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Inhibition of Mild Steel Corrosion in 1.0 M HCl Solution by Octadecylamine as Corrosion Inhibitor

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ABSTRACT

The inhibitive action of octadecylamine with various concentrations on mild steel in 1.0 M HCl solution was carried out by using chemical (weight loss method) and electrochemical techniques (Potentiodynamic polarization and Electrochemical Impedance Spectroscopy (EIS)). In general, the inhibition efficiency increased and corrosion rates decreased as the increasing concentrations of octadecylamine at each temperature with same immersion time period. But on the other hand the inhibition efficiency decreased and corrosion rate increased with increasing temperature at same concentration. The inhibition was assumed to occur via adsorption of the inhibitor molecule on the metal surface. Electrochemical impedance measurement shows that charge transfer resistance increased and double layer capacitance decreased with increasing inhibitor concentrations indicates the adsorption mechanism. Polarization curves revealed that octadecylamine behave as mixed type inhibitor in 1.0 M HCl solution. The adsorption of the inhibitor was well described by the Langmuir adsorption isotherm and the adsorption isotherm parameters were determined at different temperatures. The results from all techniques show that octadecylamine acts as a very good inhibitor.

Keywords: Mild Steel, HCl, Inhibitor, EIS, Octadecylamine, Adsorption isotherm

INTRODUCTION

Mild steel has found many applications in industrial like automobiles, pipes and as boilers etc., and it is a commonly used material because of its low cost, good ductile strength and easy availability [1-3]. Due to the industrial environment the mild steel is generally affected by corrosion attack because of uses of different acids (like hydrochloric acid, nitric acid and sulphuric acid) for different industrial process like acid pickling, descaling and chemical cleaning etc. [4-6]. Hence corrosion of mild steel is a very serious problem and most difficult challenging for many industries to protect the steel against corrosion process. Corrosion decreases the functional properties and also decays the valuable properties of mild metal. For protection the metal against corrosion many researcher work in this field and found that the use of inhibitors are one of the best approach in acidic medium [7-11]. Corrosion inhibitors are those substances which are added in small quantity in corrosive media decrease the corrosion rates of metal and cover the metal surface by formation of thin protective layer which protect against corrosion [12]. It also decreasing the dissolution of metal and enhance the adsorption process. Throughout literature survey we observed that many organic, inorganic and pharmaceutical compounds were found to be capable as corrosion inhibitor of metals especially in acidic medium. But among them the use of organic compound best approach as corrosion inhibitor because these inhibitors usually have heteroatom's (like nitrogen, sulphur, phosphorus and oxygen etc.,) [13-18] and containing aromatic rings and pi- electrons in which simply heteroatom with free electron pair are easily available for sharing with metal and donating their electron to the surface and form a bond between metal and inhibitor that reduce the corrosion rates. Hence in this process, Inhibitors act as nucleophile and metal act as electrophile centers. The effective properties of these inhibitors usually depend on their adsorption rates and inhibition efficiency depend upon molecular structure, steric factor, electron density and lone pair present on the heteroatom molecules [19-22]. These organic inhibitors are usually adsorbed on the metal surface by the formation of a coordinate covalent bond (chemical adsorption) or the electrostatic interaction between the inhibitor and the metal (physical adsorption) [23-26]. In recent few years several investigations have reported the use of long chain amine as an efficient corrosion inhibitor on mild steel in acidic medium. In the present study we investigated the inhibition activity of octadecylamine as corrosion inhibitor on mild steel in 1.0 M hydrochloric solution using different techniques like weight loss measurement, electrochemical impedance spectroscopy, potentiodynamic polarization and adsorption isotherm.

EXPERIMENTALS STUDIES

Preparation of materials

All experiments were performed using mild steel specimens. Mild steel specimens used were purchased from local market having composition (weight%): C 0.054%, Mn 0.26%, Si 0.015%, Ni 0.0098%, Cr 0.056%, S 0.017%, P 0.019%, Cu 0.010, Mo 0.018, Ti 0.002 and remaining Fe.

Inhibitor

Octadecylamine was purchased from CDH, New Delhi (AR). Molecular formula and molecular mass of octadecylamine is $C_{18}H_{39}N$, 269.51 g/mol and molecular structure of inhibitor shown in Scheme 1.



