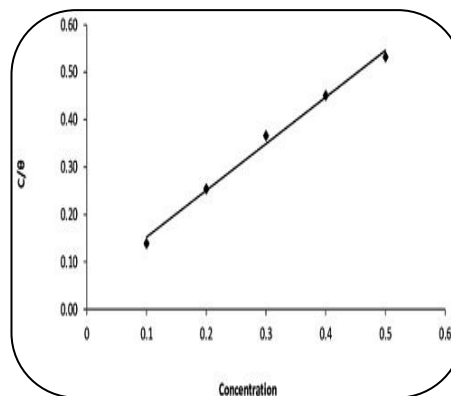_____

EFFECT OF TEMPERATURE ON INHIBITION PROCESS OF *PROSOPIS CINERARIA* LEAVES ON ALUMINIUM SURFACE IN 0.5M HYDROCHLORIC ACID

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ABSTRACT:

Ethanolic extract of Prosopis cineraria leaves behaves as a green corrosion inhibitor to replace toxic chemicals. Inhibition of corrosion of aluminium in 0.5M hydrochloric acid solution was studied in absence and presence of Prosopis cineraria by using chemical method at different temperatures ranged from 30 to 80 $\square$C. 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 30˚C. The activation energy, thermodynamic parameters (free energy, enthalpy and entropy change) and kinetic parameters for inhibition process were also calculated. These thermodynamic and kinetic parameters indicate a strong interaction between the inhibitor and the metal surface. The adsorption of inhibitor on aluminium surface is exothermic, physical, and spontaneous. The Langmuir adsorption isotherm was used to calculate the thermodynamic parameter ΔG_{ads}. The activation parameters E_{act}, ΔH_{act} and ΔS_{act} were calculated using Arrhenius equation.

KEY WORDS: *Inhibition, Corrosion, Adsorption, Aluminium, Prosopis cineraria.*

1 INTRODUCTION

Aluminum is a lightweight metal (density = 2.71 g/cm^3) having good corrosion resistance to the atmosphere and to many aqueous media, combined with good electrical and thermal conductivity. The recyclability and available new smelting processes, low cost and high strength-to-weight ratio are attractive properties. The metal and its alloy are nonmagnetic and have high electrical and thermal conductivity and high reflectivity. Aluminium and its alloy 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 which causes severe loss and malfunctioning of industrial equipment. So it is very important to combat corrosion of metals. The inhibitors can be organic or inorganic and the use of chemical inhibitors has been limited because of their environmental threat, because of environmental regulations, Plant extracts are environmentally acceptable and readily available, in addition to being a renewable source for a wide range of corrosion inhibitors. The recently researchers trend is towards environmentally friendly and organic inhibitors. In present study we have used the Ethanolic extracts of *Prosopis cineraria* leaves.

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Prosopis cineraria is 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 campestrol, cholesterol, sitosterol, stigmasterol, Tricosan-1-ol, Methyldocosanoate,Diisopropyl-10,11-leaves and pods are Aspartic acid, Glutamic acid, Serine, Glycine, Histidine, Threonine, Arginine, Alanine, Proline, Tyrosine, Valine, Methionine, Cysteine, Isoleucine, Leucine, Phenylalanine and Lysine.[3-6]

2. EXPERIMENTAL
2.1 Extraction of leaves extract- Leaves of *Prosopis cineraria* in natural condition were air dried for 8 to 10 days in shade. Then grinded and powdered, finely powdered dried material was taken in 500 ml round bottom flask and sufficient quantity of ethyl alcohol was added and left for desired period. Then the resulting solution is extracted by standard method as discussed literatures [7,8]

2.2 Specimen preparation- The rectangular coupons of copper of dimensions (3cm× 2.4cm× 0.16cm) were used with a small hole of 2mm diameter near upper edge for hanging in test media. Each coupon was given surface treatment by standard method discussed in literature for each experiment.

2.3 Test solution-The 0.5 N HCl solutions was prepared by using doubly distilled water. HCl used was of analytical reagent quality.

