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Corrosion inhibition of mild steel by using Hexylamine as corrosion inhibitor in acidic medium

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ABSTRACT

The corrosion behavior of mild steel in 1.0 M HCl was carried out at $25.0^{\circ}C$, $35.0^{\circ}C$ and $45.0^{\circ}C$ using various concentrations of hexylamine as corrosion inhibitor by weight loss technique and other different techniques i.e. potentiodynamic polarization, Electrochemical impedance spectroscopy (EIS) at $25.0^{\circ}C$. Trinocular metallurgical research microscope was used for surface study. From the weight loss measurement the maximum percentage corrosion inhibition efficiency 65.66% was observed for 800ppm concentration of hexylamine at $25.0^{\circ}C$. By electrochemical impedance study it observed that charge transfer resistance (R_{ct}) value increases and double layer capacitance (C_{dl}) value decreases with increasing the hexylamine concentration hence inhibition efficiency increases. By potentiodynamic polarization study it was observed that hexylamine behave as mixed type corrosion inhibitor. From the surface study it can be seen that on increasing the inhibitor concentration there is formation of protective layer on the surface of mild steel to protect the surface against corrosion and surface become smooth and more clean & clear. By all above techniques study shows that hexylamine is a good corrosion inhibitor for mild steel in 1.0 M HCl.

Keywords: Mild steel, HCl, Hexylamine, Weight loss, potentiodynamic polarization, electrochemical impedance and metallurgical research microscopy.

INTRODUCTION

Mild steel has been widely used in a many industries as construction materials due to its excellent mechanical properties, easily availability and low cost [1]. But there is a drawback behind the deterioration of valuable properties of mild steel is corrosion which react either by chemical or electrochemical with its environment and also due to the attack by acidic solution which are commonly used in industries processes such as acidic pickling, cleaning and descaling etc [2, 3]. There are different methods used to protect the metal against corrosion in acidic medium. Among them the use of corrosion inhibitors, even in small quantity of must play an effective role to protect the metal against corrosion and cancel the reaction on metal surface [4,5]. The use of inhibitor is very simple application and less expensive. Several types of chemical inhibitors are commonly used but among them, the applications of organic corrosion inhibitor are most effective for metal against corrosion. The effectiveness of these inhibitors are due to presence of heteroatom with high electron density such as phosphorus, sulphur, nitrogen and oxygen having multiple bonds present which enhance the adsorption capacity of inhibitor on the surface of metal hence considered as best adsorption inhibitor [6,7]. Importance of these compounds also depends upon their molecular structure, size, molecular mass and their adsorptive tendencies. The first stage of corrosion inhibitor

action mechanism is adsorption of corrosion inhibitor on metal surface [8, 9]. The adsorption property of different substances also play an important role to form a protective layer on metal surface, reduce the corrosion rates and promoted as corrosion inhibitor for metal in different aggressive medium [10-20]. Moreover, sulfur containing compounds are preferred for H_2SO_4 medium while those containing nitrogen act more effectively in HCl medium [21-23]. Adsorptions are of two type physical and chemical adsorption. Physical adsorption is due to electrostatic attractive force between inhibitor and metal surface. Chemical adsorption is due to interaction between unshared electron pair and metal surface [24-27]. Mainly inhibitors are divided into three types according to electrode reaction in which they influence i.e. cathodic, anodic and mixed type inhibitors [28]. In the present study our main object to study corrosion inhibition property of hexylamine for mild steel in acidic medium at different temperature. Hexylamine selected as a corrosion inhibitor due to the presence of hetero atom and pi electron in structure which are highly responsible for adsorption on surface of metal and protect against corrosion. Various corrosion parameters have been calculated using various techniques i.e. weight loss measurement, electrochemical impedance spectroscopy.

MATERIALS AND METHODS

Experimental detail:

Inhibitor: Hexylamine purchased from Sigma-Aldrich (99%) was used for study. The molecular mass and molecular formula is $101.19 \& C_6 H_{15} N$ respectively.



