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Inhibiting Properties of Hexamine as Corrosion Inhibitor for Zinc in (HNO_3 + H_3PO_4) Binary Acid Mixture

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Abstract: The inhibition of corrosion of zinc in (HNO_3+ H_3PO_4) binary acid mixture by hexamine has been investigated by using weight loss, temperature effect and polarization techniques. As acid concentration increases corrosion rate and inhibition efficiency (I.E.) increases. As inhibitor concentration increases corrosion rate decreases while percentage of I.E. increases. As temperature increases, corrosion rate increases while percentage of I.E. decreases. Maximum I.E. of Hexamine was found up to 81.00 % at 1.0 % inhibitor concentration in (0.10 N HNO_3 + 0.10 N H_3PO_4) solution. Higher values of mean energy of activation 'Ea' indicates physical adsorption of the inhibitor on metal. Positive value of entropy of adsorption 'ΔS_a' indicates that the corrosion process is entropically favourable. The plot of log (θ/1-θ) versus log C results in a straight line suggest that the inhibitor cover both the anodic and cathodic regions through general adsorption following Langmuir isotherm. Polarization curve indicates that inhibitor act as anodic type.

Keywords: Corrosion, Zinc, (HNO_3 + H_3PO_4) acid mixture, Hexamine, Polarization.

I. INTRODUCTION

Corrosion is the deterioration of metal by chemical attack or by reaction with its environment. The problem of corrosion is of considerable importance, nowadays due to increase in uses of metals and alloys. Zinc is one of the most important non-ferrous metals, which finds extensive use in metallic coating. Nitric acid is very strong and highly corrosive mono protic acid having ability to dissolve metals, which are inert to most ordinary acids. The main use of nitric acid is for the production of fertilizers. Phosphoric acid is a major chemical product which has many important uses especially in the production of fertilizers [1,2]. One of the methods used to reduce the rate of metal corrosion is the addition of inhibitors. Aromatic, aliphatic and heterocyclic amines have been extensively investigated as corrosion inhibitors [3,4]. Many researchers [5-9] studied corrosion inhibition of zinc in different acids using various organic and green inhibitors. Hexamine was reported as effective corrosion inhibitor for different metal in various acids [10-15]. In the present work, the corrosion of zinc by (HNO_3+ H_3PO_4) binary acid mix containing hexamine as an inhibitor was evaluated by using weight loss, temperature and Electrochemical Polarization techniques.

II. EXPERIMENTAL SECTION

A. Preparation of Sample and Solution

The zinc specimens with a chemical composition of 98.50 % Zn, 0.03 % Pb,0.02 % Cd and 0.01% Fe were used in the present study. Rectangular specimens (5.5 x 2.5 x 0.2 cm) of zinc having an area of 0.2935 dm^2 were used. The specimens were cleaned by washing with distilled water, degreased by acetone and finally dried and weighted by using electronic balance. A binary acid mixture (HNO_3+ H_3PO_4) having concentration of 0.01, 0.05 and 0.10 N was used as corrosive solution prepared by diluting analytical grade purchased from Merck using double distilled water.

B. Weight loss Measurement

For weight-loss measurement, the zinc coupons were each suspended and completely immersed in 230 mL of 0.01, 0.05 and 0.10 N acid concentration in absence and presence of different concentrations of hexamine at 301± 1 K for 24 h immersion period. After the test, specimens were cleaned by 10% chromic acid solution having 0.2 % $BaCO_3$ for a period of about 2 minutes [16]. After cleaning, test specimens were washed with distilled water followed by acetone and dried with air. From the weight loss data, corrosion rate (CR) was calculated.

C. Temperature effect

To study the effect of temperature on corrosion rate of zinc, the specimens were immersed in 230 ml of (0.05 N HNO_3+ 0.05 N H_3PO_4) mix acid. Weight loss was determined at solution temperature of 313, 323 and 333 K for an immersion period of 3 h in absence and presence of hexamine at 0.1, 0.5 and 1.0 % concentration.

