

# The Study of Corrosion Inhibition Ability of Hexamine on Mild Steel in Acetic Acid Solution

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**Abstract**—The ability of Hexamethylene tetraamine (hexamine) as corrosion inhibitors for mild steel in 0.5, 1.0, 1.5 and 2.0 M acetic acid solution have been studied by polarization, impedance, weight loss and scanning electron microscopic measurements, which confirm the inhibitive action of hexamine. The studies were carried out at room temperature as well at different temperature range of 303 to 333 K. The inhibition efficiency of hexamine increases with increase of inhibitor concentration. Thermodynamic parameters governing the adsorption process like, activation energy, standard free energy of adsorption, standard enthalpy of adsorption and standard entropy of adsorption have been calculated and discussed. The adsorption of hexamine is found to obey Langmuir adsorption isotherm. The study suggests that hexamine acts as a mixed type inhibitor, predominantly cathodic in nature. Changes in impedance parameters were indicative of the adsorption of hexamine on the mild steel surface. The SEM photographs show a clearly different surface morphology in the presence of inhibitor. The results obtained by electrochemical measurements are consistent with the results of the weight loss measurements. The results obtained show that hexamine acts as an effective corrosion inhibitor for mild steel in acetic acid and inhibition efficiency reaches up to 87.25 % at a very low inhibitor concentration of 15 mM.

**Keywords**—Corrosion, Inhibitor, Mild Steel, Hexamine, Acetic acid.

## I. INTRODUCTION

Metals and alloys are the backbones of the engineering field. Industries are heavily depending on the use of metals. The metal and alloy structures can be damaged by corrosion. Corrosion of metals draws the greatest amount of attention for safety, environment, and economic reasons [1]. Since, metal have a high electric conductivity, their corrosion is usually of an electrochemical in nature.

Due to good mechanical properties and low-cost Mild steel is an important material in many industries. It is widely used as construction material for chemical reactors, heat exchanger and boiler systems, storage tanks, and oil and gas transport pipelines [2, 3]. But it is suffering from certain type of corrosion in acidic and alkaline media [4, 5]. Acetic acid is one of the reactants in the manufacturing of cellulose acetate, plastic, acetic anhydride and in textile processing. However acetic acid is weak acid, but it provides sufficient amount of protons to act as true acids towards most metals.

Several approaches have been proposed to protect the metal from corrosion, the use of inhibitor is one of the most practical method for protection of metal against corrosion in acidic media [6]. Most of the organic compounds having hetero-atoms such as N, O, S, P in their aromatic ring or long

carbon chain and having  $\pi$ -electrons were reported as the efficient corrosion inhibitors under aggressive environments [7, 8, 9]. Hexamine was reported as effective corrosion inhibitor for different metal in various acids [10, 11, 12]

In the present work the efficiency of hexamine as corrosion inhibitor for mild steel (MS) in acetic acid media is studied by weight-loss and electrochemical methods.

## II. EXPERIMENTAL

### A. Preparation of sample and solution

The test specimens of size  $4.5 \times 2.0 \times 0.2$  cm having an effective area of  $0.2097 \text{ dm}^2$  were prepared from MS having chemical composition of C = 0.25%, Mn = 0.50%, Si = 0.35%, P = 0.09%, S = 0.07% and Fe = 98.74%. The test specimens were cleaned by washing with distilled water, degreased by acetone and again washed with doubled distilled water, finally dried and weighted by using electronic balance. Aqueous solution of acetic acid having concentration of 0.5, 1.0, 1.5 and 2.0 M was used as corrosive media, prepared by diluting acetic acid of analytical grade in double distilled water.

### B. Weight-loss measurement

For weight-loss measurement, the MS specimens were each completely suspended in 230 ml of 0.5, 1.0, 1.5 and 2.0 M acetic acid solution in beaker of volume 250ml, with and without hexamine as inhibitor having concentration of 1, 5, 10 and 15 mM at room temperature of 303 K, for 24 hour.

### C. Surface cleaning of test specimens

After exposure period, to remove the rust from MS test specimens were cleaned by Clark's solution [13, 14], followed by distilled water and after that washed with acetone and then dried with air dryer. From the weight loss data, corrosion rate (CR) was calculated.

