

Effect of Temperature on Inhibitive Propensity of *Prosopis cineraria* Leaves Extract on Acid Corrosion of Aluminium

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Abstract

An attempt has been made to investigate the anti-corrosive propensity of ethanolic extract of *Prosopis cineraria* leaves at elevated temperatures, in the range 303 to 353 K, so as to replace the use of toxic chemicals as corrosion inhibitors. Mitigation of aluminium corrosion in 0.5M hydrochloric acid was studied in absence and presence of *Prosopis cineraria* by using chemical method. The inhibition efficiency is found to increase with increasing concentration of extract and decreases with rise in temperature. The maximum inhibition efficiency 86.67% was obtained at 303K. The adsorption of inhibitor on aluminium surface was observed to be exothermic, physical, and spontaneous. Langmuir adsorption isotherm was found to be best fitted. Thermodynamic parameter ΔG_{ads} , activation parameters E_{act} , ΔH_{act} and ΔS_{act} were evaluated. These thermodynamic and kinetic parameters indicate a strong interaction between the inhibitor and the metal surface.

Keywords: Aluminium, acid corrosion, green inhibition, *Prosopis cineraria*, adsorption.

1. Introduction

Aluminium, a lightweight metal (density = 2.71 g/cm³), and its alloy are recommended for building purpose and for various internal outfits, at various industries and highly polluted places. Aluminium is used in fabricating various reaction vessels, reaction tanks, and pipes etc, for industrial uses due to their availability and low cost. However when used in aggressive media they tend to corrode causing severe loss and malfunctioning of industrial equipment. Therefore, it is necessary to protect from getting corrode and the best suited method is employing inhibitors. Now-a-days, due to environmental regulations, the organic or inorganic inhibitors causing environmental threat must be restricted and must be replaced by *green inhibitors*. In present study, *Prosopis cineraria* leaves' extract has been used to inhibit the acid corrosion of aluminium.

Prosopis cineraria is a prickly tree or shrub and commonly found in dry and arid regions of North Western India, Southern India, Pakistan, Afghanistan and Arabia. Leave are extensively used as fodder for cattle, goats and camel [1-2]. Leaves contain specigerine, steroids such as campesterol, cholesterol, sitosterol, stigmasterol, tricosan-1-ol, methyl docosanoate [3-4]; whereas leaves and pods

consists aspartic acid, glutamic acid, serine, glycine, histidine, threonine, arginine, alanine, proline, tyrosine, valine, methionine, cysteine, isoleucine, leucine, phenylalanine and lysine [5,6].

2. Experimental

2.1 Preparation of *Prosopis cineraria* leaves extract
Air and shade dried leaves of *Prosopis cineraria* were grinded and powdered. The finely powdered dried material was taken in 500 ml round bottom flask and soaked in sufficient quantity of distilled ethyl alcohol. Thereafter, it was extracted by standard procedure [7, 8].

2.2 Preparation of test coupons

Rectangular coupons of aluminium (3×2.4×0.16 cm³) were used with a small hole of 2mm diameter near upper edge for hanging in test media. Prior to experimentation, each coupon was given surface treatment as per standard procedure [7, 8].

2.3 Test solutions

Bidistilled water was used to prepare test electrolyte of 0.5 M HCl (AnaL R grade). The surface treated aluminium coupons were immersed in separate beakers containing corrosive solutions and different concentrations of inhibitor (*Prosopis cineraria* leaves extract) (EEPc) (ranging from 0.0744 to 1.0426 g/L) for desired time periods at room and elevated temperature. Before measurement, each coupon was surface treated, dried in desiccators and then weighed. From the chemical (weight loss) method, various corrosion parameters, viz., inhibition efficiency, corrosion rate, fractional surface coverage, adsorption equilibrium constant etc. were calculated using standard equation [7-8].

3. Results and Discussion

The effect of temperature on the stability and mechanism of adsorption of the EEPcL on the aluminium surface is studied using chemical method and various corrosion parameters, viz. corrosion rates, inhibition efficiency IE (%), fractional surface coverage (θ) and adsorption equilibrium constant (K_{ad}) were evaluated in 0.5M HCl

between temperature range 303-353K in the absence and presence of EEPcL at 24 hrs immersion period.

