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ADSORPTION ISOTHERM OF LAGENARIA SICERARIA LEAVES AS CORROSION INHIBITOR OF CARBON STEEL IN 1.0N HCL ACID

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Abstract: Adsorption behavior of Carbon steel in 1.0N HCl solution in the presence of Lagenaria Siceraria Leaves (LSL) extract was studied by mass loss measurements with various periods of contact and temperature. Inhibition efficiency increased with increase of inhibitor concentration. The inhibitive effect of the Lagenaria Siceraria leaves could be attributed to the presence of some active compound which is adsorbed on the surface of the Carbon steel. The LSL was found to conform by various adsorption isotherms viz., Langmuir adsorption, Temkin adsorption, Florry-Huggins adsorption, Frumkin adsorption, Freundlich adsorption and El-Awady adsorption isotherm at different concentration and temperature investigated and inhibitor obeyed Langmuir adsorption isotherm. Thermodynamic parameters also revealed that the adsorption process is spontaneous.

Key words : Carbon Steel, Corrosion inhibition, Lagenaria Siceraria Leaves, Adsorption isotherms.

1. INTRODUCTION

Corrosion of materials is a natural phenomenon that is a cause of concern as it has incurred a total damage of billions of dollars to many industries in world wide. In order to overcome this corrosion problem various preventing measures such as inhibitors, anodic protections, Cathodic protections coatings and alloying were developed. Among all of these methods, corrosion inhibitors are most popular due to the ease in application and the advantage of in situ without disruption of the process. Corrosion inhibitors are substances which when added in small concentrations to the corrosive environment will reduce the rate of corrosion[1]. The heterocyclic organic compounds and their derivatives have been successful as corrosion inhibitors, although their toxicity is an important disadvantage, for it limits their application due to environmental impact reason [2,3]. The use of corrosion inhibitors as a means of protection is necessary in many industrial cases: surface preparation, transport and storage of metals, cooling circuits, rehabilitation of reinforced concrete, painting and Cathodic protection [4-

6]. The adsorption characteristics Carbon steel of corrosion inhibitors depend upon the chemical moiety of the molecule, type of functional groups and the electron density at the donor atoms. Organic compounds, containing hetero atom's (N, O, S and P), electronegative functional groups, π-electrons and aromatic rings as electron density rich centres which are considered as good adsorptive centre [7-9]. These heterocyclic organic inhibitors get adsorb onto the steel surface or form protective insoluble layer and block corrosion sites, which reduces contact of corroding material with the corrosive medium/steel [10]. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as "Green Inhibitors". These inhibitors do not contain heavy metals or other toxic compounds. Recent studies using plants containing heteroatom such as oxygen, nitrogen and sulphur like Cnidoscolus chayamans, Solanum Torvum, Pisonia Grandis, mimusops elengi, Sauropus Androgynus, Kingiodendron pinnatum, Wrightia Tinctoria, Aloe- Vera gel, Hibiscus Rosa Sinensis and Azadirachita Indica leaves, Aihagi Maurerum and Merusnigra and Apricot Leaves, Adansonia digitata (Baobab) fruit pulp and seeds, Cupressus sempervirens, Feungreak leaves, essential oils of Alpinia Galanga, Chrysophyllum Albidum leaves, poupartiabirrea back, Acacia Tortilis, Arabinogalactan, keto sulphone drug, Artemisia Mesatlantica essential oil, spirogyraalgae, Tragacanth gum, Prunus Persic, Lemon Gross, Secang heartwood extract (Caesalpinia sappan I), Dried marjoram leaves, Lagenaria Siceraria Peel [11-36] have also been used for inhibition of corrosion. In the present study, Lagenaria Siceraria Leaves plant extracts was investigated for its corrosion inhibition potential by using weight loss measurement and adsorption studies.

2.0. MATERIALS AND METHODS

2.1. Lagenaria Siceraria Leaves used as a corrosion inhibitor

2.2. Stock solution of Lagenaria Siceraria Leaves Extract:

Lagenaria Siceraria Leaves *(LSL)* was collected from the source and dried under shadow for about 10 days, grained well, then soaked in a solution of ethyl alcohol for about 48 hrs, Then it is filtered followed by evaporation in order to remove the alcohol solvent completely and the pure plant extract was collected. From this extract, different concentration of 10 to 1000ppm stock solution was prepared using double distilled water and used throughout our present investigation.

