 and 333 K for an immersion period of 3 h. In 1M HCl solution with 1.25% inhibitor concentration, the inhibition efficiency for leaves of Jasud ark was decreased corresponds to 87.22, 85.78 and 84.88 % at temperature 313, 323 and 333 K respectively (Figure-2).

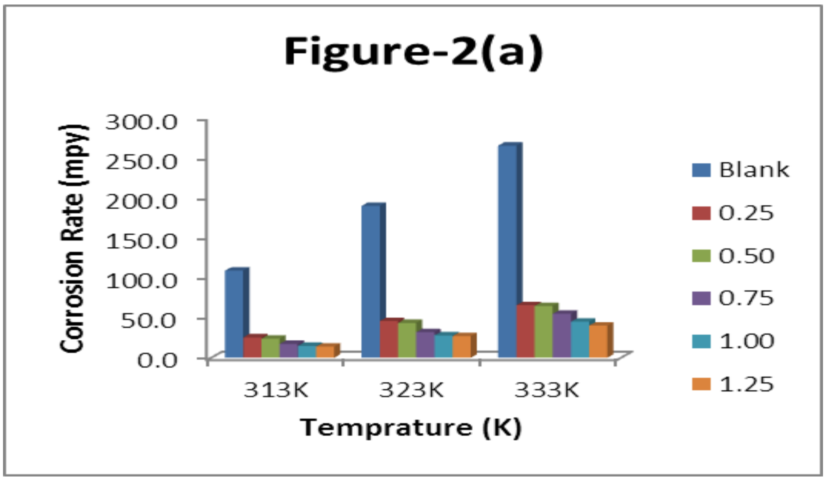


Figure 2 (a): Corrosion rate of Jasud leaf extract for mild steel in 1M HCl at different temperatures for 3h.

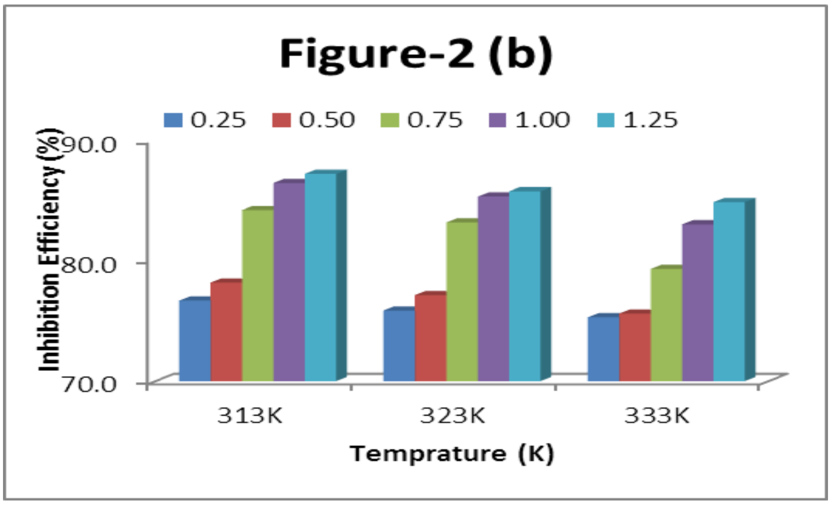


Figure 2(b): Effect of inhibition concentration of Jasud leaf extract for mild steel in 1M HCl at different temperatures for 3h.

In the present study general type of corrosion occurs predominately and less pitting. Plotting of log (θ/1-θ) versus log C, straight lines were obtained, indicating that the adsorption of the added inhibitors followed the Langmuir adsorption isotherm (Fig.3). Therefore, adsorption of these compounds is assumed to occur uniformly over the metal surface.

Plot a graph θ versus log *C* gives straight line (Figure-4) showing that the adsorption of the compound on the mild steel surface from 1 M HCl obeys also Temkin's adsorption isotherm. It is also found that the degree of adsorption of the inhibitors increases with their concentration.

For Freundlich's adsorption isotherm a plot of log **(θ)** against log C. Fig. 5, which is linear graph, was obtained, showing that the adsorption of extract of Jasud leaf on the surface of the mild steel obeys Freundlich's adsorption isotherm (Umoren et al., 2006; Umoren et al., 2008).