Structure of Hexylamine

Material:

Mild steel specimen used for all experiments having following composition C 0.054, Mn 0.26, Cr 0.056, Cu 0.010, Ti 0.002, S 0.017, P 0.019, Mo 0.018, Si 0.015, Ni 0.009 and remained iron. First of all the specimen were cut into 1x3 cm dimensions and then polished with different grade (100-1000) emery papers. Then after specimens were washed with double distilled water, degreased with acetone, dried with help of hot air dryer and then used for experiment study.

Solution preparation:

The acidic solution of 1.0 M HCl has prepared by dilution of analytical grade 37% with double distilled water. The inhibitor solution which have been used for experiment purposes were made up by dilution of 0.5mL hexylamine with 500mL of 1.0 M HCl solution to make 1000ppm solution (stock solution). The desired concentrations of hexylamine were made from stock solution taken in range 0ppm to 800ppm.

Weight loss measurement:

The weight loss study has been conducted in 1.0 M HCl solution. Mild steel samples were cut in to 1×3 cm² then abraded with emery paper of different grade (100-1000) and rinsed with double distilled water, cleaned with acetone and finally dried between filter paper then weighted. The weight loss study carried out at on previously weight sample in 30mL of HCl solution in presence and absence of various concentration of inhibitor for 24 hrs in test solution and then after sample put outside the test solution after completion of time period and cleaned with double distilled water followed by acetone, dried and then again weighted. This weight loss study was performed in triplicate and then average weight loss was recorded by this weight loss study. We can calculate corrosion inhibition efficiency surface coverage by following equation.

$\eta_w =$	$\frac{w_o - w_i}{w_o} \ge 100$	(1)
$\boldsymbol{\theta} =$	$\frac{w_o - w_i}{w_o}$	(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:-

CR (mmy⁻¹) = $\frac{87.6 \times W}{AtD}$(3)

Where w is weight loss of mild steel in mg, A is area of sample (cm²), t is exposure time (h) and D is density of mild steel.

Electrochemical measurements:

Mild steel samples of $1x5 \text{ cm}^2$ dimension are used for electrochemical study. The samples polished with (100-1000) grades emery papers, cleaned with double distilled water followed by acetone & then used for experiment study. The sample surface area was taken in range $1.0 \times 1.0 \text{ cm}^2$. Electrochemical impedance and potentiodynamic polarization were carried out by same instrument named as AUTOLAB Salatron model 1280B Potentiostat. Electrochemical process were carried out with help of three electrodes, one of them is mild steel as working electrode, platinum as counter electrode and saturated calomel as reference electrode in 50 mL beaker. Before start each experiments first of all the electrodes were immersed in test solution at open circuit potential (OCP) for 30 min. at 25.0° C temperature to attained a stabilized value of OCP [29] and then after proceeding the measurement.

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 the corrosion potential (- E_{corr}). The experiments were performed with different concentrations of inhibitor which were previously used in weight loss experiments. All impedance 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.

Potentiodynamic Polarization:

The electrochemical corrosion behaviour of mild steel sample in presence and absence of different concentrations of inhibitor in 1.0 M HCl solution at 25.0° C was carried out by the potentiodynamic polarization curves. The polarization study were taken after impedance measurement at same cell setup and potential range were taken 200 mV to +200 mV with respect to open circuit potential, at a scan rate of 1.0 mV/ sec. From the polarization curves, the corrosion parameters such as corrosion potential (-E_{corr}), corrosion current density (I_{corr}), cathodic (β_c) and anodic (β_a) Tafel constant were obtained and also a percentage inhibition value calculated of different concentrations of inhibitor.

Morphological study:

For the surface morphology analysis of mild steel samples which were used in weight loss study at 25.0^oC temperature for 24 hrs carried out by using trinocular inverted metallurgical research microscope. The morphological study gives information about the number of pores, size of pores, percentage porosity and area covered by the pores on the surface of mild steel. The different values of Percentage porosity (PP), Total object (TO), Maximum perimeter and Maximum area of corroded surface at different concentrations of inhibitor was also calculated using the Metallurgical software provided with instrument.