### D. Temperature effect

To study the effect of temperature on corrosion of MS, the test specimens were completely immersed in 230 ml of 1.0 M acetic acid solution without and with different concentrations of hexamine at 313, 323 and 333 K for 2 hour. After exposure period, the test specimens were weighed to determine corrosion rate. From the weight-loss data Inhibition efficiency (IE), Energy of activation ( $E_a$ ), Heat of adsorption ( $Q_{ads}$ ), Free energy of adsorption ( $\Delta G^{\circ}_{ads}$ ), Enthalpy of adsorption ( $\Delta H^{\circ}_{ads}$ ) and Entropy of adsorption ( $\Delta S^{\circ}_{ads}$ ) were calculated.

E. Electrochemical measurements

To study the mechanism of inhibition action and efficiency of hexamine on MS in acetic acid solution, Polarization study and Electrochemical Impedance Spectroscopy (EIS) measurements had been carried out without and with 15 mM of inhibitor concentration. The volume of 0.5 M acetic acid as corrosive medium was kept 200 ml. The test specimen having area of 1 cm<sup>2</sup> was immersed in corrosive environment act as working electrode. A platinum electrode used as auxiliary electrode and saturated calomel electrode (SCE) as reference electrode. All the experiments were carried out at constant temperature of 298 K. Initially open circuit potential (OCP) was measured as a function of time, and after the stabilization of the electrode at OCP, the electrochemical measurements were carried out. The change in potential was measured by potentiostat (CH instrument model- CHI608C, made in USA). EIS was carried out in frequency range 1 to 100 KHz and AC amplitude of 5.0 mV. A graph was drawn by plotting real impedance (Z') versus imaginary impedance (Z''). The typical Nyquist plots of MS was obtained in the absence and presence of the inhibitor in acetic acid solution. The value of charge transfer resistance (R<sub>ct</sub>) and double layer capacitance (C<sub>dl</sub>) were calculate.

III. RESULT AND DISCUSSION

A. Weight-loss measurement

The CR in mdd (mdd = mg dm<sup>-2</sup>d<sup>-1</sup>) of MS in acetic acid solution was calculated from the weight loss measurement as following:

$$\text{Corrosion rate (mdd)} = \frac{(\text{Weight loss in gram} \times 1000)}{\text{dm}^2 \cdot \text{day}} \tag{1}$$

IE in percentage has been calculated as follows:

$$\text{IE\%} = \frac{W_u - W_i}{W_u} \times 100 \tag{2}$$

Where,

W<sub>u</sub> = weight loss of the metal in uninhibited acid,

W<sub>i</sub> = weight loss of a metal in inhibited acid.

On addition of hexamine the corrosion rate was decreases with increase in inhibitor concentration (Table 1), and the percentage IE increase as shown in Table 2.

Table 1: Effect of acid and inhibitor concentration on corrosion rate (mdd) of mild steel in acetic acid environment

Inhibitor	Inhibitor concentration (mM)	Acid concentration			
		0.5 M	1.0 M	1.5 M	2.0 M
Blank		486.41	629.47	767.76	891.75
		CR (mdd)	CR (mdd)	CR (mdd)	CR (mdd)
Hexamine	1	123.99	162.14	214.59	247.97
	5	85.84	119.22	152.60	185.98
	10	66.76	100.14	123.99	162.14
	15	61.99	85.84	104.91	133.52

Table 2: Effect of acid and inhibitor concentration on inhibition efficiency (IE%) of hexamine on mild steel corrosion in acetic acid environment.

Inhibitor	Inhibitor concentration (mM)	Acid concentration			
		0.5 M	1.0 M	1.5 M	2.0 M
Hexamine	1	74.51	74.24	72.05	72.19
	5	82.35	81.06	80.12	79.14
	10	86.27	84.09	83.85	81.82
	15	87.25	86.36	86.33	85.03

B. Adsorption isotherm

The value of surface coverage θ (θ = Wu-Wi / Wu) were calculated directly from the IE% of the inhibitor by weight-loss method.