D. Potentiodynamic Polarization Measurement

For polarization study, zinc specimens having as area of 0.047 dm^2 were immersed to 230 mL binary acid mixture (0.01 N HNO_3 + 0.01 N H_3PO_4) in absence and presence of 1.0 % inhibitor concentration. The test cell includes the metal specimen as a working electrode, corrosive solution in which the specimen was to be tested and saturated calomel electrode (SCE) as a reference electrode as well as Platinum electrode as an auxiliary electrode. The polarization study was made by using Potentio-Galvano-Scan (Weaving PGS 81) meter. Before each electrochemical measurement, the working electrode were allowed to stand for 20 min. in test allowed to establish a steady-state open circuit potential (OCP). Polarization curves were plotted with potential against log current density (called Tafel plots). Cathodic and anodic polarization curves give cathodic and anodic Tafel lines correspondingly. The intersect point of cathodic and anodic Tafel lines gives the corrosion current (i_{corr}) and the corrosion potential (E_{corr}) [17]. Cathodic Tafel slope (βc) and anodic Tafel slope (βa) were calculated from the software installed in the instrument.

III. RESULTS AND DISCUSSION

A. Weight Loss Experiments

The corrosion rate of zinc in 0.01, 0.05 and 0.10 N (HNO_3+ H_3PO_4) binary acid solution in absence and presence of 0.1, 0.5 and 1.0 % concentration of Hexamine at 301 ± 1 K for an exposure period of 24 h was calculated from the weight loss data using the following equation:

$$\text{CR (mg/dm}^2\text{d)} = \frac{\text{Weight loss (gm)} \times 1000}{\text{(metal surface area) dm}^2 \times \text{day}} \qquad (1)$$

I.E. has been calculated as follows:

$$\text{I.E.} = \frac{Wu - Wi}{Wu} \times 100 \qquad (2)$$

Where, Wu is the weight loss of metal in uninhibited acid and W_i is the weight loss of metal in inhibited acid. The degree of surface coverage 'ɵ' for different concentration of the inhibitor in acidic media have been evaluated from weight loss experiment using the equation:

$$\theta = \frac{W_{uninh} - W_{inh}}{W_{uninh}} \qquad (3)$$

B. Effect of Acid Concentration

The rate of corrosion increases with increase in acid concentration. The corrosion rate was 146.50, 774.10 and 1417.03 $mg/dm^2.d$ corresponding to 0.01, 0.05 and 0.10 N (HNO_3+ H_3PO_4) binary acid concentrations respectively for an exposure period of 24 h at 301± 1 K as shown in Table-1.

Table 1 Effect of acid concentration on corrosion rate (CR) and I.E. of zinc at various concentrations of (HNO_3+ H_3PO_4) binary acid containing Hexamine as inhibitor for an immersion period of 24 h at 301 ± 1 K.

Inhibitior concentration (%)	Acid concentration					
	0.01 N		0.05 N		0.10 N	
	CR (mg/dm^2d)	I.E. (%)	CR (mg/dm^2d)	I.E. (%)	CR (mg/dm^2d)	I.E. (%)
Blank	146.50	-	774.10	-	1417.03	-
0.1	71.81	50.98	335.65	56.64	546.83	61.41
0.5	57.05	61.06	259.56	66.47	425.82	69.95
1.0	49.63	66.12	184.62	76.15	269.24	81.00

C. Effect of Inhibitor Concentration

At constant acid concentration, as the inhibitor concentration increases corrosion rate decreases while I.E. increases. In (0.10 N HNO_3+ 0.10 N H_3PO_4) binary acid mixture the I.E. was found to be 61.41, 69.95 and 81.00% corresponding to 0.1, 0.5 and 1.0 % inhibitor concentration respectively (Table-1) (Fig.1). At constant inhibitor concentration, as acid concentration increases I.E. increase. At 1.0 % inhibitor concentration, the I.E. of hexamine was 66.12, 76.15 and 81.00 % corresponding to 0.01, 0.05 and 0.10 N (HNO_3+ H_3PO_4) binary acid concentration respectively (Table-1).