2.4 (A) Chemical method-In chemical method the surface treated copper coupons were dipped in beakers containing corrosive solutions and solutions containing inhibitors various inhibitor concentrations ranging from 0.0744 to 1.0426 g/l. These coupons were made to immerse for desired immersion period at room temperature (303±1°K). Before measurement, each coupon was surface treated dried in desiccators for 24 hours and then weight was taken. From the weight loss method, the Inhibition efficiency and corrosion rate of copper were calculated using standard equation

3. EFFECT OF TEMPERATURE
3.1 *Effect of temperature on weight loss, corrosion rates, and inhibition efficiency IE (%)*
The effect of temperature on the various corrosion parameters weight loss, corrosion rates, fractional surface coverage (θ), adsorption equilibrium constant (K_{ad}) and inhibition efficiency IE (%) is studied in 0.5M HCl between temperature range 303-353K in the absence and presence of EE*Pc*L at 24 hrs immersion period. The results obtained by weight loss measurements were listed in Table-1

The stability and mechanism of adsorption of the EE*Pc*L on the aluminium surface is studied by evaluating the variation of corrosion rate and inhibition efficiency with temperature (303K-333K) at 24 hrs (Fig-1 to 3).

The aim was to evaluate the thermodynamic stability of the absorbed film of inhibitor on aluminium. Taking a critical looks at Table-1 and Fig-(1) the result indicates that as temperature increases, the weight loss and rate of corrosion increase while the inhibitory efficiency reduces. The weight loss of 0.018g at 303K and 0.3423g at 353K was obtained in blank solution. The weight loss was reduced to 0.0024g at 303K and 0.1104g at 353K in presence of highest inhibitor concentration (1.064g/l). The minimum corrosion rate 0.2012mmy-1 is observed at 303K at 24 hrs. Figure-2 also shows that corrosion rate increases at each concentration as the temperature increases from 303K to 353K. This is due to increased rate of dissolution process of aluminium and partial desorption of the inhibitor from the metal surface with increasing temperature and also at higher temperature, reacting molecules collide faster leading to more consumption of the reactant and formation of the product.[9]
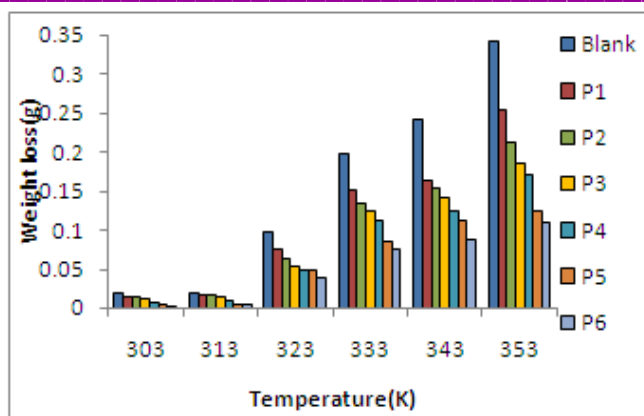
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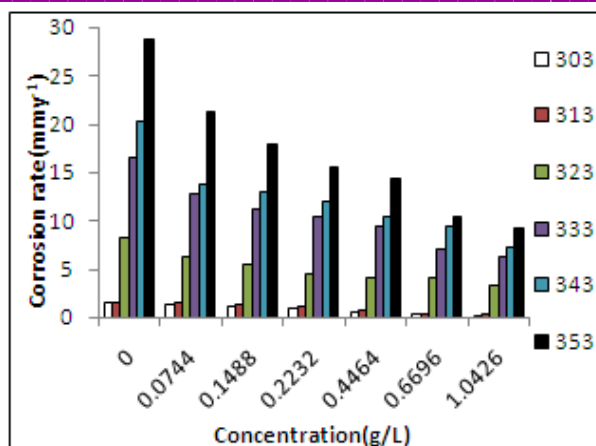Fig-1 Weight loss(g) vs temperature at different EE*Pc*L