3.1 Effect on corrosion rates and inhibition efficiency IE (%)

The results have been tabulated (Table-1) and (figs. 1-3) which clearly indicate that corrosion rate was increased with reduction in inhibitory efficacy at elevated temperatures. Loss in coupon weight (0.018g at 303K and 0.3423g at 353K) was observed in blank solution (without inhibitor), while loss in weight was reduced (0.0024g at 303K and 0.1104g at 353K) in additives presence at its highest concentration (1.064g/L). Increase in corrosion rate at elevated temperatures may be due to increased rate of dissolution process of aluminium and partial desorption of the inhibitor from the metal surface with elevation in temperature.

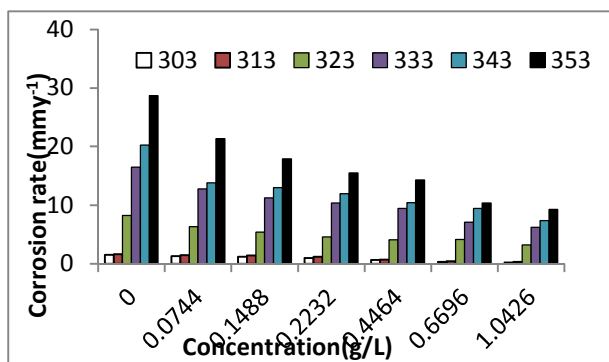


Fig.1: Corrosion rates for aluminium in 0.5M HCl in the absence and presence of different concentrations of EEPcL at different temperatures

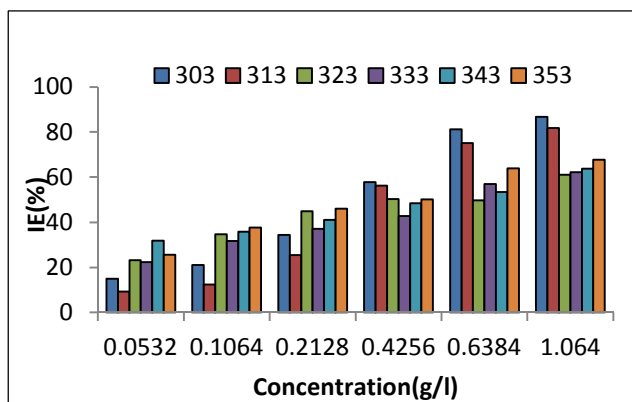


Fig.2: Inhibition efficiency (IE%) of EEPcL at its various concentrations at elevated temperatures

Inhibition efficiency increases on increasing concentration of EEPcL at all elevated temperatures. Generally elevation in temperature decreases the inhibitive process, here too, with the elevation in temperature (up to 323K), a decreased in IE% (from highest 86.67% at 303K to

60.98% at 323 K) was observed at inhibitor concentration (1.064 g/L); while on further rise in temperature, increase in inhibitory efficacy of extract was observed (60.98% at 323K to 67.75% at 353K).

Table: 1. Corrosion parameters of acid corrosion of aluminium without and with different concentrations of EEPcL at elevated temperatures for 24 hrs immersion period.