RESULTS AND DISSCUSION

Weight loss study:

Weight loss measurements carried out for mild steel at different temperature ranges $(25.0^{\circ}C, 35.0^{\circ}C, 45.0^{\circ}C)$ with different concentrations of inhibitor for 24 hrs immersion time period. The weight loss parameters such as corrosion rates, surface coverage, weight losses in mg & percentage inhibition efficiency obtained from calculation are listed in Table1. From the table results indicates that when the concentration of inhibitor in 1.0 M HCl solution is increased the inhibition efficiency increases and corrosion rates decreases at particular temperature but on the temperature scale the results is reversed . On increasing the temperature from $25.0^{\circ}C$ to $45.0^{\circ}C$ the inhibition efficiency is decreased but corrosion rates increased this behavior due to the adsorption strength of inhibitor on the metal surface is weaken and also desorption of inhibitor from the surface at higher temperature. All results show that the type adsorption is called physical adsorption (exothermic in nature) because at lower temperature the adsorption is high but at higher temperature adsorption is low. The variation of temperature and concentration vs. inhibition efficiency and corrosion rate are shown in Fig 1 & 2. The maximum inhibition efficiency was found 65.66 for 800ppm at $25.0^{\circ}C$ temperature.



Fig1: Variation of inhibition efficiency and corrosion rate with different temperature from 25.0°C to 45.0°C of hexylamine different concentration in 1.0 M HCl at 24hr



Fig2: Variation of inhibition efficiency and corrosion rate with different concentration from 0ppm to 800ppm of hexylamine at different temperature (25.0°C, 35.0°C & 45.0°C) for 24 hrs

Electrochemical impedance:

The corrosion behavior of mild steel in 1.0 M HCl solution with and without different concentration of hexylamine was investigated by electrochemical impedance spectroscopy techniques at 25.0° C temperature after immersion time 30 min to stabilized OCP value for each studied sample. The results presented in the form of Nyquist plot that are shown in fig 3. It can be concluded from fig. that the impedance shape of mild steel was significantly changed after the addition of hexylamine to the 1.0M HCl solutions. The plots are semicircle in nature and diameter of semicircle increased with increasing the concentration of inhibitor that indicates impedance of mild steel increases with inhibitor concentration and hence protected the metal surface against corrosion more effectively at higher concentration. It is also observed that the shape of Nyquist plots were not perfectly semicircle. This effect due to frequency dispersion and behavior can be attributed to in-homogeneities and roughness of the electrode surface [30]. Clearly, the impedance diagrams in which semicircles is obtained indicates that the corrosion control on mild steel surface by charge transfer process [31]. All the results were analyzed by the using equivalent circuit diagram which is combinations of charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) with the solution resistance (R_s) are shown in Fig 4.The corrosion impedance parameters such as charge transfer resistance (R_{cl}), double layer capacitance (C_{dl}) and percentage inhibition efficiency values were listed in Table2. The R_{ct} values for different concentration were obtained from the difference in impedance (Z_{real}) at higher frequency to lower frequency value.



Fig3: Nyquist plot for mild steel in 1.0 M HCl in various concentration of hexylamine as corrosion inhibitior



Fig4: Equivalent circuit diagrams for the impedance spectra

From inspection of the table it can be seen that R_{ct} value increased and C_{dl} value decreased with increasing the concentration of inhibitor. This effect due to the there is formation of surface protective layer by the inhibitor at

higher concentration which led to increases in the %IE. The maximum inhibition efficiency value are found 66.24 at 800ppm. The decrease in C_{dl} values show a result of decrease in local dielectric constant and/or increase in the thickness of electrical double layer hence suggest that hexylamine function by adsorption at the metal solution/ interface [32, 33]. Hence all these results show that hexylamine as good corrosion inhibitor for mild steel in acidic medium. The C_{dl} value can be calculated by following equation:

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

Where, f_{max} is the mid way of Z_{real} axis and also at which Z_{image} component is maximal. The percentage inhibition efficiency is calculated using R_{ct} values by following equation:

$$E_{R} \% = \frac{R_{ct\,(inh)} - R_{ct}^{0}}{R_{ct\,(inh)}} \times 100 \dots (5)$$

Where, $R_{ct (inh)}$ and R_{ct}^0 are charge transfer resistance with presence and absence of different concentration inhibitor. Fig 5 & 6 shows the variation of charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) with different concentrations of inhibitor which also indicates that charge transfer resistance increases and double layer capacitance decreases with increasing the concentration of inhibitor.