Fig-2 Corrosion rates for aluminium in 0.5M HCl at different temperatures

Fig-(3) shows and Table-1 that inhibition efficiency does not show any regular trend as the temperature increases at different concentration of EE*Pc*L. Inhibition efficiency increases on increasing concentration of EE*Pc*L at every temperature. At highest inhibitor concentration IE% first decreases on rising temperature up to 323K and then slightly increases. This implies that at relatively higher temperatures the extract could still function as good corrosion inhibitor The presence of inhibitor leads to decrease of the corrosion rate. Also, we note that the inhibition efficiency depends on the temperature and decreases with the rise of temperature from 303 to 343 K and then increases this is due the fact that at higher temperature chemical adsorption starts. The decrease in inhibition efficiency indicates that the inhibitory effect of extract is limited at elevated temperature.
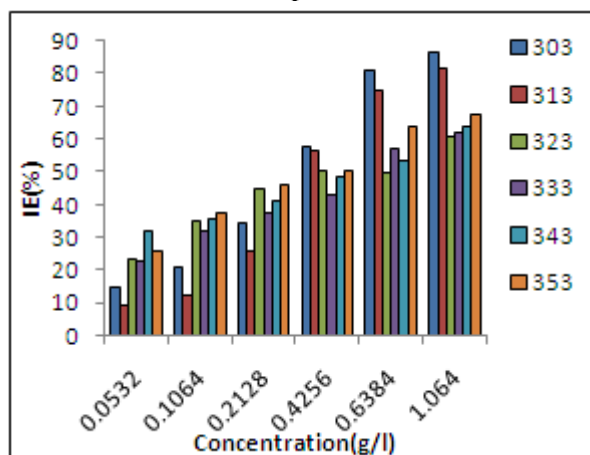

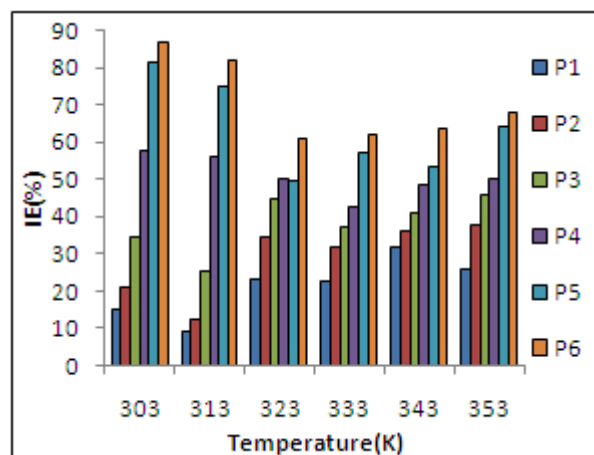Fig-3 Variation of IE (%) with EE*Pc*L at different temperatures

Fig-4: Variation of IE (%) with different temperatures in EE*Pc*L

This practically shows that the rise in temperature decreases the inhibitive process; hence the highest inhibition efficiency is at 303K.

The variation in inhibition efficiency (Fig-4) did not follow a consistent trend like in the case of the corrosion rates but for each temperature IE% increases as EE*Pc*L concentration increases. Fig-(4) also shows that for highest inhibitor concentration IE% decreases up to 333K after that it increases slightly showing that there might be chemical adsorption at higher temperature. Table-1 revealed that I.E% and θ decreases up to 323 K when compared to values obtained at 303 K. The decrease in I.E% 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

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stability of components – aluminium surface complex. However, inhibition efficiency of the EE*Pc*L   ranged between 15% - 65% for different temperatures at higher temperature

Table-1 Corrosion parameters of acid corrosion of aluminium without and with different concentrations of EE*Pc*L at various temperatures) for 24 hrs immersion period