Temp (K)	EEPc Conc. (g/L)	Corrosion parameters			
		Corros. rate (ρ_{corr}) (mm.y ⁻¹)	IE%	Fractional surface coverage (θ)	Adsorption equilibrium constant (K_{ad})
303	P0 (Blank)	1.509	-	-	-
	P1 (0.0532)	1.282	15.00	0.150	3.317
	P2 (0.1064)	1.190	21.11	0.211	2.515
	P3 (0.2128)	0.989	34.44	0.344	2.469
	P4 (0.4256)	0.637	57.78	0.578	3.215
	P5 (0.6384)	0.285	81.11	0.811	6.726
	P6 (1.064)	0.201	86.67	0.867	6.109
313	P0 (Blank)	1.609	-	-	-
	P1 (0.0532)	1.456	9.38	0.094	1.945
	P2 (0.1064)	1.408	12.50	0.125	1.343
	P3 (0.2128)	1.197	25.52	0.255	1.610
	P4 (0.4256)	0.704	56.25	0.563	3.021
	P5 (0.6384)	0.402	75.00	0.750	4.699
	P6 (1.064)	0.293	81.77	0.818	4.216
323	P0 (Blank)	8.248	-	-	-
	P1 (0.0532)	6.337	23.17	0.232	5.669
	P2 (0.1064)	5.381	34.76	0.348	5.007
	P3 (0.2128)	4.543	44.92	0.449	3.832
	P4 (0.4256)	4.099	50.30	0.503	2.379
	P5 (0.6384)	4.149	49.70	0.497	1.547
	P6 (1.064)	3.219	60.98	0.610	1.469
333	P0 (Blank)	16.462	-	-	-
	P1 (0.0532)	12.766	22.45	0.225	5.443
	P2 (0.1064)	11.249	31.67	0.317	4.356
	P3 (0.2128)	10.343	37.17	0.372	2.780
	P4 (0.4256)	9.413	42.82	0.429	1.760
	P5 (0.6384)	7.083	56.98	0.570	2.074
	P6 (1.064)	6.220	62.22	0.622	1.548
343	P0 (Blank)	20.226	-	-	-
	P1 (0.0532)	13.772	31.91	0.320	8.809
	P2 (0.1064)	12.967	35.89	0.359	5.261
	P3 (0.2128)	11.928	41.03	0.410	3.269
	P4 (0.4256)	10.436	48.40	0.484	2.204
	P5 (0.6384)	9.413	53.46	0.535	1.799
	P6 (1.064)	7.334	63.74	0.637	1.652
353	P0 (Blank)	28.691	-	-	-
	P1 (0.0532)	21.316	25.71	0.257	6.505
	P2 (0.1064)	17.887	37.66	0.377	5.677
	P3 (0.2128)	15.473	46.07	0.461	4.015
	P4 (0.4256)	14.317	50.10	0.501	2.359
	P5 (0.6384)	10.360	63.89	0.639	2.772
	P6 (1.064)	9.254	67.75	0.678	1.974

This rise in IE% was probably due the orientation of phytochemicals of the extract so as to onset the chemical adsorption at elevated temperatures (333, 343 and 353K) (fig. 2-3).

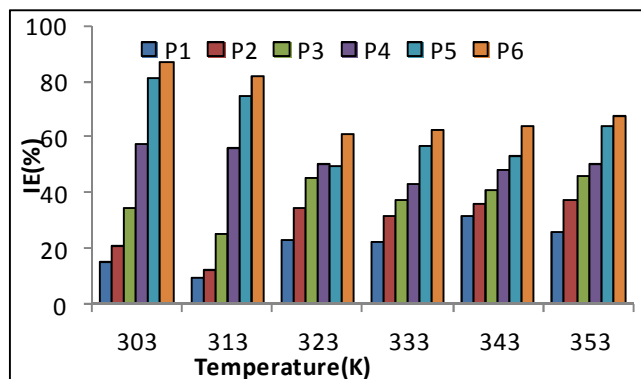


Fig. 3: IE (%) of EEPcL at its different concentrations at elevated temperatures

From fig. 3, it is clear that there is no consistent trend in IE % as it was seen in the case of corrosion rates, however, for each elevation in temperature, the IE% increases with an increase in EEPcL concentration. The decrease in IE% and θ at 323 K may be explained as a result of increase in average kinetic energy of components of extracts on increasing temperature, thus making adsorption between components of extracts and aluminium surface insufficient to retain the species at binding site. This could lead to desorption or cause species to bounce off from surface of aluminium instead of colliding and combining with it. Therefore, increase in temperature may be associated with decrease in stability of components – aluminium surface complex.