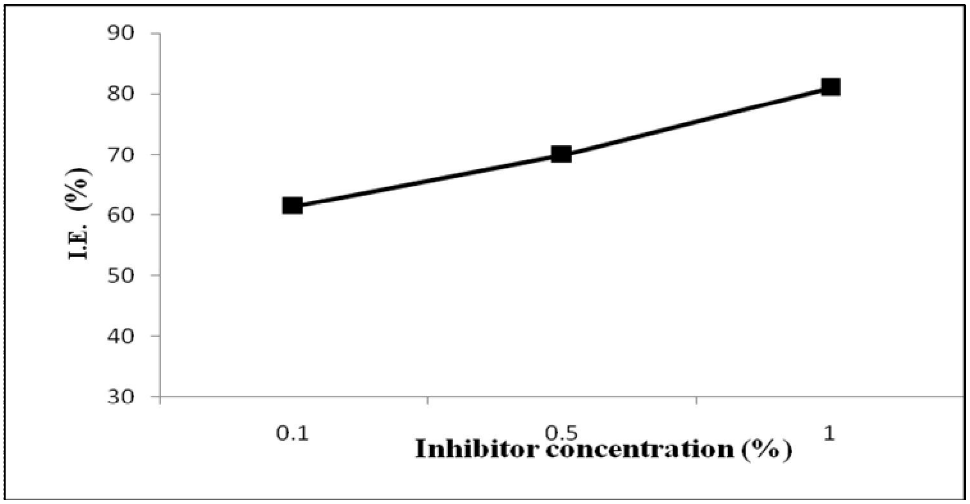

Fig.1 Effect of inhibitor concentration on I.E. of Hexamine for zinc in (0.10 N HNO_3+ 0.10 N H_3PO_4) binary acid mixture at 301 K, 24 h.

D. Temperature Effect

As the temperature increases corrosion rate increases while percentage of I.E. decreases. Corrosion rate was increase as 3952.24, 4418.32 and 4745.60 mg/dm^{2}d corresponding to 313, 323 and 333 K respectively in (0.05 N HNO_3 + 0.05 N H_3PO_4) mix acid (Table-2). Increase in corrosion rate with temperature may be due to the desorption of the adsorbed molecules inhibitor and thus exposing the fresh metal surface to further attack [18], which results in intensification of the kinetic of electrochemical reaction [19] and thus explains the higher corrosion rate at elevated temperature. In (0.05 N HNO_3+ 0.05 N H_3PO_4) at 1.0 % inhibitor concentration, the I.E. for Hexamine was 67.76, 66.41 and 57.23 % corresponding to 303, 313, 323 and 333 K respectively.

Table 2 Effect of temperature on the Corrosion rate (CR), value E_a and Q_{ads} for zinc in (0.05 N HNO_3+ 0.05 N H_3PO_4) acid at various concentrations of hexamine for an immersion period of 3 h.

Inhibitor concen-tration (%)	Temperature						Mean (E_a) From Equation (4) (kJ mol^{-1})	Qads (kJ mol^{-1})	
	313 K		323 K		333 K				
	CR (mg/ dm^{2}d)	I. E. (%)	CR (mg/ dm^{2}d)	I. E. (%)	CR (mg/ dm^{2}d)	I. E. (%)		313-323 K	323-333 K
Blank	3952.24	-	4418.32	-	4745.60	-	8.54	-	-
0.1	2391.12	39.50	3011.12	31.85	3984.16	16.06	20.90	-28.12	-79.91
0.5	1540.24	61.03	2067.36	53.21	2388.32	49.73	22.82	-26.91	-12.47
1.0	1274.24	67.76	1484.08	66.41	2030.00	57.23	23.22	-5.14	-34.93

E. Energy of Activation (E_a)

The value of 'Ea' has been calculated from the slop of log ρ versus 1/T (ρ= corrosion rate, T= absolute temperature) and also with the help of the Arrhenius equation [20].

$$\log\frac{\rho_2}{\rho_1} = \frac{Ea}{2.303R}\left[\left(\frac{1}{T_1}\right)-\left(\frac{1}{T_2}\right)\right] \qquad (4)$$

Where ρ_2 and ρ_1 are the corrosion rate at temperature T_1 and T_2 respectively. Results given in Table-2 indicates that mean ‘Ea’ values were found higher in inhibited acid (Ranging from 20.90 to 23.22 kJ mol^{-1}) than the ‘Ea’ value for uninhibited system (8.54 kJ mol^{-1}) (Table-2). The higher values of mean ‘Ea’ indicate physical adsorption of the inhibitors on metal surface [21]. The values of ‘Ea’ calculated from the slop Arrhenius plot of log p versus 1/T x 1000 (Fig.- 2) and using equation-4 were almost similar.

