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Inhibiting Properties of Hexamine as Corrosion Inhibitor for Zinc in H_3PO_4 Solutions: Kinetic, Adsorption and Synergic Effect Study

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Abstract: ***The inhibition of corrosion of zinc in H_3PO_4 acid by Hexamine has been investigated by using weight loss, temperature effect and synergistic effect. As acid concentration increases corrosion rate increases while percentage of inhibition efficiency (I.E.) decreases. 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. Kinetic parameters: Rate constant (k) and Half-life ($t_{1/2}$) has also been studied. Higher the half-life period, higher will be I. E. and lower the corrosion rate. Hexamine showed maximum I.E. of 97.42 % at 80 mM inhibitor concentration in 0.05 M H_3PO_4. Initial OCP (Eim) values decreases (more negative direction) in presence of inhibitor compared to potential of uninhibited acid suggesting polarization of local cathodes.***

Keywords: Corrosion, Zinc, H_3PO_4, Hexamine, Synergic effect, OCP.

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. The major use of zinc for corrosion controls is in the form of coatings for steel.

Phosphoric acid is used primarily in the manufacture of detergents and pharmaceuticals. 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 [5-14] 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 [15-21]. In the present work, the corrosion of zinc by H_3PO_4 acid containing hexamine as an inhibitor was evaluated by using weight loss, temperature, synergistic effect and Open Circuit Potential (OCP) 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 (4.50 x 2.03 x 0.17 cm) of zinc having an area of 0.2054 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. Phosphoric acid was used as corrosive solution having concentration of 0.01, 0.05, 0.10 and 0.15 M prepared by diluting analytical grade of H_3PO_4 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 [22]. After cleaning, test specimens were washed with distilled water followed by acetone and dried with air. From the weight loss data, corrosion loss (CL) in mg/dm^2 and corrosion rate (CR) in mg/dm^2 d were calculated.

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C. Temperature Effect

To study the effect of temperature on corrosion of zinc in 0.05 M H_3PO_4 acid, the specimens were immersed in 230 ml of the corrosive solution and weigh loss was determined at solution temperature of 313, 323 and 333 K for an immersion period of 3 h in absence and presence ethylamine at 20,40,60 and 80 mM concentration. From the data corrosion rate, inhibition efficiency (I.E.) and Energy of activation (E_a) were calculated.

D. Synergistic Effect

Synergistic effect of KI was studied by using weight loss method. For this, corrosion loss was measured in 20 mM Hexamine alone and with combination of 5 mM KI in 0.05 M H_3PO_4 at 301K.

E. Open-Circuit Potential Measurement

One of the simplest qualitative methods to study the corrosion behaviour of metals is the measurements of OCP with time. For OCP measurement, zinc specimens having as area of 0.0268 dm^2 were immersed to 230 mL of 0.01 M H_3PO_4 in absence and presence of 20 mM inhibitor concentration. Potential was measured immediately after the immersion of metal specimens using saturated calomel electrode (SCE) as a reference electrode with the help of primary circuit, as a function of time. The potential values were till the potential attained a steady state value (E s,s.).

III. RESULTS AND DISCUSSION

A. Weight Loss Experiments

The corrosion rate of zinc in 0.05 M H_3PO_4 acid solution in absence and presence of 20,40, 60 and 80 mM 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:

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

I.E. were calculated as follows:

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

Where, W_u 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 Immersion Time

Corrosion loss increases with an increase in immersion time. Corrosion loss (CL) was increases as 983.59,1217.31, 1606.85 and 1887.64 mg/dm^2 corresponding to 6, 12, 18 and 24 h immersion time respectively in 0.05 M H_3PO_4 (Table 3.1, while percentage of I.E. of Hexamine decreases. In 0.05 M H_3PO_4 acid, Hexamine shows 83.16, 72.00, 69.69 and 66.72 % I.E. correspond to an immersion period of 6, 12, 18 and 24 h respectively (Fig. 3.1).

Table 3.1. Corrosion Loss (CL) and Inhibition Efficiency (I.E.) of zinc in 0.05 M H_3PO_4 acid containing 20 mM Hexamine with time at 301 ± 1 K.