3.2 Kinetic parameters

Corrosion rate of Aluminium against the concentration of the inhibitor obeys the following kinetic relationship [9].

$$\log \rho_{\text{corr}} = \log k + B \log C_{\text{inh}}$$

where k is the rate constant and B is the reaction constant which is a measure for the inhibitor effectiveness and C_{inh} is concentration of inhibitor. Plots of $\log \rho_{\text{corr}}$ versus $\log C_{\text{inh}}$ at various immersion time intervals are illustrated in fig. 4. Kinetic parameters k and B were calculated using above equation and tabulated in Table- 2. The negative sign for the values of reaction constant B indicates that the rate of corrosion process is inversely proportional to the inhibitor concentration, meaning that the inhibitor becomes more effective with increasing its concentration. Hence, when the change of ρ_{corr} with inhibitor concentration becomes steep (high negative value for constant B) it reflects good inhibitive properties for the studied inhibitor.

It is also observed that the value of k increases with temperature (table-2).

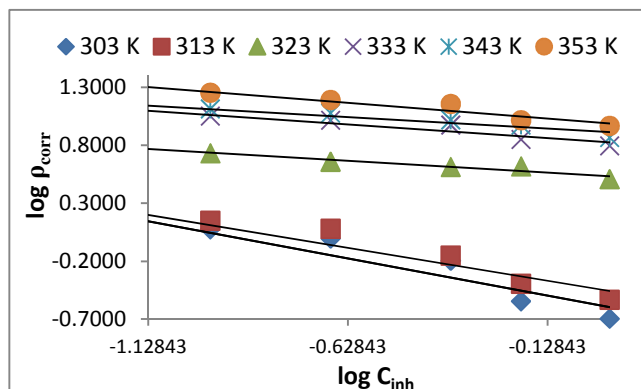


Fig. 4: Plot of $\log \rho_{\text{corr}}$ with $\log C_{\text{inh}}$ at various elevated temperatures.

Table: 2 Kinetic parameters for corrosion of aluminium in 0.5 M HCl

Temperature (K)	Kinetic parameters	
	B	k
303	-0.641	0.2636
313	-0.567	0.3614
323	-0.202	3.4435
333	-0.234	6.7453
343	-0.196	8.2604
353	-0.274	9.8401

3.3 Activation studies

Elevation in temperature has great effect on the corrosion phenomenon. Corrosion rate increases with rise in temperature (tables 1 and 3). The dependence of corrosion rate on temperature can be expressed by the Arrhenius equation [10-12]:

$$\log \rho_{\text{corr}} = \log A - E_{\text{act}}/2.303RT$$

where ρ_{corr} is the corrosion rate, E_{act} is the activation energy, R is the molar gas constant, T is the absolute temperature and A is the frequency factor.

A plot of $\log \rho_{\text{corr}}$ versus $1/T$ gave straight lines (fig. 5). E_{act} was calculated from slopes of $\log \rho_{\text{corr}}$ versus $1/T$. In presence of EEPcL the value of E_{act} increases as compared to blank solution (58.84 KJmol^{-1}), thus endorsing the adsorption of EEPcL on the test coupon surface. It was observed that the activation energy E_{act} for aggressive medium alone (blank) was lower than in inhibited medium. The activation energy of EEPcL ranged from 58.84 to 75.77 KJ/mol . Higher values of E_{act} in the presence of EEPcL indicate physical adsorption of the active molecules of inhibitor on to the metal surface, while lower values that in cases of chemisorption [9]. The value of E_{act} increases with the increase in EEPcL concentration.