The plot of C/θ versus C yields a straight line with a slope nearest to one (Fig.1). This suggests that the inhibitor was adsorbed on metal surface following Langmuir adsorption isotherm [15].

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \tag{3}$$

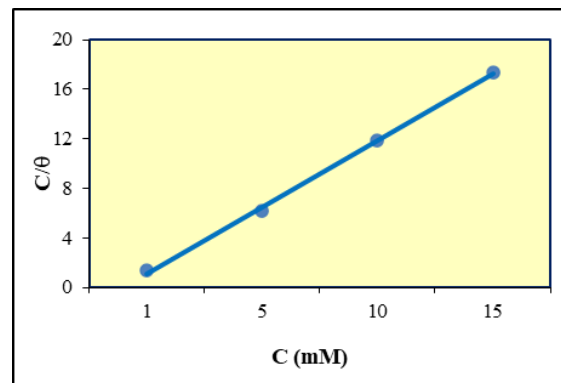


Fig. 1. Langmuir isotherm for the adsorption of hexamine on mild steel surface in 1.0 M acetic acid

C. Effect of temperature

The result was given in Table 3, shows that as the temperature increases the CR of MS is increases while IE of hexamine decreases. The IE of hexamine calculated was 80.00, 76.00 and 75.00 % correspond to 313, 323 and 333 K respectively at 15 mM inhibitor concentration. (Table 3).

Table 3: Effect of temperature on the corrosion rate (mdd), and IE% of hexamine for corrosion of mild steel in 1.0 M acetic acid

Inhibitor	Inhibitor concentration (mM)	Acid concentration					
		313		323		333	
Blank		CR (mdd)	IE (%)	CR (mdd)	IE (%)	CR (mdd)	IE (%)
		858.37	-	1430.62	-	2288.98	-
Hexamine	1	343.35	60.00	743.92	48.00	1373.39	40.00
	5	286.12	66.67	515.02	64.00	858.37	62.50
	10	228.90	73.33	400.57	72.00	686.70	70.00
	15	171.67	80.00	343.35	76.00	572.25	75.00

Decreasing tendency of the IE with increasing temperature is due to desorption of hexamine should be faster than the rate of adsorption at higher temperatures [16].

The energy of activation 'E<sub>a</sub>' was calculated from the following equation [17].

$$\log \frac{\rho_2}{\rho_1} = \frac{E_a}{2.303R} \left( \frac{1}{T_1} - \frac{1}{T_2} \right) \quad (4)$$

Where,  $\rho_1$  and  $\rho_2$  are the corrosion rate at temperature  $T_1$  and  $T_2$  K respectively and  $R$  is the gas constant ( $R = 0.008314 \text{ kJ mol}^{-1}$ ).

A comparison of  $E_a$  values in the absence and presence of inhibitor indicates that the value of  $E_a$  were higher in inhibited system than that of uninhibited system (Table 4). Higher values of  $E_a$  in inhibited systems compared to the blank suggest a physical adsorption mechanism, attributed to an appreciable decrease in the adsorption of the inhibitor on the metal surface with increase in temperature [18, 19], which is in agreement with other observation earlier published [20], according to whom for physical adsorption mechanism the  $E_a$  value should be less than  $100 \text{ kJ mol}^{-1}$ .

Table 4: Energy of activation ( $E_a$ ) and Heat of adsorption ( $Q_{ads}$ ) of various concentration of hexamine for mild steel corrosion in 1.0 M acetic acid

Inhibitor	Inhibitor concentration (mM)	Energy of activation ( $E_a$ ) ( $\text{kJ mol}^{-1}$ )		Heat of adsorption ( $Q_{ads}$ ) ( $\text{kJ mol}^{-1}$ )	
		Mean from calculat ion	From Arrhenius Plot	313-323 K	313-323 K
Hexamine	1	59.92	59.93	-40.82	-29.11
	5	47.55	47.58	-9.91	-5.44
	10	47.63	47.58	-5.61	-8.70
	15	51.98	52.20	-19.64	-4.55

Energy of activation ( $E_a$ ) has also been calculated from the slopes of  $\log \rho$  versus  $1/T$  ( $\rho =$  corrosion rate,  $T =$  absolute temperature) with the help of Arrhenius equation [21].

