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Study of corrosion inhibition properties of Tetradecylamine for mild steel in 1.0 M HCl solution

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Abstract

The corrosion inhibition behavior of Tetradecylamine on mild steel in 1.0 M HCl solution was investigated at different concentration and temperature i.e. 298, 308 and 318K for immersion time periods of 24.0 hrs by Weight loss measurement, Electrochemical impedance spectroscopy (EIS), Potentiodynamic polarization measurement, adsorption isotherm and SEM (Scanning Electron Microscope). The results shows that the Tetradecylamine act as excellent inhibitor and inhibition efficiency increases and corrosion rate decreases with concentration but reverse occur with increasing the temperature. The maximum inhibition efficiency was found 93.10% for 1000 ppm concentration at 298 K temperature for 24.0 hours. Data obtained from electrochemical impedance spectroscopy shows that charge-transfer resistance (R_{ct}) increases and the capacitance of double layer (C_{dl}) decreases with the inhibitor concentration, confirming the adsorption process mechanism. Polarization curve shows that the Tetradecylamine behave as mixed type inhibitor in 1.0 M HCl solution with different concentration. The adsorption of inhibitor obeyed the Langmuir adsorption isotherm. The formation of protective layer on the mild steel surface was confirmed by the SEM techniques.

Keywords: mild steel, Tetradecylamine, electrochemical impedance spectroscopy, potentiodynamic polarization, HCl, SEM

Introduction

Acid solution is extensively used in many industries for acid picking, acid descaling, oil-well acidifying, ore production and also for removal of rust content from the pipelines ^[1-7]. The acids used in industries are hydrochloric acid, sulphuric acid and nitric acid etc. but among them the most commonly used acid is hydrochloric acid for picking of metal because of more economical, efficient than other mineral acid ^[8]. Mild steel used in many industries mainly petroleum, power production and machinery due to its excellent ductile strength, low cost and easily availability [9-10]. The main drawback of mild steel is that it is highly sensitive to corrosion attack and it is also dissolute in acidic medium ^[11]. Hence the corrosion of mild steel is of fundamental academic and industrial concern. There is various methods to protect the metal but one of the best method is the use of corrosion inhibitors especially in acidic medium ^[12-19]. The main role of corrosion inhibitors is that it mitigates the corrosion of metal by acid attack and also reduces the corrosion attack on metal surface.

Different type of organic, inorganic and drugs are used as corrosion inhibitors in acidic medium. But among them the most well known corrosion inhibitors are organic compounds in many aggressive acidic medium to protect the metal against corrosion ^[20, 21]. The organic compounds which containing heteroatom's (i.e N, O and S etc.), conjugative double bonds, aromatic rings were found to be best corrosion inhibitors. The effectiveness of these corrosion inhibitors are mainly attributes to their molecular structure, steric factor, electron density and lone pair present on the heteroatom, adsorption tendencies etc. ^[22]. It has been seen that the nitrogen containing hetrocyclic organic compounds and amines are

approach to be best corrosion inhibitors for mild steel in acidic medium ^[23-25].

In the present study we discuss the effect of Tetradecylamine on corrosion process for mild steel in 1.0 M HCl solution at different temperature (i.e. 298, 308, and 318K) for immersion time period 24.0 hrs using weight loss measurement, electrochemical impedance spectroscopy, potentiodynamic polarization, adsorption isotherm and scanning electron microscope.

Experimental Materials and methods Material Preparation

Mild steel strips were used for study having following chemical composition (weight %): C=0.054, Mn=0.26, Cu=0.010, Ti=0.002, Cr=0.056, S=0.017, P=0.019, Mo=0.018, Si=0.015, Ni =0.009 and remained iron. Firstly, for experimental purpose mild steel sheet was cut into pieces of 1×3 cm² dimensions for weight loss experiment and into 1X5 cm² dimensions for electrochemical measurements. The specimens were then abraded with emery papers of different mesh grades (100-1000). Then specimens were washed thoroughly with double distilled water, degreased with acetone, dried in hot air blower and then placed in desiccators for experiment study.