Fig5: Variation of R_{ct} with different concentration of hexylamine



Fig6: Variation of C_{dl} with different concentration of hexylamine

Potentiodynamic polarization measurement:

The Tafel polarization curve obtained from polarization measurements for mild steel in 1.0 M HCl solution containing different concentration of inhibitor are shown in Fig7. By inspection the fig it can be seen that presence of inhibitor shifted the both cathodic & anodic branches of curve to lower value of current density same extent indicates the compound performed as both type cathodic and mixed type inhibitor. The corrosion parameters of polarization such as anodic (β_a) & cathodic (β_c) Tafel constant, Corrosion potential (-E_{corr}), Corrosion current density (I_{corr}), Polarization resistance (R_p) & Percentage inhibition efficiency at different concentrations were obtained by polarization curve are listed in Table3. From the table result shows that corrosion current density (I_{corr}) values decreases but % IE increases with increasing inhibitor concentration in 1.0 M HCl. This behavior due to the on increasing concentration the inhibitor adsorb more strongly on the metal surface and reduce the anodic dissolution and retards the hydrogen evolution at cathode and also reduce cathodic and anodic reaction on the mild steel surface. The maximum calculated IE% based on I_{corr} value found at 800ppm concentration is 60.85 and maximum reduction of I_{corr} value is 1.28 at same concentration. It can be also seen from the table that anodic Tafel constant value is higher than cathodic Tafel value that indicates the hexylamine more effective as anodic type inhibitor. The values of corrosion current density (I_{corr}) calculated by using following equation (Stern-Geary equation):

Where, $\beta_a \& \beta_c$ are the anodic, cathodic Tafel constant and R_p is the polarization resistance. According to Riggs and others [34, 35], if the value of displacement in corrosion potential (- E_{corr}) is higher than 85 mV with respect to corrosion potential of blank solution or without inhibitor, the inhibitor can be seen as anodic or cathodic type inhibitor, and if value of displacement in corrosion potential (- E_{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 47mV, which show that hexylamine performed as a mixed type inhibitor. The percentage corrosion inhibition efficiency (PCIE %) for each concentrations of inhibitor calculated from using I_{corr} values. The following equation shows the relationship between Icorr and percentage inhibition efficiency (Ep).

$$Ep \% = \frac{I_{corr^0} - I_{corr^i}}{I_{corr^0}} \times 100....(7)$$

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



Fig7: Tafel polarization curve for mild steel in 1.0 M HCl with presence or absence different concentration of hexylamine at 25.0°C

The Inhibition efficiencies obtained from different techniques i.e. weight loss (ηw), potentiodynamic polarization curves (Ep) and electrochemical impedance (E_R) are in good reasonably agreement.

Surface analysis:

For the surface study of mild steel samples used in weight loss study at 25.0^{9} C for 24 hrs were carried out by metallurgical research microscope in presence and absence of various concentrations of hexylamine as a corrosion inhibitior are shown in Fig 8 (a, b, c). Various surface analysis parameters like coating thickness, percentage porosity and total object on surface in presence and absence of inhibitior are shown in Table 4. The results from table shows that total object, percentage porosity and corrosion coating thickness decreases with increasing in concentrations of hexylamine in 1.0 M HCl solution. Here maximum perimeter and maximum area give information about size and depth of pores on the other hand side percentage porosity and total object gives idea about the roughness of the surface of mild steel [36]. It is also observed from the Figures that surface of sample becomes more clear and clean with increase in concentration of inhibitior and a smooth surface is observed this is due to the formation of protective film on mild steel which resist corrosion on surface indicating that the inhibitor protected the surface. Hence all these results show that hexylamine performed as effective corrosion inhibitor at higher concentration.