Temp (K)	Prosopis cineraria *Leaves extract Concen-tration (g/L)*	Corrosion parameters				
		Weight loss (g)	Corrosion rate (ρ_{corr}) (mmy^{-1})	Inhibition (efficiency IE%)	Fractional surface coverage (θ)	Adsorption equilibrium constant (K_{ad})(L/g)
303	P0 ((Blank)	0.018	1.5088	-	-	-
	P1 (0.0532)	0.0153	1.2824	15.00	0.1500	3.3171
	P2 (0.1064)	0.0142	1.1902	21.11	0.2111	2.5151
	P3 (0.2128)	0.0118	0.9891	34.44	0.3444	2.4691
	P4 (0.4256)	0.0076	0.6370	57.78	0.5778	3.2153
	P5 (0.6384)	0.0034	0.2850	81.11	0.8111	6.7264
	P6 (1.064)	0.0024	0.2012	86.67	0.8667	6.1090
313	P0 ((Blank)	0.0192	1.6093	-	-	-
	P1 (0.0532)	0.0174	1.4585	9.38	0.0938	1.9445
	P2 (0.1064)	0.0168	1.4082	12.50	0.1250	1.3426
	P3 (0.2128)	0.0143	1.1986	25.52	0.2552	1.6102
	P4 (0.4256)	0.0084	0.7041	56.25	0.5625	3.0209
	P5 (0.6384)	0.0048	0.4023	75.00	0.7500	4.6992
	P6 (1.064)	0.0035	0.2934	81.77	0.8177	4.2159
323	P0 ((Blank)	0.0984	8.2479	-	-	-
	P1 (0.0532)	0.0756	6.3368	23.17	0.2317	5.6689
	P2 (0.1064)	0.0642	5.3813	34.76	0.3476	5.0067
	P3 (0.2128)	0.0542	4.5431	44.92	0.4492	3.8322
	P4 (0.4256)	0.0489	4.0988	50.30	0.5030	2.3785
	P5 (0.6384)	0.0495	4.1491	49.70	0.4970	1.5474
	P6 (1.064)	0.0384	3.2187	60.98	0.6098	1.4685
333	P0 ((Blank)	0.1964	16.4623	-	-	-
	P1 (0.0532)	0.1523	12.7658	22.45	0.2245	5.4429
	P2 (0.1064)	0.1342	11.2487	31.67	0.3167	4.3561
	P3 (0.2128)	0.1234	10.3434	37.17	0.3717	2.7799
	P4 (0.4256)	0.1123	9.4130	42.82	0.4282	1.7596
	P5 (0.6384)	0.0845	7.0828	56.98	0.5698	2.0743
	P6 (1.064)	0.0742	6.2195	62.22	0.6222	1.5478
343	P0 ((Blank)	0.2413	20.2258	-	-	-
	P1 (0.0532)	0.1643	13.7717	31.91	0.3191	8.8093
	P2 (0.1064)	0.1547	12.9670	35.89	0.3589	5.2612
	P3 (0.2128)	0.1423	11.9276	41.03	0.4103	3.2693
	P4 (0.4256)	0.1245	10.4356	48.40	0.4840	2.2043
	P5 (0.6384)	0.1123	9.4130	53.46	0.5346	1.7994
	P6 (1.064)	0.0875	7.3343	63.74	0.6374	1.6520
353	P0 ((Blank)	0.3423	28.6916	-	-	-
	P1 (0.0532)	0.2543	21.3155	25.71	0.2571	6.5047
	P2 (0.1064)	0.2134	17.8872	37.66	0.3766	5.6770
	P3 (0.2128)	0.1846	15.4732	46.07	0.4607	4.0145

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P4 (0.4256)	0.1708	14.3165	50.10	0.5010	2.3593
P5 (0.6384)	0.1236	10.3602	63.89	0.6389	2.7716
P6 (1.064)	0.1104	9.2537	67.75	0.6775	1.9742

3.2 Kinetic parameters of weight loss treatment:

Corrosion rate of Aluminium against the concentration of the inhibitor obeys the following kinetic relationship [10].