Inhibitor

Tetradecylamine were purchased from Sigma-Aldrich (99%) to use as corrosion inhibitor without further purification for study. The molecular mass and molecular formula of Tetyradecylamine is 213.409 g/mol and $C_{14}H_{31}N$ respectively.



Fig 1: Structure of Tetradecylamine

Solutions Preparation

The aggressive solution of 1.0 M HCl was prepared by using concentrated HCl of analytical grade 37% by diluting with double distilled water. The solution was then used to make 1000 ppm stock solution of inhibitor. This stock solution of inhibitor was again diluted with 1.0 M HCl to make desired concentrations ranging from (0-1000ppm).

Weight loss measurement

For weight loss measurement same procedure was adopted as in ^[26, 27]. The weight loss measurements were carried out in 50mL test solution for 24.0 hrs at different temperature ranges i.e. 298, 308 and 318K. Prior to every experiment the initial weight of specimens were taken and then placed in test solution for fixed time period at different temperature. After completion of time period, the specimens were taken out, rinsed with double distilled water, cleaned with acetone and dried between folds of filter paper then finally weighted. Experiment was performed in triplicate and then average weight loss was recorded by this weight loss study. From weight loss values percentage corrosion inhibition efficiency ($\eta \le \%$) and surface coverage (θ) were calculated using the following equations.

$$\eta_{w} \% = \frac{w_{\sigma} - w_{i}}{w_{\sigma}} x 100....(1)$$

$$\theta = \frac{w_{\sigma} - w_{i}}{w_{\sigma}}...(2)$$

Where, w_o and w_i are the weight loss value of mild steel in absence and presence of inhibitor respectively. The corrosion rate of mild steel was calculated by using equation:-

Where w is weight loss of mild steel in mg, A is area of sample (cm^2), t is exposure time (hrs) and D is density of mild steel (g cm⁻³)^[28].

Electrochemical Measurement

Mild Steel samples of $1x5cm^2$ dimensions were used for electrochemical study and prepared by rubbing with (100-1000) grades of emery papers, washed with double distilled, degreased with acetone, dried and then put into desiccators for further experiment. The sample with exposed surface area 1.0 \times 1.0 cm² was selected as working electrode and remaining covered with epoxy resin. Electrochemical impedance and Potentiodynamic polarization were carried out by using AUTOLAB Salatron model 1280B Potentiostat. Three electrode assembly were used in the system where platinum act as counter electrode(CE), calomel electrode as reference electrode(RE) and mild steel as working electrode. Before starting experiments, the electrodes were immersed in 50mL test solution at open circuit potential (OCP) for about 30 minutes at room temperature so as to attain a stable value of OCP.

Electrochemical impedance spectroscopy

The impedance measurements were carried out in frequency range of 10 kHz to 0.01Hz with signal amplitude of 10 mV at its corrosion potential (- E_{corr}). The experiments were conducted in absence and in presence of different concentrations of inhibitor. All impedance measurements were automatically controlled by Z-view software and the impedance diagrams were obtained in the form of Nyquist plots. Electrochemical impedance parameters such as Charge Transfer Resistance (R_{ct}) and Double Layer Capacitance (C_{dl}) were calculated using EIS data.

Potentiodynamic Polarization

The polarization behaviour of mild steel specimens was studied in absence and presence of inhibitor by recording anodic and cathodic potentiodynamic polarization curves in the potential range of -200 mV to +200 mV with respect to open circuit potential, at a scan rate of 1.0 mV/sec. The linear Tafel segments of anodic and cathodic curves were extrapolated to corrosion potential in order to get corrosion current densities (i_{corr}). Through polarization curves, the corrosion kinetic parameters such as corrosion potential (- E_{corr}), corrosion current density (I_{corr}), cathodic (β_e) and anodic (β_a) Tafel constant were obtained.

SEM analysis

The surface morphology of specimens immersed for 24.0 hrs in absence and presence of 1000ppm concentrations of inhibitor at temperature of 298, 308 and 318K were analyzed through ZEISS Scanning Electronic Microscope.