2.3 Specimen preparation

Rectangular specimen of Carbon steel was mechanically pressed cut to form different coupons, each of dimension exactly 20cm^2 (5x4.9x1.9cm)with emery wheel of 80 and 120 and degreased with trichloroethylene, washed with distilled water, cleaned and dried, then stored in desicators for our present study.

3.0 RESULTS AND DISCUSSION

3.1 Effect of time variation

The dissolution behavior of Carbon steel in 1.0N HCl containing the presence and absence of LSL extract with various exposure times (24hrs to 360 hrs) are shown in Table-1**.** The observed values are clearly indicates that the presence of LSL extract, the corrosion rate moderately decreased from 1.4878 to 1.1624 mmpy for 24 hrs and 0.1208 to 0.0185 mmpy after 360 hrs with increase of inhibitor concentration (0 to 1000 ppm). The maximum of 84.62 % of inhibition efficiency is achieved after 360 hrs exposure time, suggests that the adsorption process occurs mainly due to the presence of active phytochemical constituents present in the inhibitor molecule especially oxygen containing species and the metal ion from the surface of the metal.

Table- 1 The corrosion parameters of Carbon steel in 1.0N Hydrochloric acid containing various concentration of LSL inhibitor at different exposure time.

Conc. of inhibitors (ppm)	24 hrs		72 hrs		120 hrs		240 hrs		360 hrs	
	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E
0	1.4878	-	0.5269	-	0.3254	-	0.1720	-	0.1208	-
10	1.3949	6.24	0.5037	4.40	0.3022	7.12	0.1464	14.88	0.1038	14.07
50	1.3484	9.36	0.4184	20.59	0.2929	9.98	0.1278	25.69	0.0805	33.36
100	1.3019	12.49	0.4029	23.53	0.2789	14.29	0.1162	32.44	0.0666	44.86
500	1.2089	18.74	0.3719	29.41	0.2371	27.13	0.0999	41.91	0.0387	67.96
1000	1.1624	21.87	0.3564	32.35	0.1859	42.87	0.0697	59.47	0.0185	84.62

3.2. Effect of Temperature

Dissolution behavior of Carbon Steel in 1.0N HCl containing various concentration of LSL extract at 303to 333K and the observed values are listed in Table-2. The observed results reveals that the corrosion rate decreased with increase of inhibitor concentrations and increased with rise in Temperature from 303 to 333K. The maximum of 73.68% inhibition efficiency is achieved at 333K. However the value of inhibition efficiency is increased with rise in Temperature may suggests and support the facts that the process of adsorption follows **chemisorption**.

Table-2. The corrosion parameters of Carbon steel in 1.0N Hydrochloric acid containing various concentration of LSL inhibitor at different temperature after one hours exposure time.

Conc. of inhibitor (ppm)	303 K		313 K		333 K	
	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E
0	32.3617	-	37.3834	-	42.4050	-
10	29.5719	8.62	34.5936	7.46	34.5936	18.42
50	27.8980	13.79	30.6878	17.91	23.9923	43.42
100	25.6662	20.68	27.8980	25.37	20.0866	52.63
500	23.4343	27.58	23.4343	37.31	13.9490	67.10
1000	21.2025	34.48	17.8547	52.23	11.1592	73.68

3.3 Activation Parameters On The Inhibition Process:

Usually, the temperature plays an important role to understanding the inhibitive mechanism of the corrosion process. To assess the temperature effect, experiments were performed at 303K- 333K in uninhibited and inhibited solutions containing different concentrations of LSL and the corrosion rate was evaluated and the values are presented in Table-3. The relationship b/w the corrosion rate (CR) of Carbon steel in acidic media and temperature (T) is expressed by the Arrhenius equation,

$$\text{Log CR} = -Ea/2.303RT + \log \lambda \text{ --------- } \rightarrow (1)$$

Where Ea is the apparent effective activation energy, R molar gas constant and λ is the Arrhenius pre- exponential factor.