$$E_a = 2.303 \times R \times \text{slope} \quad (5)$$

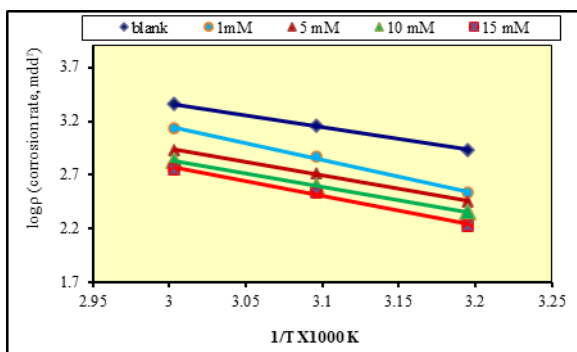


Fig. 2. Arrhenius plot for corrosion rate of mild steel in 1.0 M acetic acid in absence and presence of various concentration of hexamine.

The value of heat of adsorption ( $Q_{ads}$ ) was calculated by using the following equation (Obot et al. 2012).

$$Q_{ads} = 2.303R \left[ \log \left( \frac{\theta_2}{1-\theta_2} \right) - \log \left( \frac{\theta_1}{1-\theta_1} \right) \right] \times \left[ \frac{T_1 T_2}{T_2 - T_1} \right] \quad (6)$$

Where,  $\theta_1$  and  $\theta_2$  are the fraction of the metal surface covered by the inhibitors at temperature  $T_1$  and  $T_2$  K respectively,  $R$  is the gas constant ( $R = 0.008314 \text{ kJ mol}^{-1}$ ).

From Table 4 it was evident that  $Q_{ads}$  values were negative. The negative values of  $Q_{ads}$  show that the adsorption and hence the IE decreases with a rise in temperature [22]. The nature of adsorption process as being either a physical or chemical process may be elucidated from the magnitude of the heat of adsorption ( $Q_{ads}$ ). The value of heat of adsorption for inhibitor founds to be less than  $-40 \text{ kJ mol}^{-1}$ , indicates that the inhibitor is physically adsorbed on the mild steel surface [23]. Barrow [20] distinguished between the two types of adsorption process. He stated that the heat of adsorption for physisorption was less than  $-40 \text{ kJ mol}^{-1}$ , while it was greater than  $-80 \text{ kJ mol}^{-1}$  for chemisorption.

The values of the free energy of adsorption ( $\Delta G_{ads}^\circ$ ) were calculated with the help of the following equation [24].

$$\log B = -1.74 - \left( \frac{\Delta G_{ads}^\circ}{2.303RT} \right) \quad (7)$$

Where,  $\log C = \log \left( \frac{\theta}{1-\theta} \right) - \log B$ , and  $C$  is the inhibitor concentration and  $\log B$  is formation constant.

Table 5: Thermodynamic parameter; Free energy of adsorption ( $\Delta G_{ads}^\circ$ ) for the adsorption of hexamine on mild steel surface

Inhibitor	Inhibitor concentration (mM)	Free energy of adsorption ( $\Delta G_{ads}^\circ$ ) ( $\text{kJ mol}^{-1}$ )
Hexamine	1	-10.70
	5	-10.32
	10	-10.26
	15	-10.39

The values of  $\Delta G_{ads}^\circ$  were negative. The calculated negative value of  $\Delta G_{ads}^\circ$  indicates the strong interaction of the inhibitor molecules [25] and spontaneous adsorption of the inhibitor on the metal surface [26].

Generally, the magnitude of  $\Delta G_{ads}^\circ$  around  $-20 \text{ kJ mol}^{-1}$  or less negative can be assumed as due to an electrostatic interaction between the charged molecule of inhibitor and the charged metal surface (i.e. physisorption) [27, 28]. If the value of standard free energy of adsorption ( $\Delta G_{ads}^\circ$ ) is around  $-40 \text{ kJ mol}^{-1}$  or more negative indicates a charge sharing or transferring from organic species to the metal surface to form a coordinate type of bond [22, 29] (i.e. chemisorption). The mean values of  $\Delta G_{ads}^\circ$  for the inhibitor was less negative than  $-40 \text{ kJ mol}^{-1}$ , indicating that the inhibitor physically adsorbed on the metal surface.