Scheme 1: Structure of octadecylamine

Preparation of solutions

Aggressive solution of 1.0 M HCl was used for experimental purpose and prepared by dilution of analytical grade 37% HCl with double distilled water. Different concentrations of inhibitor were made up by dilute with 1.0 M HCl solution and concentration ranges taken from 200 ppm to 1000 ppm.

Weight loss techniques

Weight loss measurement carried out in air thermostat for mild steel in 1.0 M HCl solution to study the corrosion behavior of specimen with different immersion time periods (12, 18 and 24 h) at different temperature (25.0°C, 35°C and 45.0°C). Firstly the specimens were cut in to 1×3 cm² from freshly prepared mild steel was used. Before the experiment start all the specimens were polished with different grade emery papers using ranges 100, 200, 400, 600 and 1000 Silicon Carbide (SiC), washed with double distilled water, degreased with acetone and dry the specimen with hot air blower. After that initial weight of the polished prepared specimens were measured and then immersed in to the 30 ml test solution that taken in 50 ml beaker for time periods at different temperatures. After the completion the time periods the mild steel specimens were taken out from the test solution, washed with double distilled water and again degreased with acetone, dry with blower and finally weighted the specimens with accuracy. Triplicate experiments were carried out for each time periods and different temperature ranges and average weight loss data was recorded.

Percentage corrosion inhibition efficiency (IE%), corrosion rate (C_R) and surface coverage (θ) were calculated by using following Equations:

$$IE (\%) = \frac{w_o - w_i}{w_o} \times 100 \quad (1)$$

$$\theta = \frac{w_o - w_i}{w_o} \quad (2)$$

Where, W_o and W_i are the weight loss value of mild steel sample in absence and presence of inhibitor, respectively. The corrosion rate (C_R) of mild steel was calculated using the relation:

$$C_R (\text{mmy}^{-1}) = \frac{87.6 \times w}{A t D} \quad (3)$$

Where, 'w' is weight loss of mild steel in 'mg', A is the area of the coupon (cm²), t is the exposure time (h) and D is the density of mild steel (g/cm³).

Electrochemical measurements

The electrochemical behaviors of the mild steel specimens in 1.0 M HCl solution at room temperature were investigated by both Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic polarization slope method. These measurements were carried out in a conventional three electrodes electrochemical cell with a platinum counter electrode and Saturated Calomel Electrode (SCE) as reference electrode and mild steel specimens were used as a working electrode. Both experiments were performed using Merton Auto lab Model PGSTAT 128N potentiostat. The surface of mild steel specimens with size 1×5 cm² were polished with different grades emery paper (100-1000), washed with double distilled water, degreased with acetone then dried with hot air blower. After that exposed area of each specimen were taken 1×1 cm² and rest being covered by using commercially available lacquer. Before every measurement of EIS and polarization the mild steel specimen was immersed in test solution for 30-35 min at room temperature to stabilize the value of Open Circuit Potential (OCP) [27] after stable value of OCP then preceding the experiment.

EIS measurements were carried out at open circuit potential in the frequency range of 10 kHz to 0.01 Hz with signal amplitude of 10 mV peak to peak at the corrosion potential. The experiments were carried out in the absence and presence of different concentrations of inhibitor which were previously used in weight loss experiments. The measurements were automatically controlled by Z-view software and the impedance diagrams were given in the Nyquist representation. Electrochemical impedance parameters such as Charge Transfer Resistance (R_{ct}) and Double Layer Capacitance (C_{dl}) were calculated from EIS data.

The Potentiodynamic polarization measurements were made after EIS studies in the same cell setup, in a potential range of -200 mV to +200 mV with respect to open circuit potential, at a scan rate of 1.0 mV/s. From the Tafel plots, the corrosion potential E_{corr} , corrosion current density I_{corr} , Tafel slopes β_a and β_c were obtained in the absence and presence of inhibitors at different concentrations.

RESULT AND DISCUSSION

Weight loss measurement

Effect of inhibitor concentration

The values of corrosion rate and inhibition efficiency obtained by weight loss measurement in 1.0 M HCl solution are listed in Tables 1-3. It has been found that the corrosion rate decreases and inhibition efficiency increases as the concentration of inhibitor increases towards higher side up to 0 ppm to 1000 ppm that might be due to the increased adsorption of inhibitor on the metal surface with increasing the concentration of octadecylamine (Figures 1 and 2).