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

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. Fig-(5) represents the curves of $\log \rho_{corr}$ versus $\log C_{inh}$ at different hours of immersion. The kinetic parameters k and B calculated by above equation are given 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 from Table-2 that the value of k increases with temperature.

Table: 2 Kinetic parameters for corrosion of aluminiuim 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
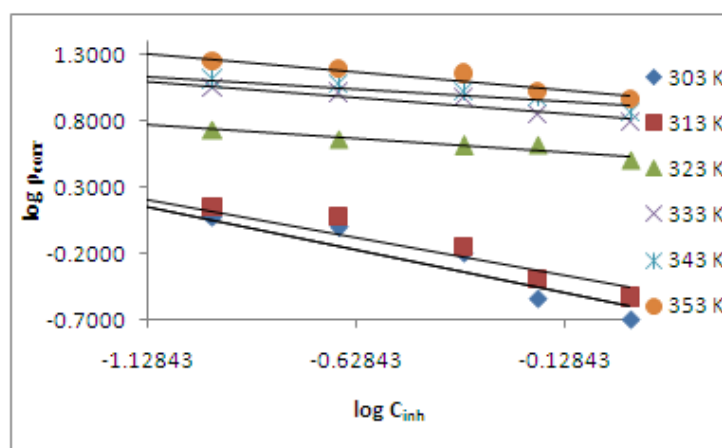
Fig-5 Variation of $\log \rho_{corr}$ with $\log C_{inh}$ for aluminium corrosion in 0.5M HCl at various temperatures.

3.3 Effect of temperature and activation studies

In this study, the effect of temperature on the corrosion and inhibition process of aluminium in 0.5M HCl in the absence and presence of different concentrations of EE*Pc*L after 24 h of immersion was followed at 303–353K using weight loss measurements. Temperature has great effect on the corrosion phenomenon. Generally, the corrosion rate increases with rise in temperature. The result of the effect of temperature on corrosion rate is presented in Table-1 and Table-3, showing that corrosion rate increases as temperature rises. The dependence of corrosion rate on temperature can be expressed by the Arrhenius equation [10-12].

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

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 $\log \rho_{corr}$ *versus* 1/T gave straight lines as shown in Fig-(6). E_{act} was calculated from slopes of $\log \rho_{corr}$ *versus* 1/T. Table-3 shows that in the presence of EE*Pc*L the value of E_{act} is increases as compared to the blank Solution(58.84 kJmol^{-1}) indicating the adsorption of EE*Pc*L on the surface of the metal. It was observed that the activation energy E_{act} for blank acid were lower than in inhibited acid. Activation energy of EE*Pc*L ranged between 58.84 to 75.77 kJ/mol. The higher values

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in the presence of EE*Pc*L inhibitor indicate physical adsorption of the inhibitor on the metal surface, while the opposite is the case with chemisorption. Similar trend of E_{act} results were reported by other researchers [13]. The value of E_{act} increases with the increase in EE*Pc*L concentration until it reaches maximum value. The results showed 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 [12-14]

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

A plot of log(ρ_{corr}/T) verses 1/T give a straight lines (Fig-7) from which the values of ΔS_{act} and ΔH_{act} were calculated. All thermodynamic parameters of the corrosion reaction are given in 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 [12-19]. The average value of the difference ($E_{act} - \Delta H_{act}$) 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 following equation.
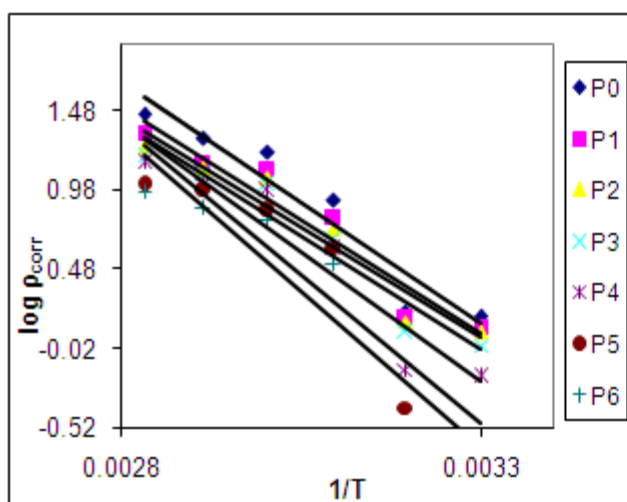
Ea - ΔH_{act} = RT