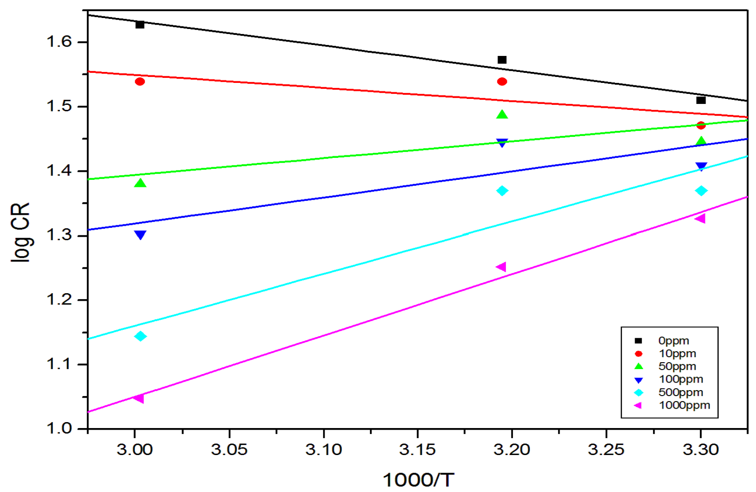

Fig-1. Arrhenius plot for Carbon steel corrosion 1.0N HCl in the absence and presence of different concentration of LSL.

A plot of log (CR) obtained by weight loss measurement versus 1/T gave straight line with regression co-efficient (R^2) close to unity as shown fig (1). The values of apparent activation energy (Ea) obtained from the slope (-Ea/2.303R) of the lines and the pre-exponential factor (λ) obtained from the intercept (logλ) are given in Table -3. It is evident from the Table that the apparent energy of activation decreased on addition of (LSL) in comparison to the uninhibited solution. These values ranged from 7.3065 to -18.2433 kJ/mol and are lower than the threshold value of 80kJ/mol required for chemical adsorption. This shows that the adsorption of ethanol extract of LSL on Carbon Steel surface is Physical adsorption. Decrease in the activation energy is attributed to appreciable increase in the adsorption of inhibitor on Carbon Steel surface by increase in the temperature. The increase in adsorption leads to decrease in corrosion rate due to the lesser exposed surface area of the Carbon steel towards 1.0N HCl.

Table:3 Activation parameters of LSL in 1.0N HCl.

Inhibitor conc. (ppm)	E_a kJ/mol	Λ	ΔH (kJ/mol)	ΔS (J/mol/k)
Blank	7.3065	600.06	4.6584	79.1663
10	3.8524	142.33	1.2062	73.9733
50	5.0012	4.07	-7.6473	61.1448
100	7.7488	1.27	-10.4007	56.9305

500	-15.4785	0.05	-18.1304	45.5445
1000	-18.2433	0.01	-20.8952	41.0283

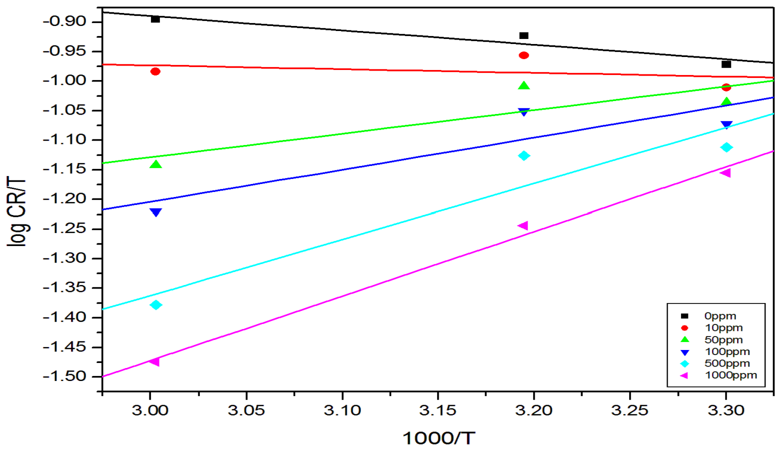

Figure-2 Transition state plot for Carbon steel corrosion in 1.0N HCl in the absence & presence of different concentration of LSL.