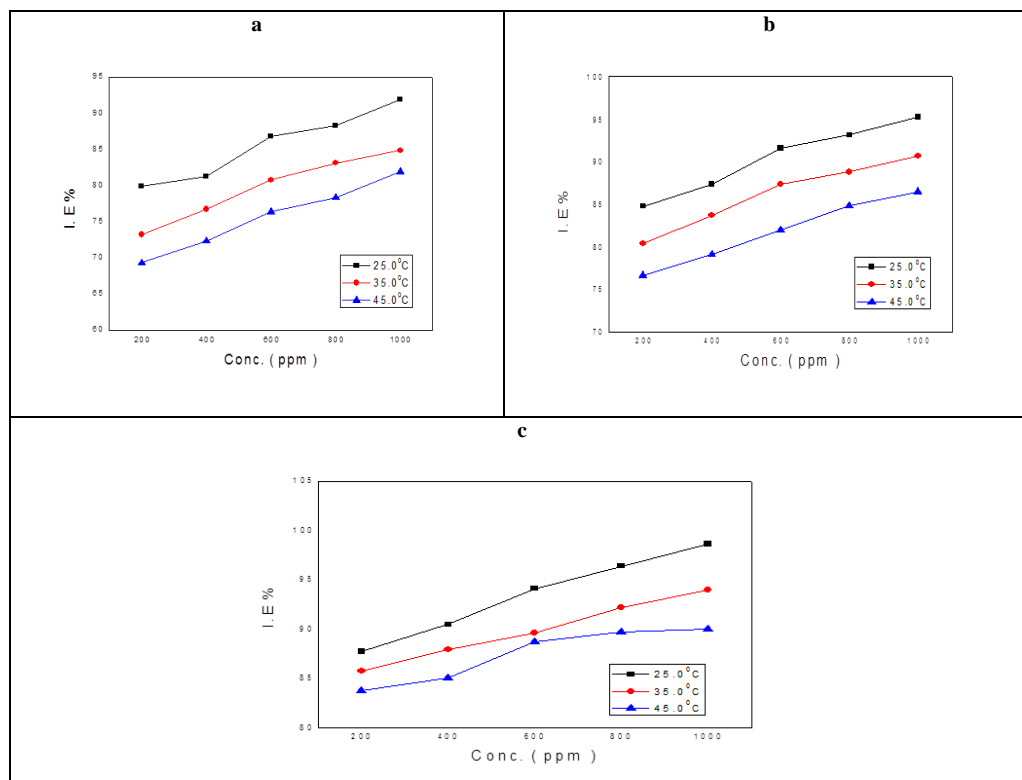
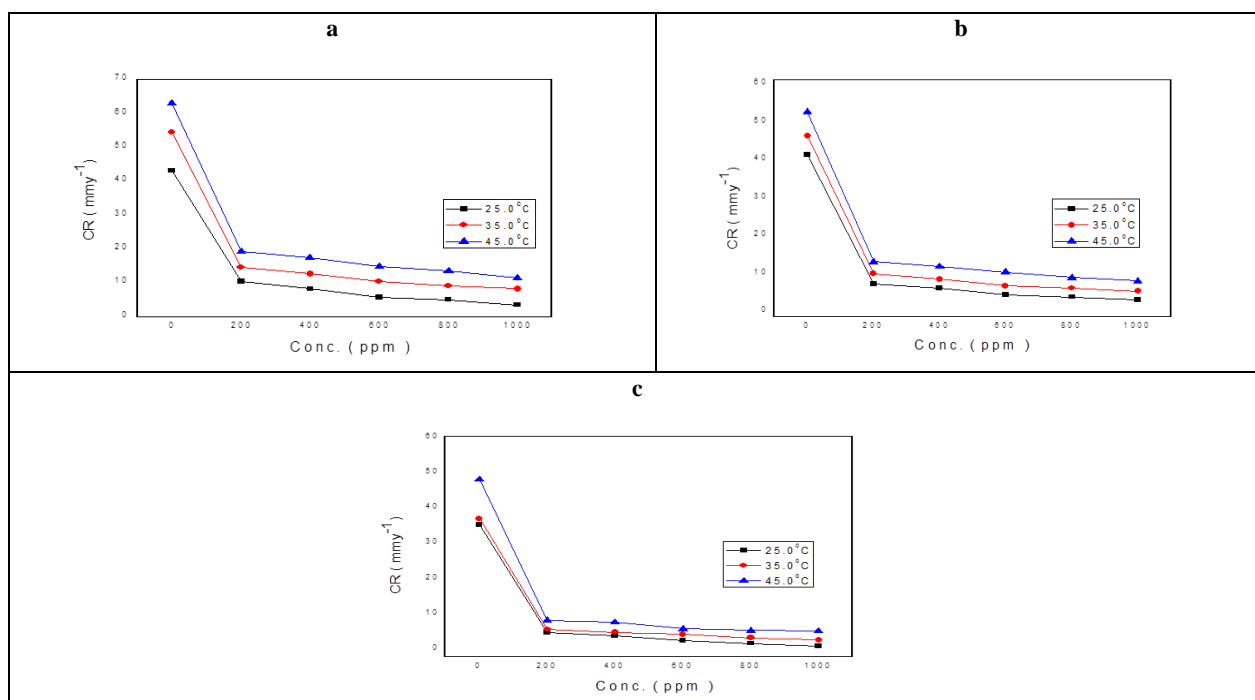