The enthalpy of adsorption  $\Delta H_{ads}^\circ$  was calculated according to the Van't Hoff equation [30,31], by plotting  $\ln K_{ads}$  versus  $1/T$  with a slope of  $(-\Delta H_{ads}^\circ/RT)$  [32].

$$\ln K_{ads} = \frac{-\Delta H_{ads}^\circ}{RT} + \text{constant} \quad (8)$$

Where  $K_{ads}$  is adsorption equilibrium constant, which is obtained from intercept of the plot of  $C$  versus  $C/\theta$  for each temperature. 'R' is gas constant and 'T' is the absolute temperature.

The entropy of adsorption  $\Delta S_{ads}^\circ$  was calculated according to thermodynamic basic equation as follow [33].

$$\Delta S^{\circ}_{ads} = \Delta H^{\circ}_{ads} - \Delta G^{\circ}_{ads}/T$$

Table 6: Thermodynamic parameters: Enthalpy of adsorption ( $\Delta H^{\circ}_{ads}$ ) and Entropy of adsorption ( $\Delta S^{\circ}_{ads}$ ) for the adsorption of hexamine on mild steel surface

Inhibitor	Temperature (K)	$\Delta H^{\circ}_{ads}$ (kJ mol <sup>-1</sup> )	$\Delta S^{\circ}_{ads}$ (J mol <sup>-1</sup> K <sup>-1</sup> )
Hexamine	313	-19.90	-30.54
	323		-29.51
	333		-29.86
	15		

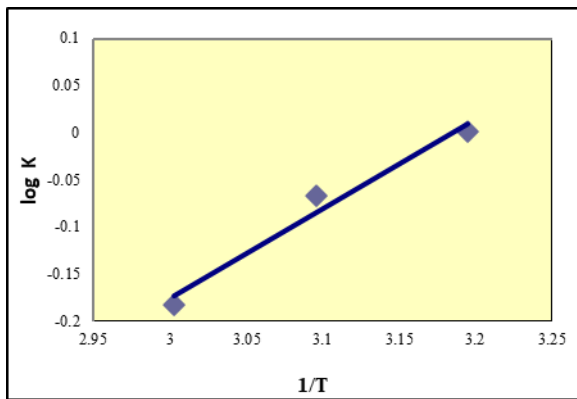


Fig. 3. Van't Hoff Plot

The negative value of  $\Delta H^{\circ}_{ads}$  suggest that the adsorption of inhibitors is an exothermic process [29, 34], which reveals that inhibition efficiency of the inhibitor decreases with raising the temperature. The behavior can be interpreted on the basis that increasing temperature leads to the increase of the desorption amount of the adsorbed inhibitor molecule from the MS surface.

The values of  $\Delta S^{\circ}_{ads}$  were large and negative that is accompanied with exothermic adsorption process. From Table-6, the negative values of  $\Delta S^{\circ}_{ads}$  describes ordered phenomenon. This order may probably be explained by the possible formation of metal complex on the MS surface [35].

#### D. Electrochemical Measurement

Various electrochemical parameters such as corrosion potential ( $E_{corr}$ ), corrosion current density ( $i_{corr}$ ) and the value of Tafel parameters ( $\beta_a$ ,  $\beta_c$ ,  $\beta_b$ ) derived from the potentiodynamic polarization measurements, were shown in Table 7.

The percentage IE can be calculated by using the following equation [36].

$$I.E.(\%) = \frac{I_{corr,u} - I_{corr,i}}{I_{corr,u}} \times 100 \quad (10)$$

Where,  $I_{corr,u}$  and  $I_{corr,i}$  are the corrosion current density for uninhibited and inhibited system respectively.

Values of  $I_{corr}$  decreases significantly in the presence of inhibitor. The value of  $I_{corr}$  for 0.5 M acetic acid is 244  $\mu A cm^{-2}$  in absence of inhibitors while in presence of inhibitors the  $I_{corr}$  value decreased to 2.76  $\mu A cm^{-2}$  (Table-7).