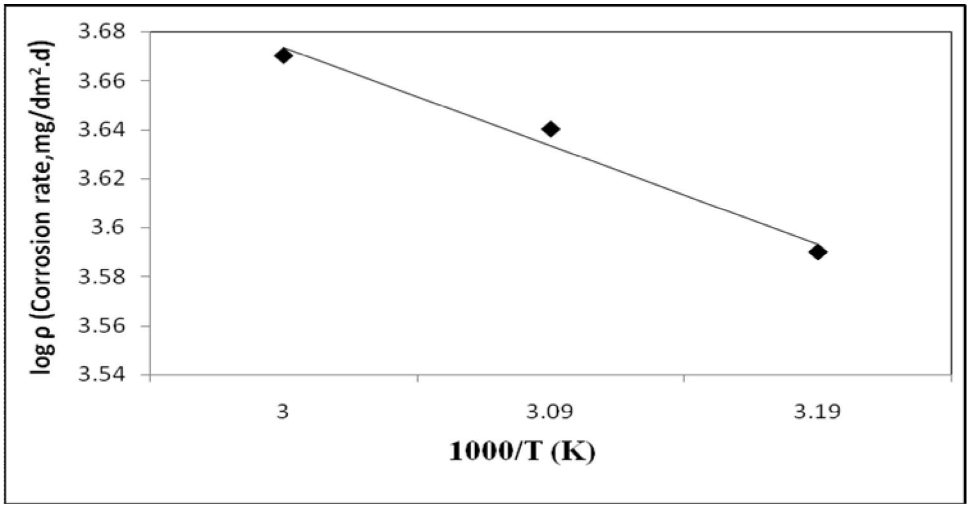

Fig. 2 Arrhenius plots for corrosion of zinc in (0.05 N HNO_3+ 0.05 N H_3PO_4) binary acid mixture in absence and presence of 0.1 % hexamine.

F. *Heat of Adsorption (Qads)*

The value of ‘Q_{ads}’ were calculated by using the following equation [22]:

$$Q_{ads} = 2.303\,R\left[\log\left(\frac{\theta_2}{1-\theta_2}\right) - \log\left(\frac{\theta_1}{1-\theta_1}\right)\right] \times \left[\frac{T_1 \times T_2}{T_2 - T_1}\right] \qquad (5)$$

Where, θ_1 and θ_2 are the fractions of the metal surface covered by the inhibitor at temperature T_1 and T_2 respectively. From Table-2, it is evident that in all cases, the Q_{ads} values were almost negative and ranging from -5.14 to -79.91 kJ mol^{-1}. The negative values of Q_{ads} shows the adsorption process and hence the I.E. decreases with rise in temperature supporting the physisorption mechanism [23].

G. *Adsorption Isotherm*

Basic information on the interaction between inhibitors and a metal surface can be provided using the adsorption isotherm [24]. The surface coverage ‘θ’ suggest that a chemical bond is formed between the metal atoms and the inhibitor molecules. The graph of log [θ / (1 - θ)] *vs.* log C for hexamine in (0.10 N HNO_3+ 0.10 N H_3PO_4) was depicted in Fig.-3, which gives straight line with slope values equal to unity indicates that the system follows Langmuir adsorption isotherm [25].

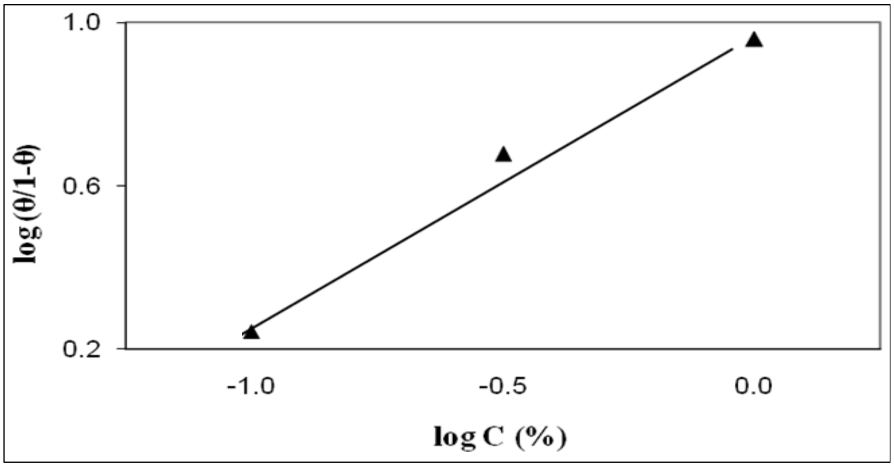

Fig. 3 Plot of log [θ / (1 - θ)] *vs.* log C for hexamine in (0.10 N HNO_3+ 0.10 N H_3PO_4) binary acid mix concentration.