Figure 1: (a-c) Variation of inhibition efficiencies with different concentration of inhibitor at different temperatures for different immersion times (12 h, 18 h, 24 h)



Figures 2: (a-c) Variation of corrosion rates with various concentration of inhibitor at different temperatures for different immersion times (12 h, 18 h, and 24 h)

Table 1: Inhibition efficiency values for the corrosion of mild steel in acid solution of 1.0 M HCl in the absence and in the presence of different concentrations of Octadecylamine from weight loss measurements at 25.0°C, 35.0°C and 45.0°C for immersion time of 12 h

Inhibitor and temperature	Concentration (ppm)	Weight before (mg)	Weight after (mg)	Weight loss (mg)	Corrosion rate (mm ^y - ¹)	Inhibition efficiency (%)	Surface coverage (θ)
Octadecyl Amine (25°C)	0	1125	988	137	43.6	-	-
	200	1103	1070	33	10.45	79.91	0.79
	400	1126	1100	26	8.23	81.28	0.81
	600	1117	1099	18	5.7	86.86	0.86
	800	1112	1096	16	5.06	88.32	0.88
	1000	1124	1113	11	3.48	91.97	0.91
(35°C)	0	1144	972	172	54.49	-	-
	200	1094	1048	46	14.57	73.25	0.73
	400	1149	1109	40	12.67	76.74	0.76
	600	1126	1093	33	10.45	80.81	0.8
	800	1120	1091	29	9.18	83.14	0.83
	1000	1113	1087	26	8.23	84.88	0.84
(45°C)	0	1132	933	199	63.05	-	-
	200	1130	1069	61	19.32	69.34	0.69
	400	1116	1061	55	17.42	72.36	0.72
	600	1123	1076	47	14.89	76.38	0.76
	800	1127	1084	43	13.62	78.39	0.78
	1000	1137	1101	38	11.4	81.91	0.81

Table 2: Inhibition efficiency values for the corrosion of mild steel in acid solution of 1.0 M HCl in the absence and in the presence of different concentrations of Octadecylamine from weight loss measurements at 25.0°C, 35.0°C and 45.0°C for immersion time of 18 h

Inhibitor and temperature	Concentration (ppm)	Weight before (mg)	Weight after (mg)	Weight loss (mg)	Corrosion rate (mm ^y - ¹)	Inhibition efficiency (%)	Surface coverage (θ)
Octadecyl amine (25°C)	0	1144	954	190	40.13	-	-
	200	1152	1123	29	6.12	84.73	0.84
	400	1146	1122	24	5.06	87.36	0.87
	600	1135	1119	16	3.37	91.57	0.91
	800	1106	1093	13	2.74	93.15	0.93
	1000	1139	1130	9	1.9	95.26	0.95
(35°C)	0	1151	937	214	45.2	-	-
	200	1147	1105	42	8.87	80.37	0.8
	400	1124	1089	35	7.39	83.64	0.83
	600	1090	1063	27	5.7	87.38	0.87
	800	1094	1070	24	5.06	88.78	0.88
	1000	1137	1117	20	4.22	90.65	0.9
(45°C)	0	1141	897	244	51.53	-	-
	200	1136	1079	57	12.03	76.63	0.76
	400	1153	1102	51	10.77	79.09	0.79
	600	1117	1073	44	9.29	81.96	0.81
	800	1123	1086	37	7.81	84.83	0.84
	1000	1119	1086	33	6.97	86.47	0.86