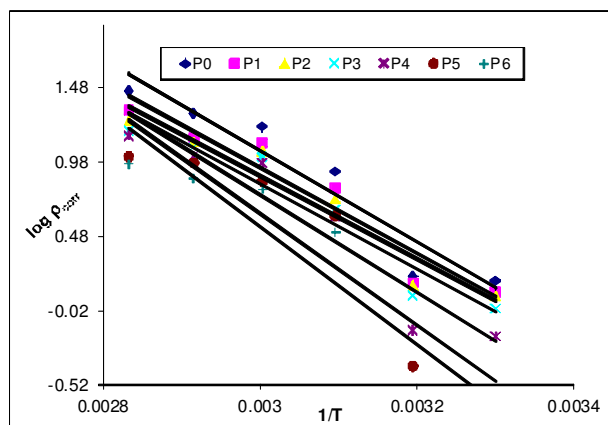
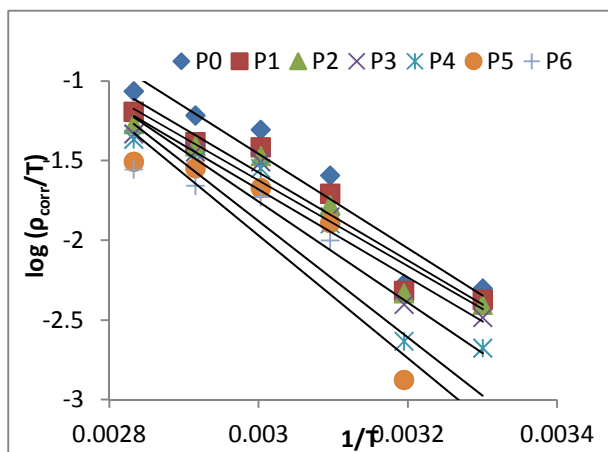

 Fig. 5: $\log \rho_{\text{corr}}$ versus $1/T$

Table: 3 Kinetic-thermodynamic corrosion parameters for aluminium acid corrosion without and with various concentrations of EEPcL.

Concen. (g/L)	ΔH_{act} (KJmol^{-1})	ΔS_{act} ($\text{JK}^{-1}\text{mol}^{-1}$)	E_{act} (KJmol^{-1})
P0 Blank	56.83	-54.99	58.84
P1 0.0532	52.94	-68.89	54.95
P2 0.1064	51.56	-73.98	53.57
P3 0.2128	52.92	-71.00	54.93
P4 0.4256	60.68	-49.15	62.71
P5 0.6384	69.91	-23.78	71.94
P6 1.0640	73.74	-14.03	75.77


 Fig. 6: $\log (\rho_{\text{corr}} / T)$ versus $1/T$

The positive sign for both E_{act} and ΔH_{act} , indicate the endothermic nature of corrosion process. The values of E_{act} and ΔH_{act} vary in the similar way. It is obviously seen that the activation energy strongly increases in the presence of the inhibitor. Other kinetic parameters of the corrosion reaction, namely, entropy ΔS_{act} and enthalpy ΔH_{act} of activation transition state are calculated by using transition state equation [12-13]:

$$\rho_{\text{corr}} = \left(\frac{RT}{Nh} \right) e^{\left(\frac{\Delta S_{\text{act}}}{R} \right)} e^{\left(\frac{-\Delta H_{\text{act}}}{RT} \right)}$$

A plot of $\log (\rho_{\text{corr}}/T)$ versus $1/T$ give a straight lines (fig. 6) from which the values of ΔS_{act} and ΔH_{act} were calculated. All thermodynamic parameters of the corrosion reaction have been tabulated (Table-3). The entropy of activation (ΔS_{act}) in the absence and presence of inhibitor has negative values. This indicates that the activated complex in the rate determining step represents an association rather than dissociation, meaning that, a decrease in disordering takes place on going from reactants to the activated complex for all systems [12-13]. The average value of the difference ($E_{\text{act}} - \Delta H_{\text{act}}$) is about 2.03 kJmol^{-1} which approximately around the average value of RT (2.69 kJ mol^{-1}); where T is in the range of the experimental temperatures, indicating that the corrosion process is a unimolecular reaction as it is characterized by the equation: $E_a - \Delta H_{\text{act}} = RT$.

3.4 Thermodynamic adsorption parameters

The thermodynamic adsorption parameters such as the free energy of adsorption (ΔG_{ads}), the heat of adsorption (ΔH_{ads}) and the entropy of adsorption (ΔS_{ads}) were also calculated from the experimental data. The ΔG_{ads} values at all studied temperature can be calculated from the equation: $\Delta G = -2.303 RT \log(K_{\text{ad}} \times 55.5)$ where 55.5 is the concentration of water in ml/L.