H. *Free Energy of Adsorption (ΔG°a)*

The values of 'ΔGa' was calculated with the help of the following equation [26].

$$\log C = \log [(\theta / 1 - \theta)] - \log B \qquad (6)$$

Where, log B = -1.74 – (ΔGa /2.303 RT), C is the inhibitor concentration and R is the gas constant.

The value of ΔG_a^o was negative which reflects that the adsorption of the inhibitor is spontaneous process. ΔG^o ads values up to -20 kJ mol^{-1} are associated with the electrostatic interaction between any inhibitor and the charged metal electrode surface (physical adsorption), and values up to -40 kJ mol^{-1} or higher indicate charge transfer sharing between the inhibitor and the charged metal surface [27,28]. In present study, mean ΔG_a^o values were found in a range from -16.63 to -25.70 kJ mol^{-1} indicates the presence of an interaction between hexamine molecules and the zinc surface, which signifies the occurrence of physical adsorption type, along with slight chemical adsorption too. This is also supported by the fact that the I.E. of the investigated inhibitor decreases at higher temperature.

I. *Enthalpy of Adsorption (ΔH°ads)*

ΔH^o_a was calculated using the following equation:

$$\Delta H^o_a = E_a - RT \qquad (7)$$

The results revealed that ΔH_a° values was positive (26.29 kJ mol^{-1}) indicating the endothermic nature of the reaction suggests that higher temperature favours the corrosion process [29].

J. *Entropy of Adsorption (ΔS°ads)*

ΔS^o_a was calculated [30] using following equation:

$$\Delta S^o_a = \Delta H^o_a - \Delta G^o_a / T \qquad (8)$$

Value of ΔS_a° was found positive (0.15 kJ mol^{-1} K^{-1}) indicates the affinity of the adsorbent for the inhibitor and the corrosion process is entropically favourable [31].

K. *Potentiodynamic Polarization Measurements*

Potentiodynamic polarization curves of zinc in (0.01 N HNO_3+ 0.01 N H_3PO_4) in absence and presence of 1.0 % hexamine were shown in Fig. 4. Electrochemical parameters such as corrosion potential (Ecorr), corrosion current density (icorr), anodic Tafel slope (βa), cathodic Tafel slope (βc) and percentage I.E. were given in Table-3. The curves show polarization of both, the cathodes as well as anodes. I.E. calculated from corrosion current obtained by the extrapolation of the cathodic and anodic lines. I.E. from Tafel plots agree well (within ± 1 %) with the values obtained from weight loss data. In general, an inhibitor is anodic or a cathodic if the variation of E_{corr} against the blank is higher or above than 85 mV [32]. In present study, the displacement of the E_{corr} was 103 mV, which suggest that hexamine function as an anodic type of inhibitor.

I.E. from (i_{corr}) was calculated using following equation [33]:

$$\text{I.E. } (\%) = \frac{i_{corr(uninh)} - i_{corr(inh)}}{i_{corr(uninh)}} \times 100 \qquad (9)$$

Where $i_{corr\ (uninh)}$ indicates corrosion current density in uninhibited acid whereas $i_{corr\ (inh)}$ indicates corrosion current density in inhibited acid.

Table 3 Polarization data and inhibition efficiency (IE %) of hexamine for zinc in (0.01 N HNO_3+ 0.01 N H_3PO_4) acid at 1.0 % Hexamine.

System	E_{corr} (mV)	I_{corr} (μA/cm^2)	Tafel slope (mV/decade)		B (mV)	I. E. (%) From methods	
			βa	- βc		By Weight Loss	By polarization
Blank	-935	0.1800	400	562	101.6	-	-
Hexamine	-832	0.0620	468	794	128.0	66.12	65.55