Table 3: Inhibition efficiency values for the corrosion of mild steel in acid solution of 1.0 M HCl in the absence and in the presence of different concentrations of octadecylamine from weight loss measurements at 25.0°C, 35.0°C and 45.0°C for immersion time of 24 h

Electrochemical impedance spectroscopy measurements

All the experimental results obtained from EIS measurements (techniques) for the corrosion of mild steel at 25.0°C temperature after immersion of 30 min in 1.0 M HCl solution with different concentrations of inhibitor are summarized in Table 4. The impedance spectra of mild steel are present in the form of Nyquist plots are shown in Figure 3. It is clear from the graph that the plots are semicircles in nature and diameter of semicircle increases with increasing the concentration of inhibitor. It shows that the impedance of mild steel is increased with increasing in concentration of inhibitor & also shows adsorption of inhibitor on surface of mild steel.

Noticeably, these impedances are not perfectly semicircle which can be attributed to frequency dispersion as a result of no homogeneity or roughness of metal surface [28,29]. The corrosion of mild steel is usually related to charge transfer resistance and double layer capacitance behavior of corrosion process. The calculated values of charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}) and percentage inhibition efficiency (IE%) are listed in Table 4.

Inhibitor and temperature	Concentration (ppm)	Weight before (mg)	Weight after (mg)	Weight loss (mg)	Corrosion rate (mm^{-1})	Inhibition efficiency (%)	Surface coverage (θ)
Octadecyl Amine (25°C)	0	1116	896	220	34.85	----	----
	200	1093	1066	27	4.27	87.72	0.87
	400	1130	1109	21	3.32	90.45	0.9
	600	1149	1136	13	2.05	94.09	0.94
	800	1091	1083	8	1.26	96.36	0.96
	1000	1124	1121	3	0.47	98.63	0.98
(35°C)	0	1088	857	231	36.59	----	----
	200	1094	1061	33	5.22	85.71	0.85
	400	1121	1093	28	4.43	87.87	0.87
	600	1102	1078	24	3.8	89.61	0.89
	800	1116	1098	18	2.85	92.2	0.92
	1000	1113	1099	14	2.21	93.93	0.93
(45°C)	0	1155	854	301	47.68	----	----
	200	1132	1083	49	7.76	83.72	0.83
	400	1116	1070	46	7.28	85.05	0.85
	600	1123	1089	34	5.38	88.7	0.88
	800	1137	1106	31	4.91	89.7	0.89
	1000	1144	1114	30	4.75	90.03	0.9

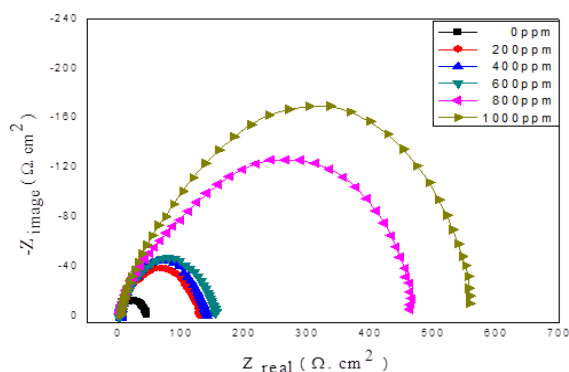


Figure 3: Nyquist plots of the corrosion of mild steel in 1.0 M HCl with and without the different concentrations of inhibitor at 25.0°C temperature

It is observed from the table that by increasing the concentration of inhibitor the double layer capacitance (C_{dl}) values decreases and charge transfer resistance values increases which shows the adsorption of inhibitor on the metal/solution interface surface leading to the formation of thin layer film [30]. The decreases in the (C_{dl}) values with concentration the situation were the result from a decrease in local dielectric constant [9,31] and increase in the thickness of the electric double layer.