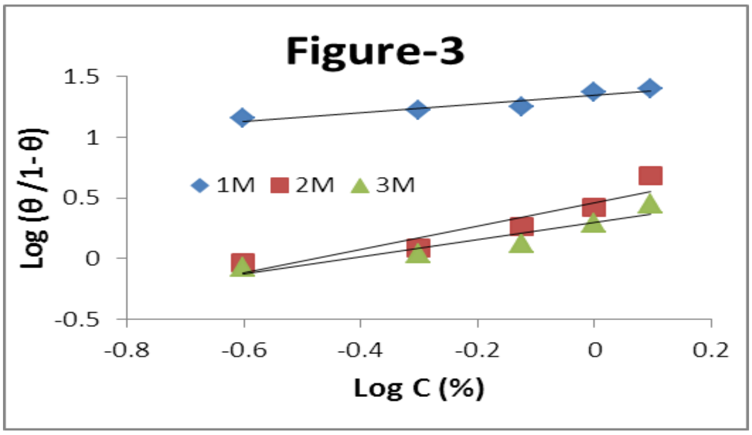


Figure 3: Plot of log (θ /1- θ) versus log C for Jasud leaf extract at different concentrations of HCl for 24 h at 301 K.

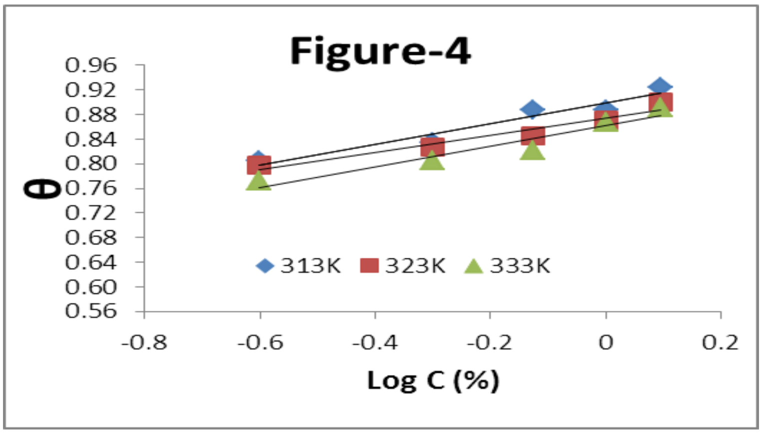


Figure 4: Plot of θ versus log C for Jasud leaf extract at different concentrations of HCl for 3h.

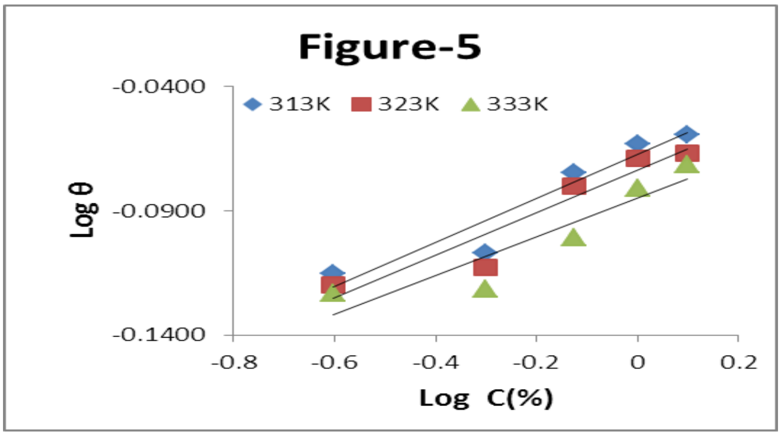


Figure 5: Plot of Log θ versus Log C for Jasud leaf extract at different concentrations of HCl for 3 h .

The values of the free energy of adsorption (ΔG^{0}_{ads}) were calculated with the slope of the following equation (Abdel and Saied, 1981).

$$LogC = Log\left(\frac{\theta}{1-\theta}\right) - LogB \qquad (3)$$

Where $LogB = -1.74 - \left(\frac{\Delta G^{0}_{ads}}{2.303RT}\right)$ and C is the inhibitor concentration. The mean ΔG^{0}_{a} values are negative almost in all cases and lie in the range of -33.59 to -36.14 kJ. mol^{-1} shown in Table-1. More negative value of ΔG^{0}_{a} shows very good inhibition efficieny. This suggests that they be strongly adsorbed on the metal surface. This statement was supported by the work of Talati and Darji (1988). The values of heat of adsorption (Q_{ads}) were calculated by the following equation.

$$Q_{ads} = 2.303R\left[Log\left(\frac{\theta_2}{1-\theta_2}\right) - Log\left(\frac{\theta_1}{1-\theta_1}\right)\right] X \left[\left(\frac{T_1T_2}{T_2-T_1}\right)\right] \qquad (4)$$

From Table -1, it is evident that in all cases, the (Q_{ads}) values are negative and ranging from -2.68 to -22.78 kJ. mol^{-1}. The negative values show that the adsorption, and hence the inhibition efficiency, decreases with a rise in temperature (Bhajiwala and vashi, 2001).