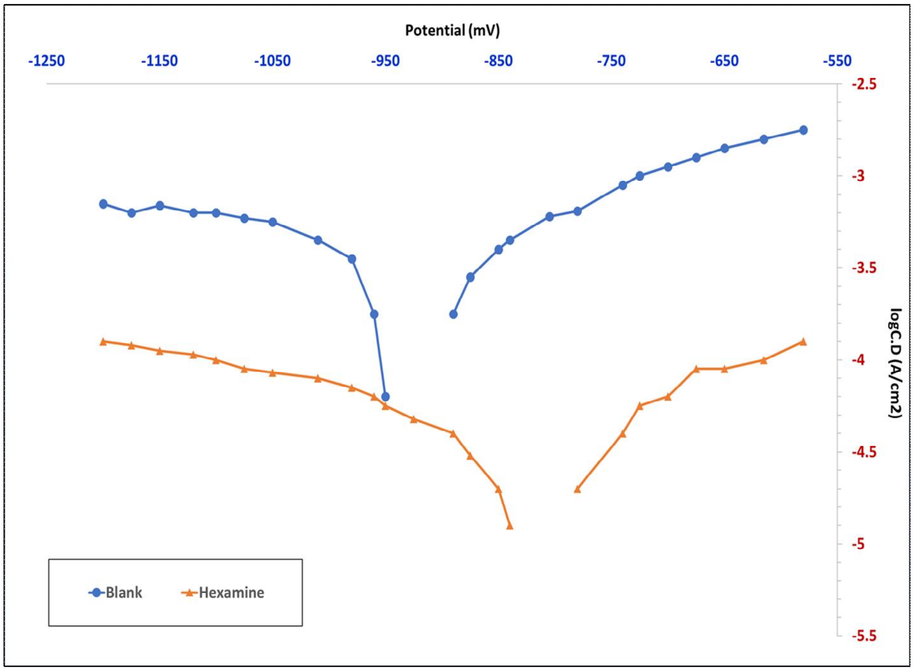

Fig .4 Polarisation cruves for corrosion of zinc in (0.01 N HNO_3+ 0.01 N H_3PO_4) binary acid mixture in absence and presence of 1.0 % hexamine.

L. Mechanism of Corrosion

Generally, zinc dissolves in mix acid solution due to somewhat hydrogen type of attack, the reaction taking place at the microelectrodes of the corrosion cell being represented as,

$Zn \rightarrow Zn^{2+} + 2e^-$ (anodic reaction) (10)

Reduction reaction is indicated by a decrease in valence or the consumption of electrons.

$2H^+ + 2e^- \rightarrow 2H$ (ads) (cathodic reaction) (11)

H_2 gas is liberated by any of the two following reactions:

$H + H = H_2 \uparrow$ (12)

Or $H + H^+ + e^- = H_2 \uparrow$ (13)

Or $H + H_3O^+ + e^- = H_2 \uparrow + H_2O$ (14)

M. Mechanism of Corrosion Inhibition by Hexamine

The mechanism of inhibition of corrosion is believed to be due to the formation and maintenance of a protective film on the metal surface.

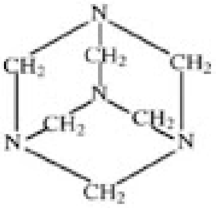

Fig.5 Structure of Hexamine

Macro molecular size and higher number of N-atoms of hexamine [34] might have covered almost all active source of zinc. Four nitrogen atom of the hexamine having high electron density must have functioned as the reaction centre [35] and the hexamine molecules might have been chemisorbed to form a thin monolayer on the zinc surface. The inhibitor action can be accounted by the interaction of lone pair of electrons in the nitrogen atom on the positively charged metal surface. The presence of six methylene groups also helps to lead to an enhancement of electron density at the nitrogen atom, which enhances its adsorption on the metal surface and basic strength of hexamine by inductive effect.

IV. CONCLUSION

On the basis of the study the following conclusions can be drawn:

A. As acid concentration increases corrosion rate increases with increase in I.E.
B. At constant acid concentration, as inhibitor concentration increases corrosion rate decreases while I.E. increases.
C. As temperature increase corrosion rate increases while I.E. decreases.
D. Hexamine showed maximum I.E. of 81.00 % at 1.0 % inhibitor concentration in (0.10 N HNO_3 + 0.10 N H_3PO_4).
E. The values of Ea obtained in the presence of the inhibitor were higher compared to the blank which indicates that inhibitor was more effective at lower temperature.
F. The values of ΔG^o ads were negative, which reveals the spontaneous adsorption of inhibitor onto metal surface.
G. Plot of log [θ / (1 - θ)] vs. log C shows straight line with almost unit slope, which suggest that the inhibitor cover both anodic and cathodic regions through general adsorption following Langmuir isotherm.
H. Polarization curves indicates that hexamine act as anodic type of inhibitor.

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