Table 4: Impedance parameters and their corresponding inhibition efficiency in presence and absence of different concentrations of inhibitor at 25.0°C

Inhibitor name	Concentration (ppm)	R_{ct} ($\Omega \cdot \text{cm}^2$)	C_{dl} ($\mu\text{F} \cdot \text{cm}^{-2}$)	%IE
Octadecyl amine	0	43.82	151.47	-
	200	128.25	51.75	65.83
	400	137.17	48.38	68.05
	600	153.18	43.33	71.39
	800	462.8	14.34	90.53
	1000	554.58	11.96	92.09

The charge transfer resistance value calculated from the difference in impedance (Z_{real}) at lower to higher frequencies [32]. The value of double layer capacitance are obtained from the frequency at which the imaginary component of impedance is ($-Z_{max}$) maximal and calculated from following Equation [33,34]:

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad (4)$$

Where, f_{max} is the frequency value at the top of semicircle and it is midway of Z_{real} & also at which Z_{image} Component is maximal. The percentage inhibition efficiency is calculated from using charge transfer resistance value by following Equation [35].

$$I.E. \% = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \quad (5)$$

Where, R_{ct} and $R_{ct(inh)}$ are the charge transfer resistance in absence and presence of different concentrations of inhibitor. The maximum efficiency found to be 92.09% at higher concentration (1000 ppm).

Potentiodynamic polarization measurements

The effect of octadecylamine on the cathodic and anodic polarization curve for mild steel in 1.0 M HCl solution in presence and absence of various concentrations of inhibitor are shown in Figure 4 as Tafel plots. From inspection of the figure it found that the inhibitor shifted the both cathodic and anodic branches of Tafel curve to lower value of current density, it indicates the inhibitor act as mixed type inhibitor (both cathodic and anodic). It also shows that the inhibitor compound retard the corrosion process on the surface of mild steel. The various electrochemical polarization parameters value obtained from these curves including corrosion potential (E_{corr}), corrosion current density (I_{corr}), cathodic (β_c) and anodic (β_a) Tafel constant and inhibition efficiency are given in Table 5.

By the investigation of table it is revealed that, with increasing the concentration of inhibitor the corrosion current density (I_{corr}) values decreases significantly. Hence these results shows that the reduction of the anodic dissolution and retards the hydrogen evolution at cathode or controlled the cathodic and anodic reactions on the mild steel surface. This is due to the effect of inhibitor which adsorb strongly on the surface at higher concentration. Also, the data from table showed that the anodic Tafel constant (β_a) and cathodic Tafel constant (β_c) values were slightly changed with addition inhibitor concentrations, this also indicates that these inhibitors affected both the anodic and cathodic reactions. But the values of cathodic Tafel constant are higher than anodic which show the inhibitor effect on the cathodic hydrogen evolution reactions than on the anodic dissolution reactions.

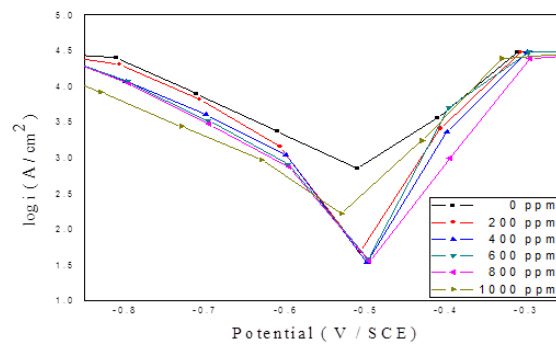


Figure 4: Potentiodynamic polarization curves for mild steel in 1.0 M HCl without and with the different concentrations of inhibitor at 25.0°C temperature

The values of corrosion current density (I_{corr}) calculated by using following equation (Stern-Geary equation). The values of polarization resistance, cathodic & anodic Tafel constant are used to calculate the corrosion current (I_{corr}).

$$I_{corr} = \frac{\beta_a \times \beta_c}{2.303(\beta_a + \beta_c)} \times \frac{1}{R_p} \quad (6)$$

Where, R_p is the polarization resistance, β_a and β_c are the cathodic and anodic Tafel constant. The inhibition efficiency attains a maximum value that based on I_{corr} value found to be 83.97% at 1000 ppm concentration of inhibitor and maximum reduction of I_{corr} value found 1.54 $\mu A cm^{-2}$ at same concentration, which also conform the inhibitor is good inhibitor in 1.0 M HCl solution. The efficiency is calculated from using following equation [36].

$$IE\% = \left(1 - \frac{I_{corr}^i}{I_{corr}^0}\right) \times 100 \quad (7)$$

Where, I_{corr}^0 and I_{corr}^i are the corrosion current density without and with different concentrations of inhibitor respectively.