Mean 'E_a' value was calculated by using equation (5) for mild steel in 1M HCl is 38.32 kJ.mol^{-1} while in acid containing inhibitor, the mean E_a values are found to be higher than that of an uninhibited system (Table 1). Higher values of E_a in the presence of the extract which acts as inhibitor is a good indication of strong inhibiting action of the extract by increasing the energy barrier for the corrosion process. Higher values of E_a in the presence of extract can also be correlated with the increase in thickness of the double layer that enhance the E_a of the corrosion process (Singh et al., 2008). The values of E_a calculated from the slope of an Arrhenius plot (Figure 6) and using equation (5) are almost similar. Energy of activation (E_a) has been calculated from the slopes of log p versus 1/T (p = corrosion rate, T = absolute temperature) and also with the help of Arrhenius equation.

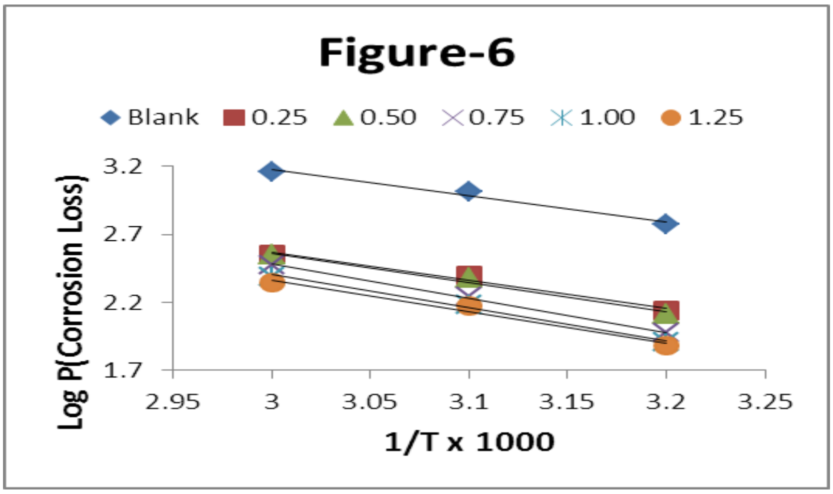


Figure 6: Arrhenius plots for corrosion of mild steel in 1 M HCl in absence and presence of Jasud leaf extract.

$$Log\frac{P_2}{P_1} = \frac{Ea}{2.303R}\left[\left(\frac{1}{T_1}\right)-\left(\frac{1}{T_2}\right)\right] \qquad (5)$$

Where P_1 and P_2 are the corrosion loss at temperature T_1 and T_2 respectively. The enthalpy of adsorption (ΔH^0_{ads}) and entropy of adsorption (ΔS^0_{ads}) were calculated using the following equation (6) and (7).

$$\Delta H^0 ads = Ea - RT \qquad (6)$$

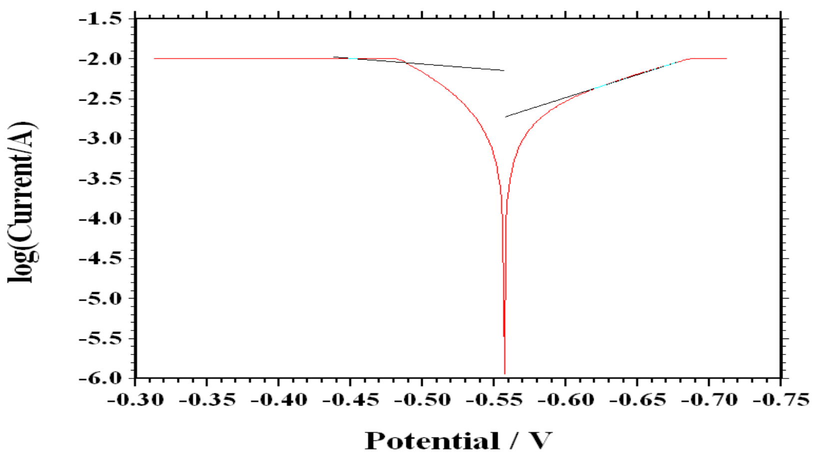
$$\Delta S^0{}_{ads} = \frac{\Delta H^0{}_{ads} - \Delta G^0{}_{ads}}{T} \qquad (7)$$

The enthalpy changes (ΔH^0_a) are positive, indicating the endothermic nature of the reaction suggesting that higher temperature favors the corrosion process (Agrawal et al., 2003). The entropy (ΔS^0_a) values are positive, confirming that the corrosion process is entropically favorable (Issa et al., 2008).