The value of λ is also lower for inhibited solution than for the uninhibited soln. It is clear from equation (1) that corrosion rate is influenced by both Ea & λ. Moreover increase in concentration of (LSL) in leads to an decrease in the value of Ea, indicating that the weak adsorption of the inhibitor molecules on the metal surface.

Experimental corrosion rate values evaluated from the weight loss data for Carbon steel in 1.0N HCl in the presence and absence of LSL was used to determine the enthalpy of activation (ΔH) and apparent entropy of activation (ΔS) for the formation of the activation complex in the transition state equation (2). An alternative formula for the Arrhenius equation is the transition state equation

$$CR = RT/Nh \exp(\Delta S/R) \exp(-\Delta H/RT) \text{ --------------(2)}$$

A plot of log (CR/T) versus 1/T is shown is fig (2), a straight lines were obtained with slope (-ΔH/2.303R) and intercept of [log (R/Nh)+(ΔS/2.303R)], from which ΔH and ΔS were calculated and listed out in Table -3. The negative value of enthalpy of activation (ΔH) in the presence and absence of various concentration of inhibitor reflects that the exothermic natures of Carbon steel. The values of entropy of activation (ΔS) listed in Table-3. It is clear that the entropy of activation decreased in the presence of the using inhibitor compared to free acid solution. The decrease in the entropy of activation (ΔS) in the presence of inhibitor may decreases in the disordering on going from reactant to activated complex is difficult.

3.4 ADSORPTION STUDIES:

Process of adsorption are very important phenomenon to determine the corrosion rate of reaction mechanism. The most frequently use of isotherms are viz: Langmuir, Temkin, Frumkin, Flory- Huggins, Freund lich, Bockris-Swinkles, Hill-de Boer, Parson's and the El-Amady, thermodynamic-kinetic model.

3.4.1. Langmuir Isotherm:

Langmuir adsorption isotherm is expressed according to equation (3)

$$\text{Log } C/\theta = \log C - \log K \text{ --------- } \rightarrow (3)$$

Plotting log (C/θ) against log C gave a linear relationship as shown in fig.3, and the adsorption parameters are presented in Table- 4. The average regression value (R^2= 0.9938) move close to unity suggest that the adsorption of extract of LSL on surface of Carbon steel indicated that there is no interaction b/w the adsorbate & adsorbent.

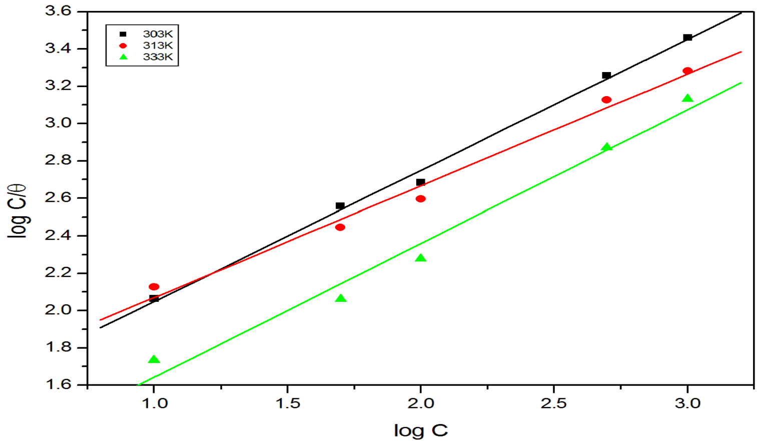

Figure -3. Langmuir isotherm for adsorption of ethanol extract of Lagenaria Siceraria Leaves on Carbon steel surface.

3.4.2. Temkin Isotherm:

Temkin adsorption isotherm, the degree of surface coverage(θ) is related to the inhibitor concentration (c) according to equation(4),

$$\text{Exp}(-2a\,\theta) = KC \text{-------------- (4)}$$

K-adsorption of equilibrium constant and a is the attractive parameter, Rearranging & taking logarithm of both sides of equation (E) gives equation(5)

$$\Theta = (-2.303\log k/2a) - (2.303\log C/2a) \text{------------(5)}$$

Plots of θ against log c are presented in fig-4 gave linear relationship, which shows that the adsorption data fitted Temkin Adsorption Isotherm. Adsorption parameters obtained from Temkin adsorption isotherm are recorded in Table-4. The average regression co-efficient value (R^2) is 0.9850 close to unity. The values of attractive parameter (a) are positive in all cases, indicating that the no repulsion exists in the adsorption layer.