Result and Discussion Weight loss method Effect of inhibitor concentration

By the weight loss measurement the obtained value of the corrosion inhibition efficiency, surface coverage and corrosion rates using various concentrations of tetradecylamine as corrosion inhibitor in 1.0 M HCl solution at different temperature i.e 298, 308 and 318K for immersion time periods of 24 hrs are summarized in Table 1. The data in Table 1 shows that inhibition efficiency increases with increasing the concentration of inhibitor. Therefore, the corrosion rates value decreases with increasing with the inhibitor concentration in 1.0 M HCl solution. This behavior can be explained on the basis of strong interaction of inhibitor compound on to the mild steel surface that shows the adsorption of inhibitor ^[29, 30].

Effect of temperature

The effect of temperature on inhibition efficiency was studied by weight loss measurement. The variation of corrosion inhibition efficiencies and corrosion rates with different concentration and temperature are shown in Figure 1 and 2. From the figures, it is apparent that the inhibition efficiency



Fig 1: Variation of Corrosion Rates with Various Concentration of Tetradecylamine at Different Temperatures (298, 308 and 318K) for Immersion Time Period 24.0 hours.

decreases but corrosion rates increases with increasing temperature from 298 to 318K.



Fig 2: Variation of Inhibition Efficiency with Different Concentrations of Tetradecylamine at Different Temperatures (298, 308 and 318K) for Immersion Time Period 24.0 hours.

Corrosion	Immersion Time	Concentrations	Weight	Corrosion	Percentage Inhibition	Surface	
Inhibitor Name	Periods	(nnm)	Loss	Rates	Efficiencies	Coverage	
	(hrs)	(ppm)	(mg)	(mmy ⁻¹)	(% I. Ew)	(θ)	
	298	0	0.203	32.15	-	-	
		200	0.039	6.17	80.78	0.80	
		400	0.031	4.91	84.72	0.84	
		600	0.028	4.43	86.20	0.86	
		800	0.026	4.11	87.19	0.87	
		1000	0.014	2.21	93.10	0.93	
	308	0	0.222	35.16	-	-	
T. (200	0.053	8.39	76.12	0.76	
amine (24.0 hrs)		400	0.045	7.12	79.72	0.79	
		600	0.043	6.81	80.63	0.80	
		800	0.039	6.17	82.43	0.82	
		1000	0.027	4.27	87.83	0.87	
	318	0	0.301	47.68	-	-	
		200	0.097	15.36	67.77	0.67	
		400	0.081	12.83	73.08	0.73	
		600	0.072	11.40	76.07	0.76	
		800	0.059	9.34	80.39	0.80	
		1000	0.051	8.07	83.05	0.83	

Table 1: Inhibition Efficiency and Corrosion Rate 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 Tetradecylamine from weight loss measurements at 298, 308 and 318K for 24.0 hours of immersion time

According to Solomon *et al.* ^[31] and others ^[32] the decreases in corrosion inhibition efficiency with increase in temperature indicates that physisorption of Tetradecylamine compounds on the mild steel surface ^[33], while the reverse behavior suggests the molecules absorbed surface by the action of chemisorption. Hence result from table1 shows the physical adsorption of inhibitor. This behavior due to the desorption of Tetradecylamine from the mild steel surface at higher temperature.

The maximum corrosion inhibition efficiency was found to be 93.10% and maximum reduction of corrosion rates 2.21 mmy⁻¹ at 298K temperature for 24 hrs at higher concentration.

Adsorption isotherm

The adsorption isotherm study is must be essential to describe the adsorption behaviour of inhibitor on to the metal surface ^[34]. Different type isotherms can be used to determine the adsorption behavior of inhibitor on the mild steel surface i.e., Langmuir, Frumkin, Hill de Boer, Flory-Huggins, Parsons– Temkin, Dhar–Flory-Huggins, and Bockris–Swinkels. Temkin, etc. But in the present work the Langmuir isotherm is the best fit for the experimental data.

A linear relationship was obtained on plotting C/ θ versus C with strong correlation coefficient nearly equal to 0.99 is shown in Figure 3 with a slope of ideally unity, which

suggesting that the adsorption of Tetradecylamine on mild steel surface follow Langmuir adsorption isotherm. Figure 3 shows the dependence of the fraction of the surface covered $C_{inh/\theta}$ as a function of the concentration of corrosion inhibitor.