Table-1—Energy of activation (E_a), heat of adsorption (Q_{ads}) and free energy of adsorption (ΔG^0_{ads}) for mild steel in 1 M HCl containing inhibitors.

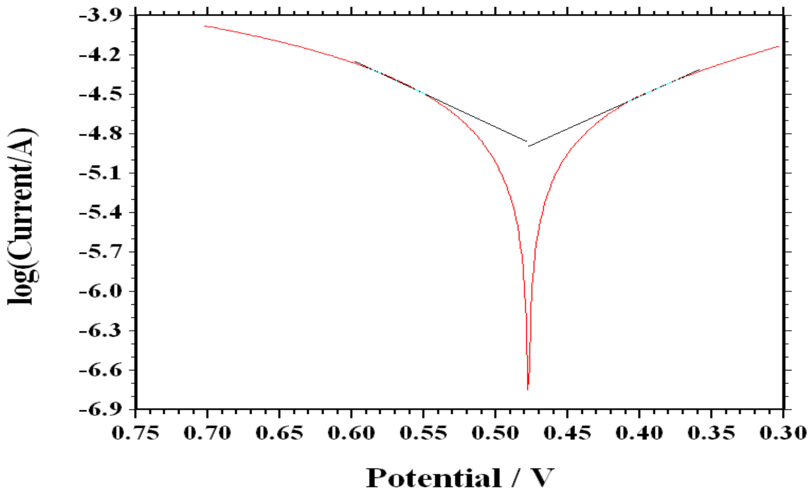
Inhibitor Concentration	Mean E_a from Eq. (2) (kJ.mol^{-1})	E_a from Arrhenius plot (kJ.mol^{-1})	Q_{ads} (kJ.mol^{-1})		Mean value (kJ.mol^{-1})		
			313 –323 (K)	323-333 (K)	ΔG^0_{ads}	ΔH^0_{ads}	ΔS^0_{ads}
Blank	38.32	37.02	-	-	-	35.68	-
0.25	40.81	39.42	-3.86	-2.68	-36.14	38.16	0.2325
0.50	43.20	41.67	-5.05	-7.62	-34.43	40.55	0.2348
0.75	50.22	48.24	-6.30	-22.78	-34.22	47.58	0.2565
1.00	48.24	46.44	-7.79	-15.60	-33.98	45.60	0.2495
1.25	45.56	44.01	-10.39	-6.42	-33.59	42.92	0.2399

Anodic and Cathodic polarization curves for mild steel in 1M HCl at 1.25 % inhibitor concentration of the presence and absence of inhibitors are shown in Fig.7. The value of the corrosion potential with inhibitors were found become more positive than the without inhibitors. Polarization study reveals that the inhibitors function as little anodic, but significant cathodic inhibitors, inhibitor functions as a mixed inhibitor. It is evident from the figure that cathodic tafel slopes (βc) remain almost unchanged with increasing inhibitor. This indicates that hydrogen evolution is activation controlled and the addition of inhibitor did not change the mechanism of cathodic hydrogen evolution reaction (Ateya et al., 1976; Li et al., 2008). The values for the Tafel parameters obtained from this plot with and without inhibitors are given in Table-2.



(a)

Figure 7:(a) Polarisation curves for corrosion of mild steel in 1 M Hcl without inhibitors.



(b)

Figure 7:(b) Polarisation curves for corrosion of mild steel in 1 M Hcl containing 1.25% Jasud leaf extract inhibitors.