Inhibitor	Immersion time (h)							
	6		12		18		24	
	CL (mg/dm^2)	I.E. (%)	CL (mg/dm^2)	I.E. (%)	CL (mg/dm^2)	I.E. (%)	CL (mg/dm^2)	I.E. (%)
Blank	983.59	-	1217.31	-	1606.85	-	1887.64	-
Hexamine	165.55	83.16	340.84	72.00	486.92	69.69	628.13	66.72

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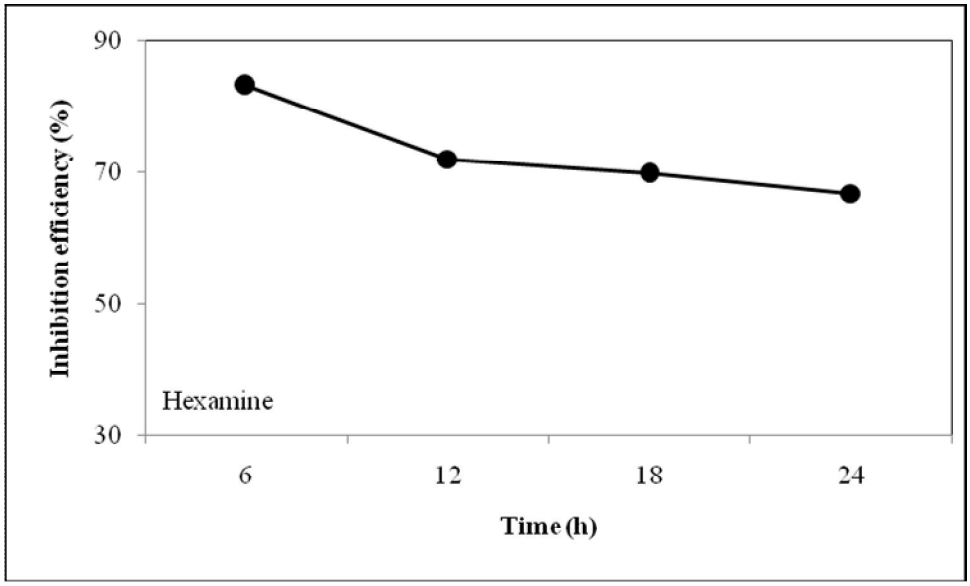

Fig.3.1 Effect of immersion time on corrosion of Zn in 0.05 M H_3PO_4 containing 20 mM Hexamine.

C. Effect of Inhibitor Concentration

At constant acid concentration, as the inhibitor concentration increases I.E. increases. In 0.05 M H_3PO_4 the I.E. was found to be 66.72, 93.03, 96.38 and 97.42 % corresponding to 20,40,60 and 80 mM inhibitor concentration respectively (Fig. 3.2).

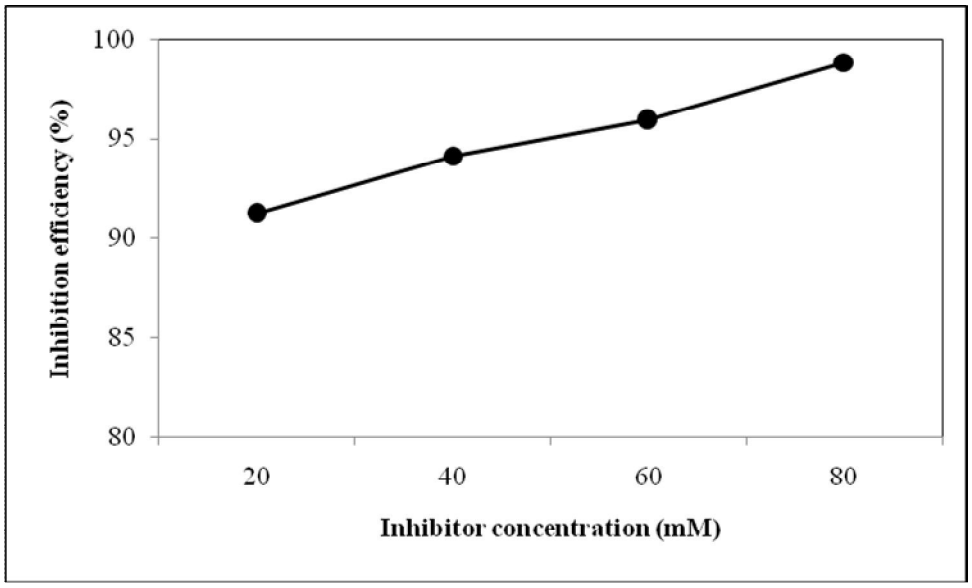

Fig. 3.2: Effect of inhibitor concentration on I.E. of Hexamine for zinc in 0.05 M H_3PO_4 at 301 K for an immersion period of 24 h.