The deviation of the slope from unity is often interpreted as a sign that the adsorbing species occupy more or less a typical adsorption site at the metal/solution interface ^[35, 36].



Fig 3: Langmuir adsorption isotherm plot for the mild steel in 1.0M HCl solution with different concentration of Tetradecylamine at different temperatures for immersion time 24.0 hours.

The Langmuir adsorption isotherm gives a relation between degree of surface coverage (θ) which determined from the weight loss measurement and the corrosion inhibitor concentration. The Langmuir adsorption isotherm is presented by following equation:

Where, C_{inh} is the concentration of corrosion inhibitor, θ is the degree of surface coverage and K_{ads} is the the equilibrium constant for adsorption process it could be calculated from the intercepts of the straight lines on the $C_{inh/\theta}$ axis, which also related to the standard free adsorption energy (ΔG^{0}_{ads}) which are obtained by using following Equation:

Where, R is the universal gas constant (8.314 J mol⁻¹ K⁻¹), K_{ads} adsorption constant T is absolute temperature and 55.5 is the concentration of water ^[37, 38] in solution (mol/lit.).

The linear correlation coefficient adsorption parameters K_{ads} and ΔG^0_{ads} values at different temperature for 24hrs immersion time periods are illustrate in Tables 2.

Table 2: Thermodynamic adsorption parameters for mild steel in 1.0 M HCl in the presence of various concentrations of Tetradecylamine at different temperatures for 24 hrs time periods.

Corrosion Inhibitor Name	Immersion Time (hrs)	Temperature (Kelvin)	Log 55.5 Kads	-ΔG ⁰ ads (kJ mol ⁻¹)
	24.0	298	4.388	-25.041
Tetradecylamine		308	4.382	-25.844
		318	4.185	-25.483

From the Table 2 it can be seen that on increasing the the temperature the K_{ads} value gradually decreases and the The ΔG^{o}_{ads} values obtained for Tetradecylamine on the mild steel surface in 1.0 M HCl are between -25.041 to -25.844 kJ mol⁻¹. It is reported by several authors that if the value of ΔG^o_{ads} is less than -20 kJ mol⁻, they are consistent with the electrostatic interaction between the charged molecules and the charged metal called physical adsorption (physisorption), and while those that are -40 kJ/mol or 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) ^[39, 40]. Hence these calculated value of ΔG^o_{ads} indicates that the adsorption process of corrosion inhibitor on metal surface involving the mixed physical and chemical adsorption of the inhibitor^[41]. The negative value of ΔG^o_{ads} also indicate spontaneous

adsorption process of corrosion inhibitor molecule on the metal surface and more negative value of reflects the increasing adsorption capability ^[42-44].

Electrochemical impedance measurement

Electrochemical impedance spectroscopy (EIS) is a wellestablished and powerful tool in the study of corrosion process of mild steel in acidic medium. The corrosion behavior of the mild steel in 1.0 M HCl solution in presence and absence of Tetradecylamine has been investigated by using electrochemical impedance measurement at 298K temperature after an exposure periods of 30 min. The impedance spectra are presented as Nyquist plot are shown in Figure 4.



Fig 4: Nyquist Plots for Mild Steel Samples in 1.0 M HCl Solution with Presence and Absence of Different Concentrations of Tetradecylamine as Corrosion Inhibitors at 298K Temperatures.

It is conclude from the Figure 4 that impedance of mild steel was significantly changed after the increasing the concentration of inhibitor in corrosive solution. It is also found that Nyquist plots are not perfectly semicircles in nature due to frequency dispersion and this behavior attributed may be of non homogeneity or roughness of metal steel surface (electrode surface) ^[45-50]. It can be also seen from the diagram that diameter of semicircle increases with increasing concentration of inhibitor in 1.0 M HCl solution.

The bode plot relation for log Z vs. frequency obtained for the mild steel in the absence and presence different concentrations of Tetradecylamine in 1.0 M HCl solution at 298K temperature are shown in Figure 5.