Other adsorption parameters ΔH_{ads} and ΔS_{ads} are obtained from the equation [14-15]:

$$\Delta G_{\text{ads}} = \Delta H_{\text{ads}} - T\Delta S_{\text{ads}}$$

Straight line was obtained on plotting ΔG_{ads} versus T ; the value of ΔS_{ads} was obtained from the slope and the intercept leads to ΔH_{ads} (Table-4).

The negative values of ΔG_{ads} ensure the spontaneity of the adsorption process and stability of the adsorbed layer on the metal surface. Generally, values of ΔG_{ads} up to -20 KJ/mol are consistent with electrostatic interaction between the charged molecules and the charged metal (physisorption), while those around -40 KJ/mol or higher are associated with chemisorptions as a result of sharing or transfer of electrons from the organic molecules to the metal surface to form a coordinate type of bond. In general, two kinds of adsorption can be considered; physical adsorption or chemical adsorption and sometimes both of these. It has been found that values of ΔH_{ads} are negative, for higher inhibitor concentration suggesting that the adsorption of inhibitor is an exothermic process. Basically, an exothermic process means either physisorption or chemisorptions, while an endothermic process is associated to chemisorption. In an exothermic process, physisorption is distinguished from chemisorption by considering the absolute value of a physisorption process. In physisorption,

the values are lower than 40 KJ/mol while the adsorption heat of chemisorptions process approaches 100 KJ/mol. In the 0.5M HCl, an exothermic adsorption was detected at higher inhibitor concentration. The positive values of ΔH_{ads} indicate endothermic adsorption with a disordered phenomenon as indicated by positive values of ΔS_{ads} , this disorderliness decreases at higher concentration. The decrease in entropy is clearly indicative for an association rather than dissociation at higher concentration. This order or association may be explained by the possibility of formation of complex with inhibitor, inhibitor molecules may freely move in the bulk of solution before the adsorption process, while with progress in adsorption the inhibitor molecules were orderly adsorbed on the metal surface, which resulted in the decrease in entropy [13].

Table: 4 Thermodynamic parameters for the adsorption of EEPcL on aluminium in 0.5M HCl at different Temperatures.

ΔG_{ads} KJ/mol at 303- 353 K	Conc. (g/L)					
	P1	P2	P3	P4	P5	P6
	0.0532	0.1064	0.2128	0.4256	0.6384	1.0640
303 K	-13.141	-12.444	-12.397	-13.062	-14.922	-14.680
313 K	-11.795	-10.860	-11.320	-12.905	-14.019	-13.745
323 K	-15.448	-15.110	-14.396	-13.115	-11.960	-11.820
333 K	-15.813	-15.197	-13.953	-12.686	-13.142	-12.331
343 K	-17.147	-15.719	-14.402	-13.310	-12.748	-12.512
353 K	-16.307	-15.930	-14.970	-13.498	-13.945	-13.005
ΔH_{ads} KJ mol ⁻¹	15.28	15.85	6.734	-10.31	-20.50	-23.85
ΔS_{ads} JK ⁻¹ mol ⁻¹	92	91	61	08	21	33

3.5 Adsorption considerations

Adsorption isotherms are usually used to describe the adsorption process. In order to obtain the adsorption isotherm, the degree of surface coverage (θ) for various concentrations of the inhibitor has been calculated applying the following equation (4). Assuming that the adsorption of EEPcL extract molecules was mainly due to a monolayer adsorption and ignoring the interaction between the adsorbed molecules, then the Langmuir adsorption isotherm can be employed. Langmuir isotherm [16-17] was tested for examine fitness to the experimental data. Langmuir isotherm is expressed as:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ad}} + C_{inh} \quad (1)$$

where θ is the degree of surface coverage, C_{inh} the molar inhibitor concentration in the bulk solution and K_{ad} is the equilibrium constant of the process of adsorption. The plot of C_{inh}/θ versus C_{inh} was linear. Plot of C_{inh}/θ against C_{inh} gave straight lines (fig. 7) which clearly show that inhibitory action of EEPcL obeys Langmuir adsorption