Table 5: Potentiodynamic polarization measurement for mild steel samples in 1.0 M HCl with and without different concentration of inhibitor at 25.0°C

Inhibitor name	Concentration (ppm)	β_a (mVd ⁻¹)	β_c (mVd ⁻¹)	R_p ($\Omega.cm^2$)	$-E_{corr}$ (mV vs. SCE)	i_{corr} ($\mu A cm^{-2}$)	%IE
Octadecyl amine	0	412	1326	14.6	-489	9.61	-
	200	351	1227	29.8	-507	3.97	58.68
	400	327	1284	34.3	-499	3.29	65.76
	600	364	1179	43.6	-538	2.76	71.28
	800	319	1212	59.7	-495	1.83	80.95
	1000	343	1131	73.8	-530	1.54	83.97

There was no definite trend in the shift of E_{corr} values with the presence and absence of inhibitor in 1.0 M HCl solution which can be recognized as a classification evidence of the compounds whether it is mixed-type inhibitors [36-39]. According to Riggs and Ferreira [40,41]. (a) If the value of displacement in $(-E_{\text{corr}})$ is higher than 85 mV with respect to $(-E_{\text{corr}})$, the inhibitor can be seen as anodic or cathodic type inhibitor, and (b) if value of displacement in $(-E_{\text{corr}})$ is lower than 85 mV, the inhibitor can be acts as mixed type inhibitor.

In present work the maximum displacement in (E_{corr}) is 49 mV, which clearly indicates that octadecylamine act as mixed type inhibitor or exhibited both anodic and cathodic type inhibition.

Adsorption isotherm

The adsorption isotherms are important in determining and understanding the mechanism of corrosion inhibitor and also adsorption behavior inhibitor on the metal surface. The isotherm can provide additional information about the inhibitor properties.

The several isotherms like Temkin, Freundlich, Frumkin and Langmuir isotherm etc., are used most frequently as adsorption isotherm. Throughout these various types of adsorption isotherm the in Langmuir isotherm straight line was found to best fit for the experimental data shown in Figures 5a-c. This behavior is due to strong correlation ($R^2 > 0.99$) suggest that the adsorption of octadecylamine obeyed this type of adsorption isotherm on the mild steel surface. Figures show that straight line is observed from the plot of C_{inh}/θ Vs C_{inh} as given in following Equation [42,43].

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K_{\text{ads}}} + C \quad (8)$$

Where, C_{inh} is the concentration of inhibitor, θ is the surface covered which is obtained from the weight loss by using equation inhibition $E\%/100$ and K_{ads} could be calculated from the intercepts of the straight lines on the C_{inh}/θ axis, the equilibrium constant of the adsorption relation to the standard free adsorption energy is calculated from the equilibrium constant using following Equation:

$$-\Delta G_{\text{ads}} = -RT (\ln 55.5 K_{\text{ads}}) \quad (9)$$

Where, R is the universal gas constant ($8.314 \text{ J}\cdot\text{mol}^{-1} \text{ K}^{-1}$), T is absolute temperature and 55.5 is the concentration of water [44,45] in solution (mol/l) The thermodynamic adsorption parameters, K_{ads} and $-\Delta G_{\text{ads}}$ values are illustrate in Table 6. Generally it can be seen that if the absolute value of ΔG_{ads} is lower than -20 kJ/mol , they are consistent with the electrostatic interaction between the charged molecules and the charged metal called physical adsorption (physisorption) [46], while those that are more negative than -40 kJ/mol , which involve sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a coordinate bond called chemical adsorption (chemisorption) [47-49]. From the table the value of ΔG_{ads} for octadecylamine are found to be ranged from -23.83 to $-28.34 \text{ kJ}\cdot\text{mol}^{-1}$ calculated by using Equation 9, which indicate that the adsorption process of inhibitor on metal surface is mixed physical and chemical adsorption [50-23]. Also the negative values of ΔG_{ads} revealed that the adsorption of inhibitor on to the mild steel is a spontaneous process for different immersion periods usually shows the strong interaction on the surface.