Table 7: Polarization measurements of mild steel in absence and presence of hexamine in 0.5 M acetic acid at 298 K

system	$E_{corr}$ (V)	Corrosion current density ( $I_{corr}$ ) ( $\mu A cm^{-2}$ )	Tafel Slope (V/decade)			Inhibition efficiency (I.E.%)
			$\beta_a$	$\beta_c$	B (V)	
Blank	-0.610	244	5.195	4.659	1.068	-
Hexamine	-0.645	2.762	5.936	5.223	1.208	98.97

The values of  $E_{corr}$  were slightly shifted in the presence of inhibitor suggesting that this inhibitor inhibit the corrosion of MS in acetic acid by controlling the redox reactions. The corrosion potential ( $E_{corr}$ ) was found to be -0.610 V (-610 mV) in uninhibited system. While for inhibited system the corrosion potential ( $E_{corr}$ ) shifted towards more negative (cathodic) region i.e. -0.645 V (Table- 7). This indicates that hexamine reduce the corrosion rate predominantly by cathodic mode.

An inhibitor is classified as cathodic or anodic type if the difference in corrosion potential is more than 0.085 V (i.e. 85 mV) with respect to the corrosion potential of uninhibited system [37]. In the present investigation, the deviation is 0.035 V is less than 0.085 V (i.e. 85 mV). Which reveals that hexamine act as mixed type inhibitor, and can retard both anodic and cathodic reaction.

Generally, the curves show polarization of both the anodes as well as the cathodes (Fig.- 4). The cathodic and anodic Tafel slopes ( $\beta_a$  and  $\beta_c$ ) changed with respect to uninhibited system.

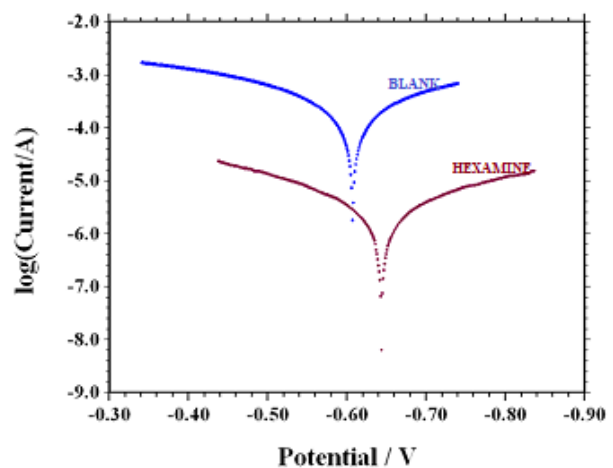


Fig. 4. Polarization curves for corrosion of mild steel in 0.5 M acetic acid in absence and in presence of 15 mM concentration of Hexamine at 298 K.

The impedance parameters derived from EIS measurement were given in Table 8. Nyquist plots for MS in 0.5 M acetic acid in the absence and presence of hexamine were shown in Fig. 5.

Table 8: EIS measurements of mild steel in absence and presence of hexamine in 0.5 M acetic acid at 298 K

System	$R_{ct}$ (ohm cm <sup>2</sup> )	$C_{dl}$ ( $\mu F cm^{-2}$ )	Inhibition efficiency (I.E. %)
Blank	56.22	145	-
Hexamine	18643.10	0.0025	99.70

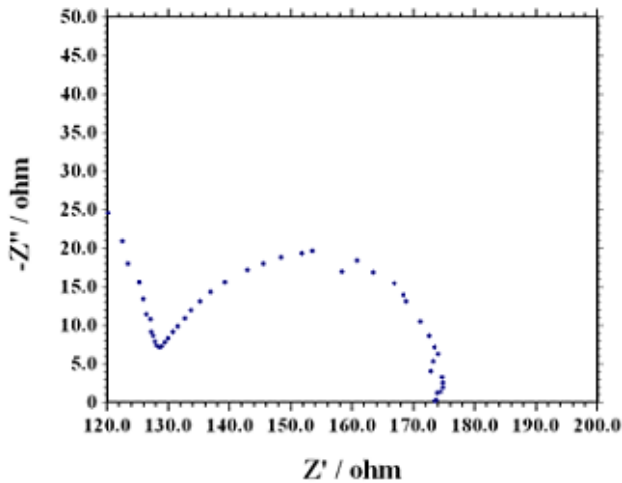


Fig. 5(a). Nyquist plot for corrosion of MS in 0.5 M acetic acid in absence of inhibitor at 298 K.