D. Temperature Effect

To investigate the influence of temperature on corrosion of zinc, the weight loss experiments were also carried out at 313, 323 and 333 K in 0.05 M H_3PO_4 in absence and presence of 20, 40, 60 and 80 mM inhibitor concentration for an immersion period of 3h. As the temperature increases corrosion rate increases while percentage of I.E. decreases. Corrosion rate was increase as 993.32, 1192.96 and 1314.70 mg/dm^2 corresponding to 313, 323 and 333 K respectively in 0.05 M H_3PO_4 (Table-1). 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 [23], which results in intensification of the kinetic of electrochemical reaction [24] and thus explains the higher corrosion rate at elevated temperature.

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Table 3.2. Effect of temperature on the Corrosion loss (CL), value for zinc in 0.05 M H_3PO_4 acid at various concentrations of hexamine for an immersion period of 3 h.

Inhibitor	Inhibitor Conc. (mM)	Temperature (K)						Mean E_a (kJ mol^{-1})	'Ea' from Arrhenius Plot
		313		323		333			
		CL (mg/dm^2)	I.E. (%)	CL (mg/dm^2)	I.E. (%)	CL (mg/dm^2)	I.E. (%)		
Blank	-	993.32	-	1192.96	-	1314.70	-	12.7	15.3
Hexamine	20	87.64	91.17	107.12	91.02	121.73	90.74	14.2	14.1
	40	83.30	93.62	77.90	93.47	92.51	92.96	17.8	17.7
	60	43.82	95.58	53.56	95.51	68.16	94.81	19.4	19.1
	80	14.60	98.53	19.40	98.36	29.21	97.77	30.8	30.4

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 [25].

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

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 calculated from Arrhenius plot were found higher in inhibited acid (Ranging from 14.1 to 30.4 kJ mol^{-1}) than the 'Ea' value for uninhibited system (15.3 kJ mol^{-1}) (Table-2). The higher values of mean 'Ea' indicate physical adsorption of the inhibitors on metal surface [26]. The values of 'Ea' calculated from the slop Arrhenius plot of log p versus 1/T x 1000 (Fig.- 3.3) and using equation-4 were almost similar.