Fig 5: Bode digram for Mild Steel Samples in 1.0 M HCl Solution with Presence and Absence of Different Concentrations of Tetradecylamine as Corrosion Inhibitor at 298K Temperature.

Figures show that, in the higher frequency region, the Log Z tends to be very low. It also shows that the value of impedance increases on increasing the concentration of corrosion inhibitor in 1.0 M HCl solution.

The various calculated experimental values obtained from impedance measurement i.e charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}) and percentage corrosion inhibition efficiency (IE %) are illustrated in Table 3.

Table 3: Impedance parameters and their corresponding inhibition efficiency in presence and absence of different concentrations of Tetradecylamine at 298K temperature.

Corrosion Inhibitor Name	orrosion Inhibitor Temperature Name		$R_{ct}(\Omega.cm^2)$	Cdl(µF.cm ⁻²)	Percentage Corrosion Inhibition Efficiencies (%I.E _R)	
	298K	0	48.64	608.50	-	
		200	149.51	197.96	67.46	
Tatradaaulamina		400	197.17	150.11	75.33	
Tetradecylamine		600	248.63	119.04	80.43	
		800	446.01	66.36	89.09	
		1000	514.16	57.56	90.53	

Result from the Table3 showed that with increasing the concentration of inhibitor, the charge transfer resistance (R_{ct})value increased and the double layer capacitance (C_{dl}) value decreased which indicates that there is adsorption of inhibitor compound on the metal/solution interface surface leading to the formation of thin layer film ^[51]. The decreased in double layer capacitance value with increasing concentration indicated that there is formation of protective layer on the metal surface ^[52].

The charge transfer resistance value calculated from the difference in impedance (Z_{real}) value at lower to higher frequencies ^[53]. The value of double layer capacitance are calculated from following equation ^[54].

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}}.$$
 (6)

Where, f_{max} is the frequency value at the top of semicircle and it is mid way of Z _{real} & also at which Z _{image}. Component is maximal.

The inhibition efficiency was evaluated by charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) values of

the impedance. In present result the efficiency calculated from R_{ct} by using following equation $^{\left[55\right] }:$

$$I.E.\% = \frac{R_{ct} (inh) - R_{ct}}{R_{ct} (inh)} \ge 100....(7)$$

Where R_{ct} and $R_{ct (inh)}$ are the charge transfer resistance in absence and presence of different concentrations of inhibitor. The maximum percentage corrosion inhibition efficiency (IE_R%) was found to be 90.53% at 1000 ppm concentrations for Tetradecylamine.

Potentiodynamic polarization measurement

Potentiodynamic polarization curves of mild steel in presence and absence of different concentration Tetradecylamine in 1.0 M HCl solution at room temperature are shown in Figure 6. Figure show that inhibitor absorbed on mild steel surface more strong as concentration increased and also block the active sites of surface, hence suppressed both cathodic and anodic reaction. It also suggested that inhibitor compound decrease in the anodic dissolution and slow down the hydrogen evolution process at cathode, indicates that Tetradecylamine perform act as both anodic and cathodic inhibition effects [56]. Therefore,

Tetradecylamine compound act as mixed type inhibitor [57-59].



Fig 6: Potentiodynamic Polarization Curves for the Corrosion of Mild Steel in 1.0 M HCl Solution with and without different Concentrations of Tetradecylamine as Corrosion Inhibitor at 298K Temperature.

The various electrochemical corrosion parameters obtained by extraplotation of Tafel line to the corrosion potential $^{[60]}$ are corrosion potential (-E_{corr}), corrosion current density (I_{corr}),

cathodic (β_c) and anodic (β_a) Tafel constant are summarized in Table 4.

 Table 4: Potentiodynamic polarization measurements for mild steel samples in 1.0 M HCl with and without different concentration of Tetradecylamine at 298K Temperature.