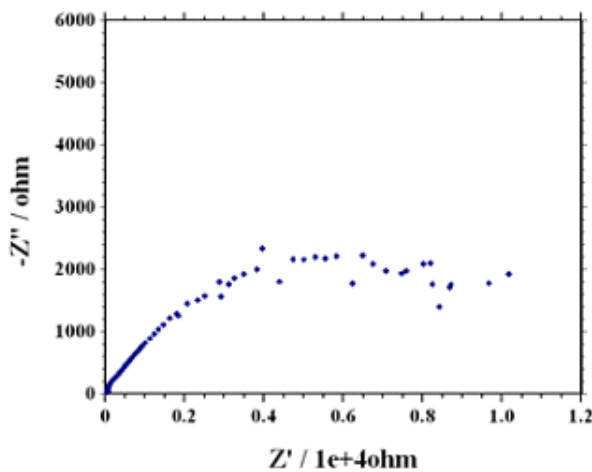


Fig. 5(b). Nyquist plot for corrosion of MS in 0.5 M acetic acid in presence of 15 mM Hexamine at 298 K.

The charge transfer resistance ( $R_{ct}$ ) were obtained from Nyquist plots by determining the difference in the values of impedance at low and high frequencies [38], and double layer capacitance ( $C_{dl}$ ) was calculated from the frequency at which the impedance imaginary component  $-Z''_{max}$  is maximum using following equation [39].

$$C_{dl} = \frac{1}{2\pi f_{(-Z''_{max})} R_{ct}} \quad (11)$$

Where,  $f_{max}$  is the frequency at which the imaginary component of the impedance is maximum.

The value of IE% of the inhibitor was calculated from the following equation [40].

$$IE(\%) = \frac{R_{ct,i} - R_{ct,u}}{R_{ct,i}} \times 100 \quad (12)$$

Where  $R_{ct,i}$  and  $R_{ct,u}$  are the charge transfer resistance values in the absence and presence of inhibitor, respectively.

From the EIS spectra, the electrochemical behavior of the system is interpreted. High  $R_{ct}$  value  $18643.10 \text{ ohm cm}^2$  was observed in the presence of hexamine of 15 Mm concentration, while the  $R_{ct}$  value was  $56.22 \text{ ohm cm}^2$  for uninhibited acid. The  $C_{dl}$  value decreases from  $145.00 \mu\text{F cm}^2$  to  $0.0025 \mu\text{F cm}^2$  in presence of hexamine (Table-8). This

decrease in  $C_{dl}$  value was due to the adsorption of inhibitor on the metal surface [41]. These results suggest that the inhibitor acts by the formation of a protective layer on the surface, which modifies the metal/solution interface [40].

The decrease in  $C_{dl}$  values can be attributed to a decrease in local dielectric constant and/or an increase in the thickness of the electric double layer which leads to an increase in the IE. Therefore, it is suggested that the inhibitors act by adsorption at the MS surface or solution interface and the change in  $C_{dl}$  values is caused by the displacement of water molecule by the adsorption of organic molecules on the metal surface, thus decreasing the extent of the metal dissolution [42, 43].

SEM images were used to investigate the surface morphology of MS before and after 24 h of immersion in the - 0.5 M acetic acid solution without and with inhibitor. Fig.- 6(a) indicates that the surface of the MS is smooth. Fig.- 6(b) shows the image of the MS sample immersed in 0.5 M acetic acid for 24 hours. It is noted that the surface of the MS was rigorously corroded because of the aggressive attack of 0.5 M acetic acid. Fig.- 6(c) shows the SEM image of the MS immersed in the acid solution in the presence of 15 mM hexamine for 24 hours. From the comparison of Fig.- 6(a), (b), (c), and the results obtained above, it is clear that a protective film has been formed by the inhibitory molecules on the surface of the MS, which leads to a significant inhibition of the corrosion of MS in 0.5 M acetic acid.