8 (c) 800 ppmCoating thicknessPorosity %Fig8: Surface analysis of mild steel coupons at different concentrations (0, 400, 800 ppm) of inhibitor

Inhibitor	Conc.(ppm)	Wt. loss (mg)	Surface coverage(θ)	CR(mmy ⁻¹)	IE%
	0	198	-	31.36	-
Hexylamine	200	121	0.38	19.16	38.89
-	400	106	0.46	16.79	46.47
$(25^{\circ}C)$	600	87	0.56	13.78	56.06
	800	68	0.65	10.77	65.66
	0	229	-	36.27	-
	200	177	0.22	28.04	22.70
(2 , 7), (),	400	148	0.35	23.44	35.37
(35°C)	600	126	0.44	19.96	44.97
	800	109	0.52	17.26	52.40
	0	324	-	51.32	-
$(45^{0}C)$	200	273	0.15	43.24	15.74
(+3 C)	400	251	0.22	39.76	22.53
	600	218	0.32	34.53	32.71
	800	197	0.39	31.20	39.19

Table1: Weight loss measurements data such as corrosion rate, surface coverage and inhibition efficiency of mild steel in 1.0 M HCl for24 hr at different temperature ranges (25.0°C, 35.0°C & 45.0°C)

Table2: Impedance parameters and corresponding inhibition efficiency of mild steel in 1.0 M HCl containing different concentrations of hexylamine at 25.0°C temperature

Inhibitor	Conc. (ppm)	$R_{ct}(\Omega.cm^2)$	C_{dl} (µF.cm ⁻²)	%I.E
	0	47.74	112.07	-
Hexylamine	200	84.92	63.00	43.78
	400	88.40	60.52	45.99
	600	117.53	45.52	59.38
	800	141.41	37.82	66.24

Table3: Potentiodynamic polarization study data and inhibition efficiency for mild steel in 1.0M HCl solution in presence and absence of various concentration of hexylamine as corrosion inhibitior at 25.0°C

Inhibitor	Conc. (ppm)	βa (mVd ⁻¹)	βc (mVd ⁻¹)	Rp ($\Omega.cm^2$)	-Ecorr (mVvs.SCE)	Icorr (µAcm ²)	%I.E
	0	589	547	37.6	-455	3.27	-
Hendensine	200	431	423	44.1	-463	2.10	35.78
Hexylamine	400	476	337	48.0	-470	1.78	45.56
	600	487	308	54.6	-476	1.50	54.12
	800	434	310	61.2	-502	1.28	60.85

Table4: Surface parameters like coating thickness, maximum area, Perimeter, total object and pore percentage of mild steel after immersion period of 24hrs at 25.0°C temperature

Conc. (ppm)	Maximum area (micron)	Maximum perimeter (micron)	Coating thickness		Total object	Pore %
			Maximum	Avg.		
			Thickness	Thickness		
Blank	830159.27	3986.20	1052.63	330.79	209	100.19
400	31765.72	2136.62	260.52	16.41	447	13.20
800	22846.26	972.26	197.36	24.95	51	3.82

CONCLUSION

Weight loss study shows that the inhibition efficiency of hexylamine increases with increasing concentration but decreases with increasing in temperature. From the electrochemical impedance measurement it can be seen that when inhibitor concentrations increasing charge transfer resistance increases and double layer capacitance decreases. Hence percentage inhibition efficiency increases. By potentiodynamic polarization it showed that hexylamine is a mixed type corrosion inhibitor. The results obtained from Metallurgical research micrograph techniques shows that corrosion inhibition by formation of protective layer on the surface of mild steel and surface become more clear with increasing concentration. All these results indicated that the investigated compound hexylamine is an effective corrosion inhibitor for mild steel in 1.0 M HCl solution.

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