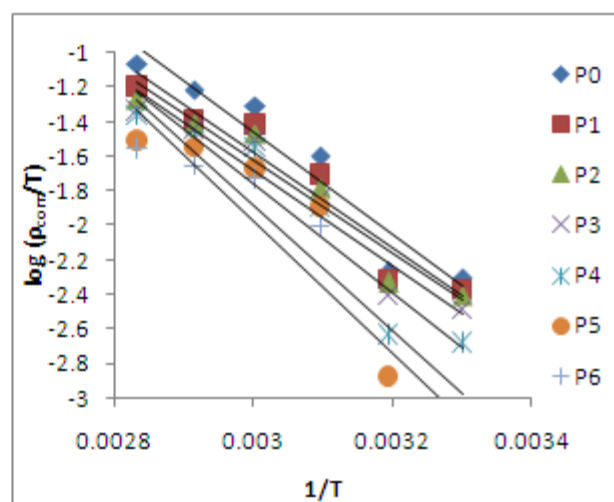**Fig-6 logρ_{corr} versus 1/T**   **Fig-7 log (ρ_{corr}/T) verses 1/T**

Table-3 Kinetic-thermodynamic corrosion parameters for aluminium corrosion in absence and presence of various concentrations of EE*Pc*L in 0.5 HCl

C_{inh}(g/l)	ΔH_{act} (kJmol^{-1})	ΔS_{act} (JK^{-1}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 (1.064)	73.74	-14.03	75.77

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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 following equation

$$\Delta G = -2.303\ RT \log(K_{ad} \times 55.5)$$

here 55.5 is the concentration of water in ml/l. Other adsorption parameters ΔH_{ads} and ΔS_{ads} are obtained from the following equation [18,19]

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

The variation of ΔG_{ads} *versus* T gives a straight line. The slope gives ΔS_{ads} and the intercept leads to ΔH_{ads} .The obtained values are given in Table-4.

The adsorption parameters are listed in 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. The literature pointed out that an exothermic process means either physi- 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 solution, an exothermic adsorption was detected at higher inhibitor concentration. The positive values of ΔH_{ads} show that the adsorption is endothermic with an disordered phenomenon described by the positive values of ΔS_{ads} this disorderness decreases at higher concentration. The decrease in entropy suggested that in the rate there is 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.[14]

Table-4 Thermodynamic parameters for the adsorption of EE*Pc*L on aluminium in 0.5M HCl at different Temperatures

Temp/ concen- tration	ΔG_{ads}(kJ/mol)						ΔH_{ads} (kJ mol^{-1})	ΔS_{ads} (JK^{-1} mol^{-1})
	303K	313K	323K	333K	343K	353K		
P1 (0.0532)	-13.1409	-11.7952	-15.4477	-15.8132	-17.1466	-16.3067	15.28	92
P2(0.1064)	-12.4435	-10.862	-15.114	-15.1965	-15.7193	-15.9299	15.85	91
P3 (0.2128)	-12.397	-11.3199	-14.396	-13.9528	-14.4018	-14.9703	6.734	61
P4 (0.4256)	-13.0623	-12.9053	-13.1148	-12.6864	-13.3103	-13.4984	-10.31	8
P5 (0.6384)	-14.9221	-14.0185	-11.9603	-13.142	-12.7482	-13.9445	-20.5	21
P6 (1.064)	-14.6795	-13.745	-11.8197	-12.3313	-12.5116	-13.005	-23.85	33