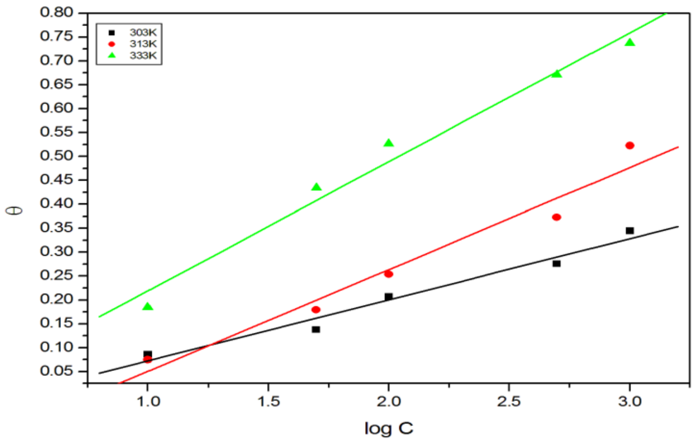

Figure-4. Temkin isotherm for adsorption of ethanol extract of Lagenaria Siceraria Leaves on Carbon steel surface.

3.4.3 Florry-Huggins Isotherm:

Florry- Huggins adsorption isotherm can be expressed according to equation (6)

$$\text{Log}\ (\theta/C) = \log K + x\log(1-\theta) \text{ ---------------- } \rightarrow (6)$$

The plots of log θ/c against log (1- θ) are shown in fig 5, and this data conformed to Florry huggins isotherm with average regression co-efficient (R^2) value 0.8954. It is less than unity. The values of the size parameter x are positive as shown in Table -4. This indicates that the adsorbed species of ethanol extracts of LSL is bulky. Since it could displace more than one water molecule from the Carbon steel surface.

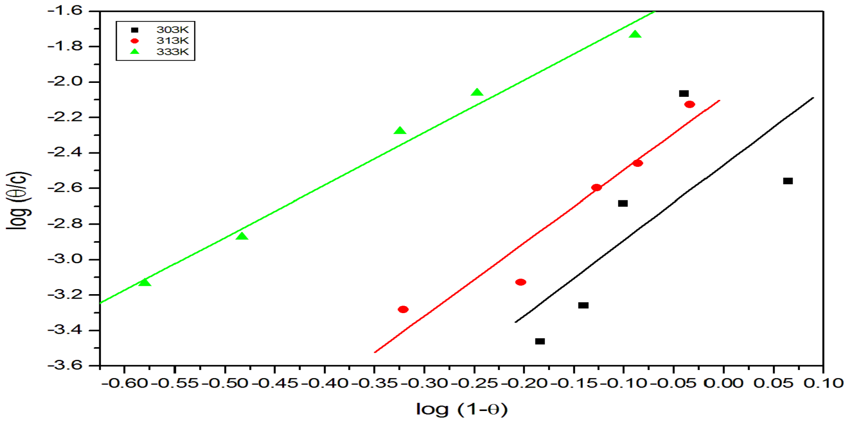

Figure-5. Florry-Huggins isotherm for adsorption of ethanol extract of Lagenaria Siceraria Leaves on Carbon steel surface.

3.4.4. Frumkin Isotherm:

Frumkin adsorption isotherm is given by equation (7)

$$\log \{ [C]^* (\theta/1-\theta)\} = 2.303 \log K + 2\alpha\theta \text{ ----------} \rightarrow (7)$$

where k is the adsorption –desorption constant and α is the lateral interaction term describing the interaction in adsorbed layer plots of log { [C]* (θ/1- θ)} versus θ as presented were linear which shows the applicability of Frumkin isotherm. The values for Frumkin adsorption parameters were recorded in Table 4. The average regression co-efficient value (R^2=0.9856) is almost close to unity and obeys Frumkin adsorption isotherm. Also shows that values of the adsorption parameters α are positive suggest that the attractive behaviour of the inhibitor on the surface of Carbon Steel.