Corrosion	Concentrations	ß (mVd-1)	β (mVd ⁻¹)	Rp	-Ecorr	icorr	Percentage Corrosion
Inhibitor Name	(ppm)	pa(mvu)	pc(mvu)	$(\Omega.cm^2)$	(mV vs.SCE)	(µA cm ⁻²)	Inhibition Efficiencies (%I.E _P)
Tetradecyl Amine (298K)	0	511	1755	34.71	-498	4.98	-
	200	620	1264	110.84	-525	1.63	67.26
	400	545	1485	124.23	-501	1.39	71.91
	600	468	1303	137.10	-528	1.09	77.97
	800	424	1207	159.66	-540	0.85	82.93
	1000	408	1237	194.60	-544	0.68	86.34

Result from the Table 4 Shows that the anodic Tafel constant (β_a) and cathodic Tafel constant (β_c) values were changed with increasing the concentration of inhibitor, this indicates that the inhibitor affected both the anodic and cathodic reactions ^[61]. The value corrosion current density (I_{corr}) calculated by using equation (8) (Stern-Geary equation).

$$I_{corr} = \frac{\beta_a X \beta_c}{2.303 (\beta_a + \beta_c)} X \frac{1}{R_p} \dots (8)$$

Where, β_a and β_c are the cathodic and anodic Tafel constant, R_p is the polarization resistance, respectively.

The inhibition efficiency (IE $_{p}$ %) values were calculated from the following equation ^[62].

IE p% =
$$(1 - \frac{I_{corr^4}}{I_{corr^0}}) \times 100$$
(9)

Where, I_{corr^0} and I_{corr^i} are the corrosion current density without and with different concentrations of inhibitor, respectively. It is also clear from table that the corrosion current density I_{corr} value decreased and inhibition efficiency values increased with increasing inhibitor concentration in 1.0 M HCl solution. This is due to there is formation of protective layer on the mild steel surface which protect against corrosion process.

Generally, if the change of corrosion potential $\mbox{-}E_{\rm corr}$ is least than 85 mV with respect

-Ecorr of unhibited or blank solution, then the corrosion inhibitor can be act as mixed type inhibitor ^[63, 64] but if the value of change in corrosion potential - E_{corr} is higher than 85 mV with respect to unhibited solution, the inhibitor can be seen as anodic or cathodic type corrosion inhibitor. In the present work, the maximum shift of corrosion potential - E_{corr} is 46 mV, suggesting that tetradecylamine act as mixed type inhibitor for mild steel in 1.0 M HCl solution.

The maximum percentage inhibition efficiency value for Electrochemical impedance and polarization are 90.53 and 86.34 %, respectively. But these values are lower than the IE % obtained by the weight loss method 93.10% for Tetradecylamine. This discrepancy may be explained by the fact that, in electrochemical processes, the instantaneous corrosion current is measured. However, in the case of the weight loss method, IE % is calculated after a long time

Surface Study

Scanning electron microscope

SEM image for mild steel in 1.0 M HCl solution with presence and absence of 1000ppm of tetradecylamine at different temperature for 24 hrs are shown in figure 7 (a, b).



Fig 7: SEM images of mild steel after 24 hrs immersion in (a) Blank HCl solution (b) with 1000 ppm concentration of Tetradecylamine.

From the fig it can be clearly seen that when mild steel sample dipped in to 1.0 M solution in absence of inhibitor (Figure 7a) the surface was found to be corroded and surface is badly damaged due to metal dissolution. In presence of 1000 ppm of inhibitor the sample was found smooth surface (Figure 7b) it is attributed due to adsorption of inhibitor on metal surface which protect against corrosion.

Conclusion

- 1. Tetradecylamine act as a good corrosion inhibitor for the corrosion of mild steel in 1.0 M HCl solution at different temperature for 24.0 hrs immersion time period.
- 2. The inhibition efficiency value decreased with temperature but increased with concentration of inhibitor.
- 3. Tetradecylamine follow the Langmuir adsorption isotherm and adsorption is spontaneous process.
- 4. EIS measurement shows that there is decreased in Cdl value and increased in Rct value with increasing the inhibitor concentration.
- 5. Potentiodynamic polarization measurement reveals that Tetradecylamine belonged to the mixed type inhibitor.
- 6. The surface analysis through SEM (Scanning Electron Microscopy) images indicated that Tetradecylamine form a productive film on the mild steel surface and thereby reduces the corrosion rate

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