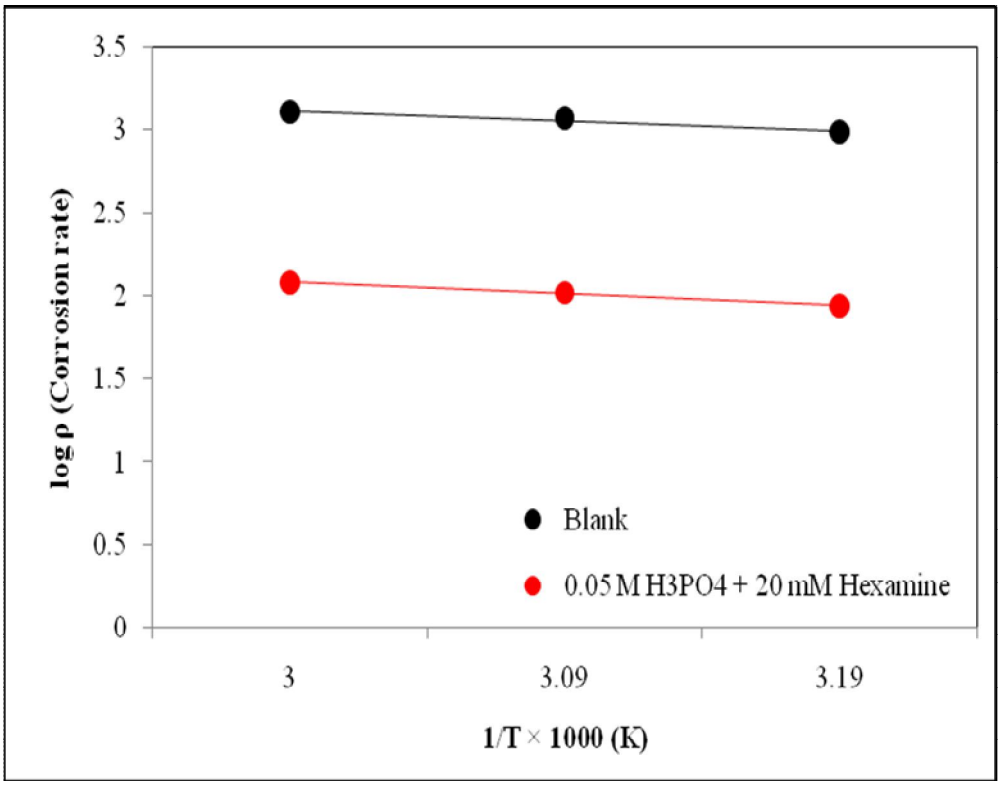

Fig. 3.3 Arrhenius plots for corrosion of zinc in 0.05 M H_3PO_4 in absence and presence of 20 mM hexamine.

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F. Synergistic Effect

Synergism is a combined action of compounds greater in total effect than the sum of individual effects. It has been reported that iodide ion is the most adsorbable of halide ions on steel [27] therefore; addition of iodide ion with an organic inhibitor enhances the I.E. Table-3.3 gives I.E of 20 mM Hexamine alone and for 20 mM Hexamine in combination with 5 mM KI in 0.05 M H_3PO_4 at 301K. It is generally accepted that the presence of halide ions in acidic media synergistically increases the I. E. of some organic compounds. It is thought that the anions are able to improve adsorption of the organic cations in solution by forming intermediate bridges between the metal surface and the positive end of the organic inhibitor. Thus, the inhibitor is not adsorbed directly on the metal itself but rather by columbic attraction to the adsorbed halide ions on the metal surface. Corrosion inhibition synergism then results from increased surface coverage arising from ion-pair interactions between the organic cations and the anions. The greater influence of the iodide ion is often attributed to its large ionic radius, high hydrophobicity, and low electronegativity, compared to the other halide ions [28,29].

Table 3.3. Synergistic effect of KI (5 mM) on inhibition of zinc in 0.05 M H_3PO_4 acid containing 20 mM Hexamine at 301 ± 1 K.

Inhibitor	I. E. (%)		Percentage increment
	Without KI	With KI	
I	II	III	IV
Hexamine	66.72	84.17	18.05

G. Kinetic parameters: Rate constant (k) and Half-life ($t_{1/2}$)

The rate constant 'k' was calculated using the following equation [30] ,

$$k = 1/t \quad \ln (W_i/ W_f) \qquad (5)$$

Where, 'W_i' is the initial weight of the sample, 'W_f' is the final weight of the sample, 't' is an immersion time (in hours).

The values of half-life ($t_{1/2}$) were calculated by using the following equation [31] ,

$$t_{1/2} = 0.693 / k \qquad (6)$$

where, 't' is time in hours and 'k' is rate constant.

Corrosion rate constant 'k' and Half life '$t_{1/2}$' for Zn in H_3PO_4 was shown in Table-3.4.

Table-3.4 : Kinetic parameters: Rate constant 'K' and Half-life '$t_{1/2}$' for the corrosion of zinc in 0.01, 0.05, 0.10 and 0.15 M concentration of H_3PO_4 containing various concentration of Hexamine.

Inhibitor	Inhibitor concentration (mM)	Acid concentration							
		0.01 M		0.05 M		0.10 M		0.15 M	
		Rate const. ($k\times10^{-3}$) (day^{-1})	Half - life ($t_{1/2}$) (day)	Rate const. ($k\times10^{-3}$) (day^{-1})	Half - life ($t_{1/2}$) (day)	Rate const. ($k\times10^{-3}$) (day^{-1})	Half-life ($t_{1/2}$) (day)	Rate const. ($k\times10^{-3}$) (day^{-1})	Half-life ($t_{1/2}$) (day)
Blank	---	8.52	81.32	45.26	15.30	87.03	7.96	129.80	5.34
Hexamine	20	1.03	672.81	14.92	46.44	46.10	15.03	72.48	9.56
	40	0.79	877.21	3.10	223.54	31.41	22.06	53.11	13.07
	60	0.56	1237.50	1.58	438.60	12.11	57.22	32.57	21.27
	80	0.34	2038.23	1.14	607.89	5.51	125.77	14.04	49.35