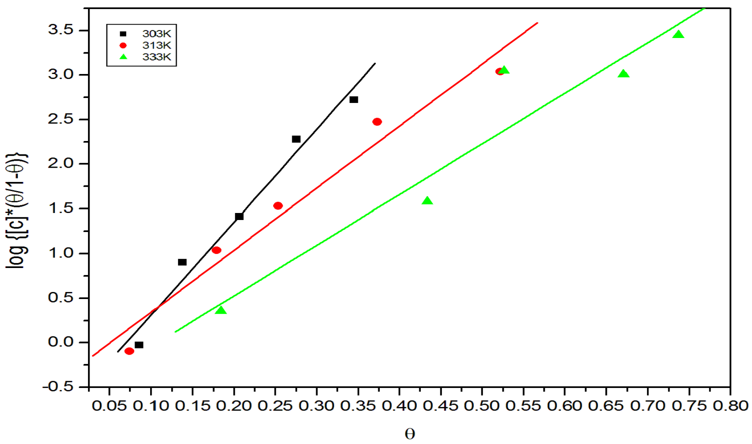

Figure-6. Frumkin isotherm for adsorption of ethanol extract of Lagenaria Siceraria Leaves on Carbon steel surface.

3.4.5 Freundlich Isotherm:

The Freundlich adsorption isotherm can be also be applied

$$\Theta = Kc^{n} \text{ ------------} \rightarrow (8)$$

Freundlich model equation(8) can be rearranged as

$$\text{Log } \theta = \log K + n\log C \text{ --------} \rightarrow (9)$$

This can be plotted as log θ vs log C from the intercept of the values of K can be obtained. Note that the values of the slopes and intercepts were taken from the straight line eqns. The higher values of K indicate that the inhibitor strongly adsorbed on the metal surface.

The magnitude of the exponent n gives an indication on the favourability of adsorption. It is generally stated that values of n in the range 2-10 represent good, 1-2 moderately difficult and less than 1 poor adsorption characteristics. Thus LSL inhibitor adsorbed on the metal surface by physical process.

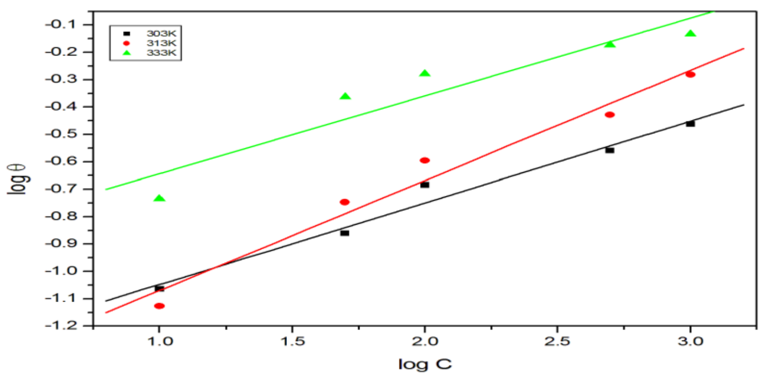

Figure -7. Freundlich isotherm for adsorption of ethanol extract of Lagenaria Siceraria Leaves on Carbon steel surface.

3.4.6. El Awady Isotherm:

The El-Awady adsorption isotherm is given by

$$\text{Log } (\theta/1\text{-}\theta) = \log K + y\log C \text{------------- } (10)$$

Where C is molar concentration of inhibitor in the bulk solution, θ is the degree of surface coverage, K is the equilibrium const of adsorption process, $k_{ads} = k^{1/y}$ and y represents occupying a given active site. Value of 1/y less than unity implies the formation of multilayer of the inhibitor on the metal surface, while the value of 1/y greater than unity means that a given inhibitor occupy more than one active site [26,27,28]. Curve fitting of the data to the thermodynamic/kinetic model [El-Awady et al.,] is shown in fig(8). The plot gives straight lines which show that the experimental data fits the isotherm. The values of k_{ads} and 1/y calculated from the El-Awady et al isotherm model is listed in table (4).