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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 EE*Pc*L 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[21-24] was tested for examine fitness to the experimental data. Langmuir isotherm is given by equation

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ad}} + C_{inh} \qquad\qquad .....(4)$$

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} as shown in Fig-(8) gave straight lines which clearly show that aluminium corrosion inhibition in HCl by EE*Pc*L at room temperature obeys Langmuir adsorption isotherm. The correlation coefficient, slopes, and adsorption coefficients obtained from Langmuir isotherm plots are shown in Table-5. The free energy of adsorption, ΔG_{ads}, is associated with water adsorption equilibrium. ΔG_{ads} is calculated from the relation in equation (5)

$$K = \frac{1}{(55.5)e^{\left(\frac{-\Delta G_{ads}}{RT}\right)}} \qquad\qquad .....(5)$$

The negative value of ΔG_{ads}, here indicate that the adsorption process on aluminium surface is spontaneous[20-24]. The correlation coefficient (R^2) and slope obtained was nearly equal to unity best fitted in experimental data. The ΔG_{ads} value indicates that the adsorption is physical. If the values are in the order of -20kJ/mol or less than this indicate a physical adsorption, while values of -40kJ/mol or higher imply chemical adsorption which involve charge sharing or a transfer from the inhibitor molecules to the metal surface to form a physical bond.
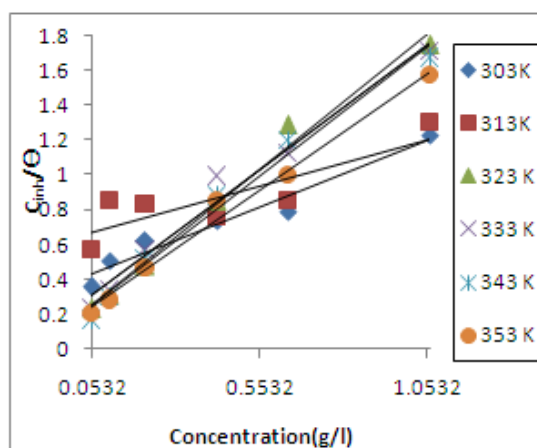

Temp(K)	Slope	R^2	K_{ad}	ΔG_{ads} (KJmol^{-1})
303	0.764	0.955	2.59	-12.5181
313	0.537	0.732	1.57	-11.6315
323	1.553	0.987	6.02	-15.6109
333	1.431	0.977	4.33	-15.1792
343	1.482	0.983	5.92	-16.5264
353	1.339	0.988	5.88	-16.9909

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

Fig: 8 Langmuir adsorption isotherm of EE*Pc*L for aluminium

Conclusions- The following conclusions may be drawn from our studies:

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1. Ethanolic Extract of *Prosopis cineraria* Leaves (EE*Pc*L) is as an inhibitor in the corrosion of aluminium in 0.5MHCl solution. The maximum inhibition efficiency of 86.67% was obtained at 303K for 1.064g/l .

2. The weight loss and corrosion rate was found to be greater at elevated temperature increased with increase in temperature.

3. Inhibition efficiency increased with increase in the concentration of the extract but decreases with increase in temperature.

4. 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.

5. The activation energy E_{act} for blank acid were lower than in inhibited acid. Activation energy of EE*Pc*L ranged between 58.84 to 75.77 KJ/mol. The higher values in the presence of EE*Pc*L inhibitor indicate physical adsorption of the inhibitor on the metal surface.

6. The adsorption of the extract on the aluminium surfaces obeyed Langmuir adsorption isotherms at elevated temperatures. The adsorption process is spontaneous.

7. Heat of adsorption was negative at higher inhibitor concentration which shows that adsorption is exothermic and hence inhibition efficiency decreases with rise in temperature. Entropy of adsorption decreases with increase in EE*Pc*L indicates association of inhibitor molecules.

Overall, the EE*Pc*L was found to be efficient for corrosion inhibition of aluminium in 0.5M HCl at lower temperatures.

ACKNOWLEDGMENTS

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