The values of corrosion current densities in the presence and absence of inhibitor were obtained from the graph while percentage efficiency (η %) was calculated using the Equation (8) and (9). The inhibition efficiency from Tafel plots agrees well (within ± 6 %) with the values obtained from weight loss data.

$$\eta(\%) = \left[\frac{i_{corr}(u) - i_{corr}(i)}{i_{corr}(u)}\right] \times 100 \qquad (8)$$

The linear polarization study was carried out from cathodic potential versus OCP to an anodic potential of versus OCP with a sweep rate 0.01 V S^{-1} to determine the polarization resistance ($R_p^{'}$). From the measured polarization resistance value, the inhibition efficiency has been calculated using the relationship:

$$\eta\% = \frac{Rp^{'} - Rp^{0}}{Rp^{'}} X\ 100 \qquad (9)$$

Where R_p^0 and $R_p^{'}$ are the polarization resistance in the absence and in the presence of inhibitor, respectively.

Mild steel dissolves in acid solutions due to the hydrogen evolution type of attack. The reaction-taking place at the micro electrodes of the corrosion cell being represented as under,

$$Fe \longrightarrow Fe^{+3} + 3e^{-} \qquad (10)$$

$$Fe^{+2} \rightarrow Fe^{+3} + e^{-} \qquad (11) \quad \text{(anode)}$$

$$H^{+} + e^{-} \rightarrow H \qquad (12) \quad \text{(cathode)}$$

Followed by the reaction

$$H + H \rightarrow H_2 \qquad (13)$$

The following secondary reaction can also take place in acid solutions (Godard et al., 1967).

$$2M + 2H^+ \rightarrow H_2 + 2M^+ \qquad (14) \quad \text{(anode)}$$

$$O_2 + 4H^+ + 4e^- \rightarrow 2H_2O \qquad (15) \quad \text{(cathode)}$$

Therefore, only if the hydrogen evolution type of attack is predominate and no other factors influence the corrosion process, corroded by the strong acid should be maximized.

The inhibitory mechanism is a separation process involving (i) the inhibitor is adsorbed on the surface of the metal forming a compact protective thin layer and (ii) the inhibitor forms a precipitate on the surface of the metal, acting on the aggressive media to form protective precipitates or remove the aggressive agents (El-Sayyed et al., 2010).

The chemical composition of hibiscus rosa-sinensis contain majorly Anthocyanins & flavonoids ; cyanidin-3,5-diglucoside, cyanidin-3-sophoroside-5-glucoside, quercetin-3,7-diglucoside, quercetin- 3-diglucoside (Gupta et al., 2005). And the other A cyclopeptide alkaloid (Khokhar and Ahmed, 1992), cyanidin chloride, quercetin, hentriacontane (Shrivastava, 1974) and vitamins : riboflavin, ascorbic acid & thiamine (The Welth of India, 1992). Alkaloid, vitamins and flavonoids are aromatic heterocyclic compounds and responsible to prevent corrosion process. Also the de-localized ת- electrons of ark of hibiscus rosa-sinensis leaves facilitate its strong adsorption on the mild steel surface leading to the outstanding corrosion inhibition.

Table 2— Polarization data and inhibition efficiency of Jasud for mild Steel in 1 M HCl. Inhibitor concentration: 1.25% Jasud; Effective specimen area: 0.0932 dm²

System	E_{corr} (mV)	I_{corr} (A/cm²)	*Rp* (ohm)	Tafel slope (V/decade)			Inhibition efficiency (%)		
							Pol. Method		Wt. loss method
				-βc	+βa	β	From I_{corr}	From *Rp*	
Blank	-557	4.866 x 10^{-03}	12	5.751	1.436	2.64	-	-	_
Jasud	-479	1.806 x 10^{-05}	2398	4.929	5.110	5.77	99.63	99.49	93.70

CONCLUSIONS

As a constant inhibitor concentration, the inhibition efficiency of inhibitors decreases as the concentration of acid increases. At all concentration of acid, as the inhibitor concentration increases inhibition efficiency increases and corrosion rate decreases. As the temperature increases corrosion rate increases in plain acid. Addition of inhibitors in corrosive media indicates that as the temperature increases corrosion rate increases while inhibition efficiency decreases. In all cases, the value of heat of adsorption (Q_{ads}) and the value of free energy of adsorption (ΔG^0_a) is negative.The Value of change of enthalpy (ΔH^0_a) and entropy of adsorption (ΔS^0_a) is positive. A mean value of 'Ea' in inhibiting acid is higher than the value of 'Ea' in acid only. In almost all the cases, the inhibition efficiency from Tafel plots agrees well (within ± 6 %) with the values obtained from weight loss data.

ACKNOWLEDGEMENT

The author is thankful to the University Grant Commission (Western region office), Pune for providing a financial support and also thanks full to Department of Chemistry, Arts, Science and Commerce College, Kholwad, Surat for providing laboratory facilities.

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