isotherm. The correlation coefficient, slopes, and adsorption coefficients obtained from Langmuir isotherm plots were tabulated (Table-5). The free energy of adsorption, ΔG_{ads} , is associated with water adsorption equilibrium. ΔG_{ads} was calculated using equation (2):

$$K = \frac{1}{(55.5)e^{\left(\frac{-\Delta G_{ads}}{RT}\right)}} \quad (2)$$

The negative value of ΔG_{ads} , indicate that the adsorption process on aluminium surface is spontaneous. The correlation coefficient (R^2) and slope obtained was nearly equal to unity best fitted in experimental data. ΔG_{ads} value indicated physical adsorption.

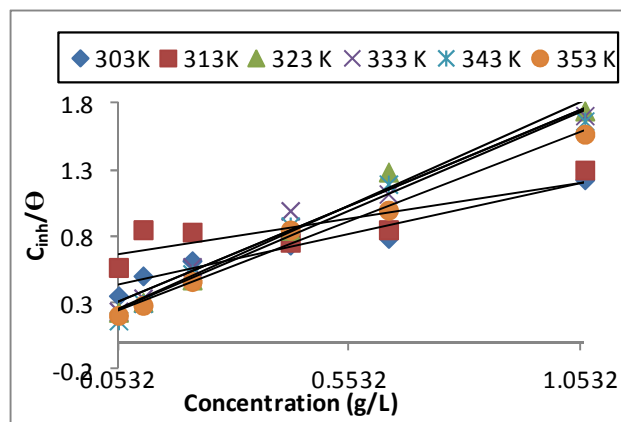


Fig. 8: Langmuir adsorption isotherm of EEPcL for aluminium coupons

Table: 5 Correlation coefficient, slopes and ΔG_{ads} from Langmuir adsorption.

Temp(K)	Slope	R^2	K_{ad}	ΔG_{ads} (KJmol ⁻¹)
303	0.764	0.955	2.59	-12.5181
313	0.537	0.732	1.57	-11.6315
323	1.553	0.987	6.02	-15.6109
333	1.431	0.977	4.33	-15.1792
343	1.482	0.983	5.92	-16.5264
353	1.339	0.988	5.88	-16.9909

4. Conclusions

Following conclusions may be drawn on the basis of the results obtained from study of the effect of elevation in temperature on the inhibitory efficacy of *Prosopis cineraria* leaves extract on acid corrosion of aluminium in 0.5 M HCl:

- *Prosopis cineraria* Leaves (EEPcL) can be good inhibitor to impede acid corrosion of aluminium in 0.5M HCl at 303K with maximum inhibition efficiency of 86.67% at its 1.064g/L concentration.

- Elevation in corrosion rate at high temperature, but with the additive, a reasonable decrease in corrosion rate was observed.
- IE % increased with increase in the concentration of inhibitor and at elevated temperature (353K) the inhibitor could impede reasonably good, yielding 67.75 % IE.
- The negative sign for reaction constant B indicate rate of corrosion process being inversely proportional to the inhibitor concentration, thus the inhibitor becomes more effective with increasing its concentration.
- Activation energy E_{act} in aggressive medium alone (blank) was observed much lower as compared to that in inhibited test solutions. Activation energy of EEPcL was found in the range of 58.84 to 75.77 KJ/mol and much high values in the presence of EEPcL inhibitor thus indicating physical adsorption of the inhibitor on the metal surface.
- The adsorption of the extract on the aluminium surfaces was spontaneous and obeyed Langmuir adsorption isotherms at elevated temperatures.
- At higher concentrations of inhibitor, negative heat of adsorption was obtained which indicate adsorption.
- Entropy of adsorption decreases with increase in EEPcL indicates association of inhibitor molecules.
- Overall, it can be concluded that *Prosopis cineraria* can be used as *green inhibitor* to replace toxic chemicals used to impede aluminium corrosion in 0.5 M HCl at elevated temperatures up to 353 K.

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