As concentration of inhibitor increases rate constant 'k' decreases whereas the half-life values are increases [32]. Corrosion rate constant 'k' increases with increase in acid concentration (Table-3.4). The action of organic inhibitors depends on the type of interaction between the substance and the metallic surface. This interaction causes a change either in the electrochemical process mechanism or in the surface available to the process [33].

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H. Effect of Open Circuit Potential (OCP)

Initial Values of OCP (Eim) and steady state (Es.s) for zinc in 0.01 M in H_3PO_4 in absence and presence of 20 mM Hexamine at room temperature were shown in Figure 3.4. It is noted that the potential for the solution without inhibitor gradually decreases with time i.e. from -921 mV and stabilizes at the value of -956 mV, after 20 min. of immersion. Whereas, in the presence of inhibitor, the OCP (Eim) values decreases (more negative direction) compared to initial potential (Eim) value of uninhibited acid (-921 mV) i.e. -1019 mv for Hexamine which suggesting polarization of local cathodes. This finding indicated the formation of a protective layer at the metallic surface [34]. Thereafter the potential moves in less negative direction with time and settles at -961 mV (Es.s) in 24 min for Hexamine.

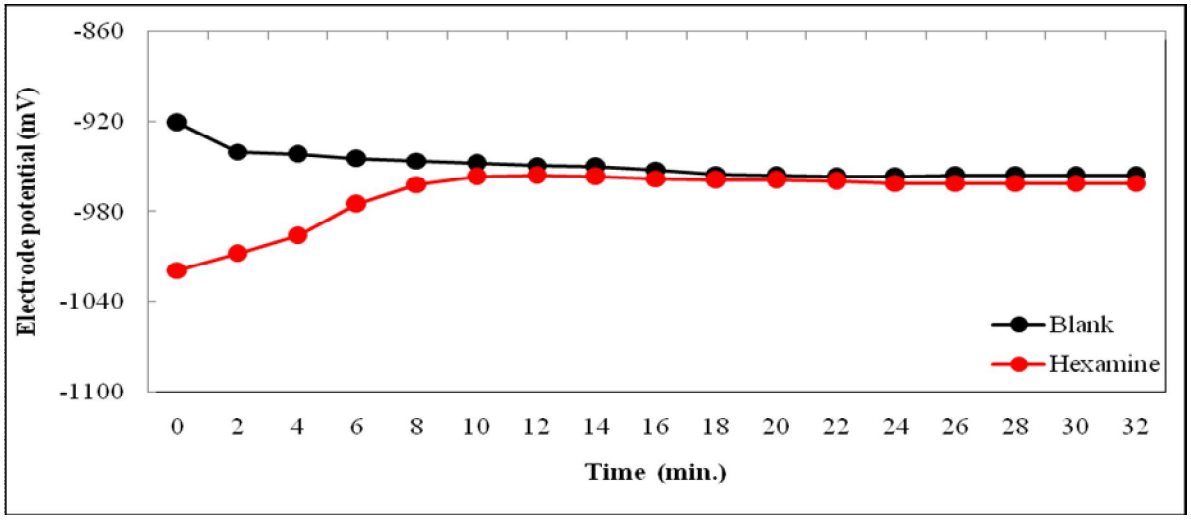

Fig.3.4: Variation in OCP with time for zinc 0.01 M H_3PO_4 in absence and presence of 20 mM of Hexamine.

It is quite understandable that change in potential with time depends on a number of factors such as area of local anodes and cathodes, kinetics of anodic and cathodic reactions, anions present, dissolved oxygen and hence over simplification is difficult and also not desirable.