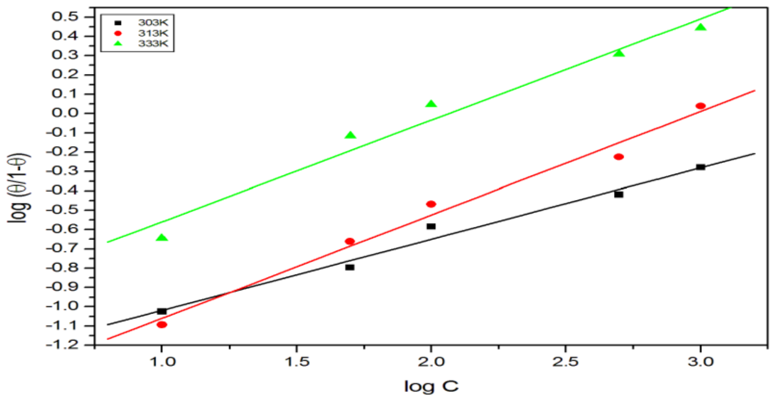

Figure-8. El-Awady isotherm for adsorption of ethanol extract of Lagenaria Siceraria Leaves on Carbon steel surface.

3.5. FREE ENERGY OF ADSORPTION

The equilibrium const of adsorption of ethanol extract of (LSL) on the surface of Carbon steel is related to the free energy of adsorption (ΔG) according to equation (11)

$$\Delta G= -2.303RT \log (55.5K) \text{ -------------- } (11)$$

Where R is gas constant and T is the temperature. The free energy of adsorption was calculated from values of k obtained from Langmuir, Temkin, Florry –Huggins, Frumkin, Freundlich and El-Awady according to equation (11) and is recorded in Table-4. The results show that free energy of adsorption ΔG are negative & less than the threshold value of -40kJ/mol required for chemical adsorption, indicating that adsorption of ethanol extract of LSL on Carbon Steel surface is spontaneous and occurred according to the mechanism of physical adsorption. Since phenomenon is attributed to electrostatic interactions between the charged metal and charged molecules.

Table: 7.3.Adsorption parameters for adsorption of ethanol extract of LSL on Carbon steel surface.

Isotherm	Temperature	R^2	K	ΔG_{ads} kJ/mol	
Langmuir	303K	0.9978	22.1717	-17.9274	
	313K	0.9933	29.6278	-10.4361	
	333K	0.9905	8.4566	-17.0334	
					a
Temkin	303K	0.9851	0.0021	5.3180	8.9960
	313K	0.9801	0.0038	4.0168	5.4035
	333K	0.9898	0.0009	8.1718	4.2663
					x
Florry-Huggins	303K	0.7314	0.0034	4.1891	4.2533
	313K	0.9621	0.0082	2.0360	4.1168
	333K	2.9580	0.0401	-2.2159	2.9580

					α
Frumkin	303K	0.9879	0.4815	-8.2782	5.2064
	313K	0.9820	0.7043	-9.5412	3.4719
	333K	0.9556	0.5448	-9.4398	2.8355
					n
Freundlich	303K	0.9882	0.0451	-2.3123	0.2979
	313K	0.9854	0.0337	-1.6296	0.4019
	333K	0.9440	0.1182	-5.2086	0.2841
					1/y
El-Awady	303K	0.9909	0.0001	11.7085	2.7100
	313K	0.9923	0.0010	7.4010	1.8656
	333K	0.9843	0.0086	2.0448	1.9029

4. CONCLUSIONS

Using Lagenaria Siceraria Leaves (LSL) extract Carbon steel in 1.0N Hydrochloric acid Lagenaria Siceraria Leaves have shown excellent inhibition performance for Carbon steel in 1.0N Hydrochloric acid. The inhibition efficiency increased with the increase of inhibitor concentration (0 to 1000ppm). The maximum percentage of inhibition efficiency was achieved 84.62%. Also, the inhibition efficiency gradually increased with the rise in temperature i.e., to 73.68% for 333K. It follows physical adsorption mechanism. The activation energy (E_a), Standard free energy adsorption (ΔG_{ads}), enthalpy (ΔH), entropy (ΔS), suggests that, Physisorption, exothermic, spontaneous process respectively. The LSL inhibitor obeys Frumkin adsorption isotherm.

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