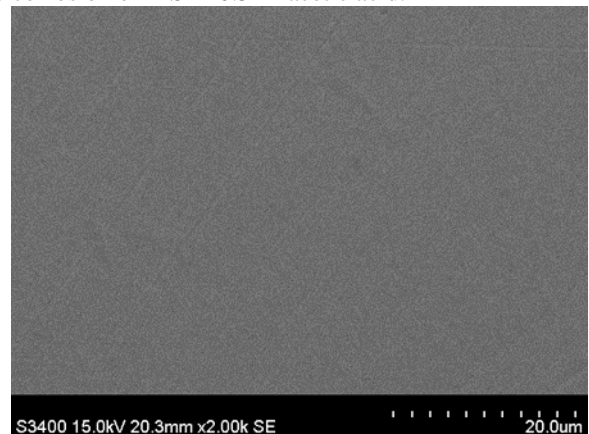


Fig. 6(a). SEM micrograph of MS (Polished) surface.

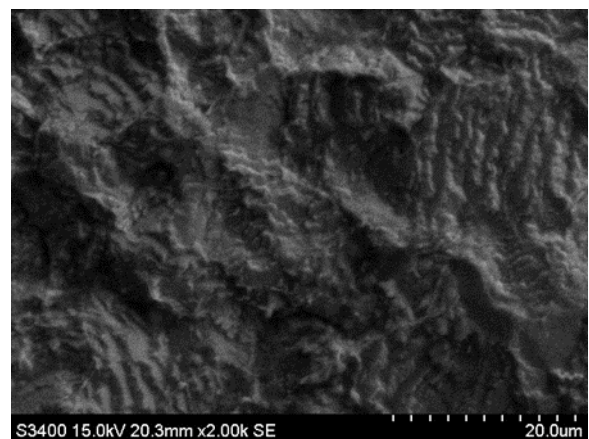


Fig. 6(b). SEM micrograph of MS immersed in 0.5 M acetic acid for 24 h without inhibitor.

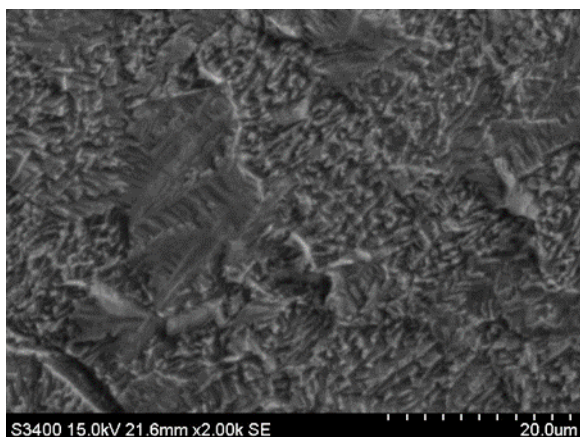
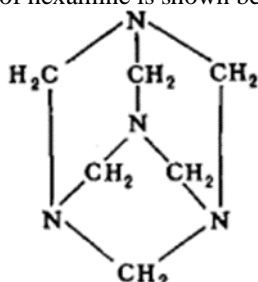


Fig. 6(c). SEM micrograph of MS immersed in 0.5 M acetic acid for 24 h in presence of 15 mM hexamine.

### E. Mechanism of inhibition

The structure of hexamine is shown below:



The four nitrogen atoms of hexamine having high electron density must have functioned as the reaction center. The inhibition action can be accounted by the interaction of lone pair of electrons at the nitrogen atom on the positively charged metal surface [44]. The presence of six ethylene groups also helps to lead to enhance electron density at the nitrogen atoms, which enhances its adsorption on the metal surface, this in turn leads to an increase in IE. Macro molecular size and higher number of 'N' atoms of hexamine [45] might have covered almost all active sites of MS. Hence the mechanism of the inhibitive action is probably due to the blocking of anodic and cathodic sites by adsorption, which enables the formation of protective insoluble film.

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