I. Mechanism of Corrosion Inhibition by Hexamine

The mechanism of inhibition of corrosion of zinc by hexamine is believed to be due to the formation and maintenance of a protective film on the metal surface. Macro molecular size and higher number of N-atoms of hexamine [35] 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 [36] 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. At constant acid concentration, as inhibitor concentration increases corrosion loss decreases while I.E. increases.
B. As temperature increase corrosion rate increases while I.E. decreases.
C. Hexamine showed maximum I.E. of 97.42 % at 80 mM inhibitor concentration in 0.05 M H_3PO_4.
D. 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.
E. Hexamine suggesting polarization of local cathodes.

V. ACKNOWLEDGEMENT

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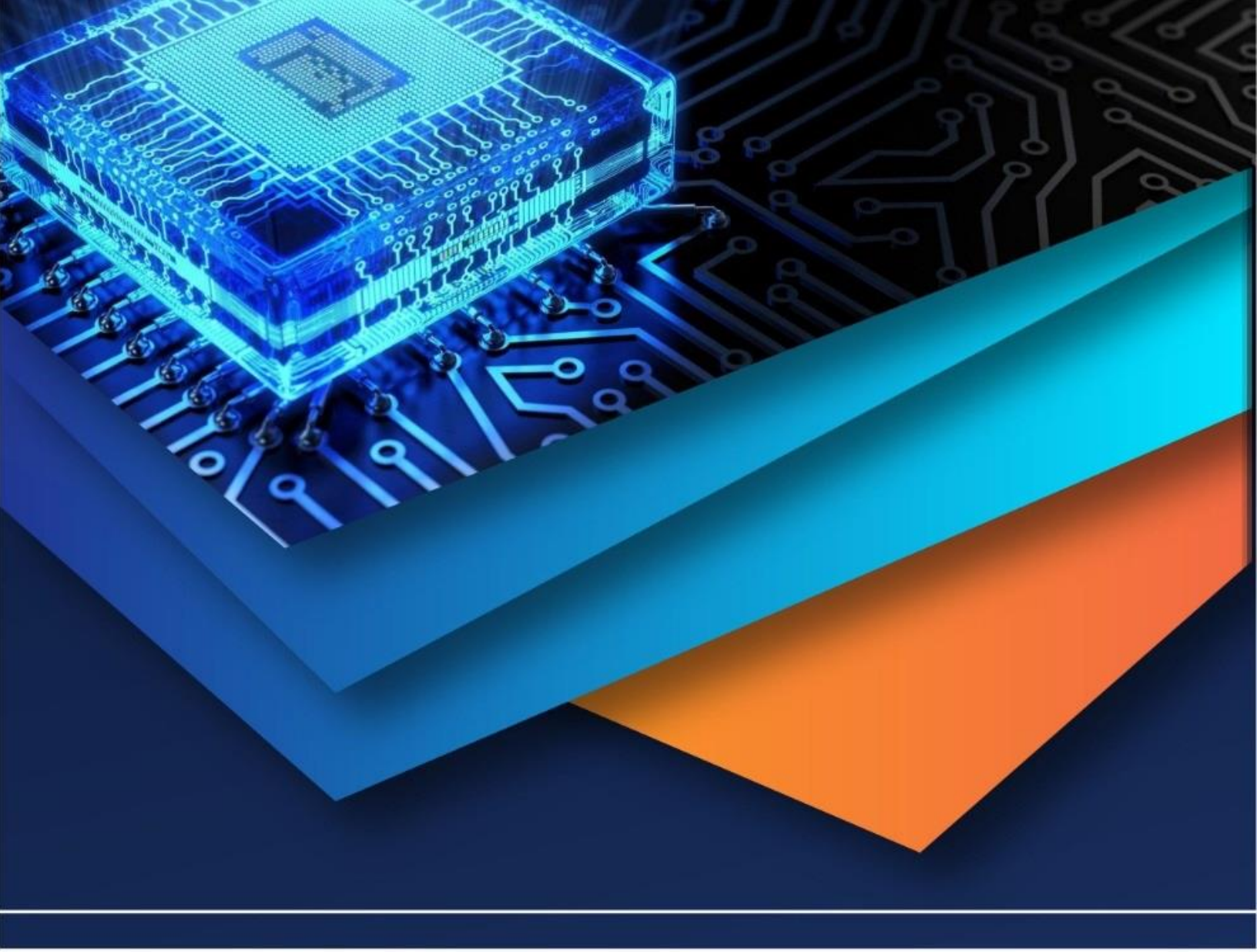

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