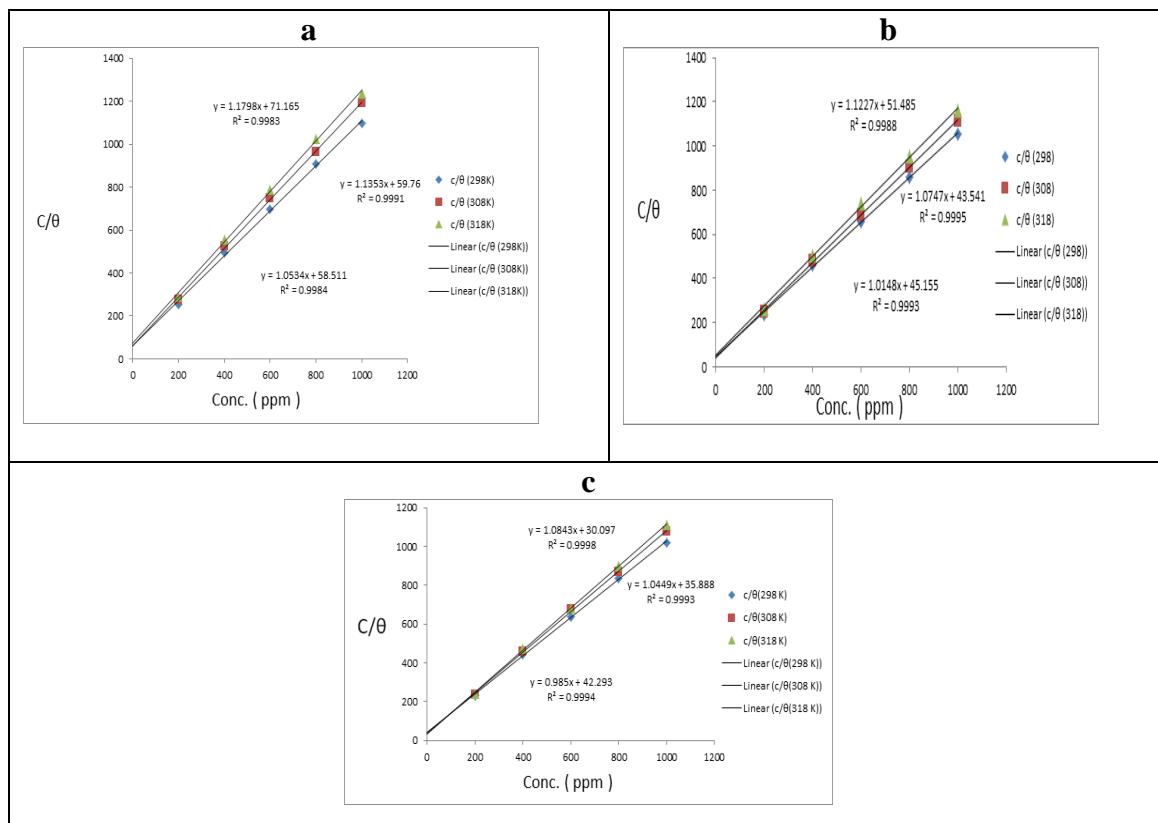


Figure 5): (a-c) Langmuir adsorption isotherm plot for the mild steel in 1.0M HCl solution with different concentration of octadecylamine at different temperatures for immersion time 12, 18 h and 24 h

Table 6: Thermodynamic adsorption parameters for mild steel in 1.0 M HCl in the presence of various concentration of octadecylamine at different temperatures

Inhibitor name	Temperature (k)	Log 55.5 K _{ads}	-ΔG _{ads} (kJ mol ⁻¹)
Octadecyl amine 12 h	298	4.176	-23.83
	308	4.357	-25.694
	318	4.281	-26.067
18 h	298	4.478	-25.555
	308	4.494	-26.505
	318	4.421	-26.923
24 h	298	4.5	-25.717
	308	4.57	-27.001
	318	4.65	-28.343

CONCLUSION

The following conclusions are drawn by the obtained result from various techniques used: Octadecylamine act as a good corrosion inhibitor for the corrosion of mild steel in 1.0 M HCl solution. By weight loss measurement the inhibition efficiency increases with increasing concentrations of inhibitor but decrease with increasing temperature. The inhibition efficiency reaches at maximum value i.e., 98.63% at 1000 ppm.

Potentiodynamic measurement indicates that it is a mixed type corrosion inhibitor by decreasing both the anodic metal dissolution and cathodic hydrogen reduction reactions. EIS data depicted increases in charge transfer resistance and decreases in double layer capacitance when the increasing concentrations of inhibitor in 1.0 M HCl solution. This explained the formation of protective layer film on to the mild steel surface. The adsorption of Octadecylamine on mild steel surface obeys the Langmuir adsorption isotherm.

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