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Inhibition of corrosion of zinc in sulphuric acid by ethylamines

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Abstract

The corrosion of zinc in sulphuric acid and its prevention by ethylamines has been studied in this investigation. In plain acid, corrosion increases with the concentration of acid and with the temperature. At constant acid concentration, the inhibition efficiency (I.E.) of ethylamines increases with inhibitor concentration. Similarly, at constant inhibitor concentration, the I.E. increases with the increase in acid concentration. Diethylamines shows excellent inhibition in all acid concentrations as well as at all inhibitor concentration at 310 K. At all acid concentrations and at 30, 40, 50 and 60 mM inhibitor concentration at 310 K, the I.E. of inhibitors decreases in the order: diethylamine > triethylamine > ethylamine. As temperature increases, percentage of inhibitor decreases. Plot of $\log(\theta/1-\theta)$ versus $\log C$ results in a straight line suggest that the inhibitors cover both the anodic and cathodic regions through general adsorption following Langmuir isotherm. Galvanostatic polarization curves show both anodic and cathodic polarization.

Keywords: Corrosion, zinc, sulphuric acid, ethylamines

Introduction

Sulphuric acid (H_2SO_4) is a strong acid and is used as a cleaner for rust, algae and scale from condensers and cooling tower^[1]. Zinc is one of the most vital nonferrous metal, having extensive coating and in construction material. Zinc sheet immersed in various concentration in sulphuric acid indicates a various corrosion rate viz. 2200 mil/y for 12% and 730 mil/y for 20% sulfamic acid solution for 48-95 h at 20 to 25 °C^[2]. Vashi and Bhajiwala^[3] studied ethylamines as corrosion inhibitors for zinc in (HNO_3+HCl) binary acid mixture and ethylamine has been found to be as efficient inhibitor. Vashi and Naik^[4] studied ethylamines as corrosion inhibitor for zinc in phosphoric acid and found that triethylamine shows better I.E. than ethylamine and diethylamine at 80 mM inhibitor concentration in 0.01 M acid. The present study was undertaken to evaluate various ethylamines as corrosion inhibitors for zinc in sulphuric acid.

Experimental

Materials

Rectangular specimens (5.5 X 2.5 X 0.2 cm) of zinc having an area of 0.3013 dm² were used for the determination of the corrosion rate. The chemical composition of test specimen contain 99.390% Zn, 0.490% Mn and 0.121% Co. All specimens were cleaned by buffing to obtain a mirror like finish. All chemicals used were of AR grade. The corrosive solution was prepared in double distilled water.

Weight loss measurements

For weight-loss measurement, the zinc coupons having an area of 0.3013 dm² were each suspended and completely immersed in 230 ml of 0.1, 0.3 and 0.5 M H_2SO_4 solution without and with different concentrations of ethylamine, diethylamine and triethylamine at 301 ± 1 K for 24 h immersion period. After the test, the specimen was cleaned by using 10% CrO_3 acid solution having 0.2% $BaCO_3$ ^[5]. After cleaning, test specimens were washed with double distilled water followed by acetone and dried with air drier. The mean value of corrosion rate was reported as $mg\ dm^{-2}\ d^{-1}$ shown in Table 1. The coupons were retrieved after 24 h, washed by distilled water, dried well and reweighed. From the weight loss data, the corrosion rate (CR) were calculated.

Temperature effect

To study the effect of temperature on corrosion of zinc in 0.1 M sulphuric acid, the specimens was immersed in 230 ml of the corrosive solution and weigh loss was determined at solution

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temperature of 313, 323 and 333 K for an immersion period of 3 h without and with ethylamines at 60 mM concentration. Attention is paid to compensate the evaporation loss of corrosive media. Wesley [6] and ASTM [7] pointed out that thermostatic control to within ± 1 °C usually be considered satisfactory. From the data, % I.E., energy of activation (Ea) and heat of adsorption (Q_{ads}) were calculated.

Polarization measurements

For polarization study, metal specimens having an area of 1 cm² were immersed to 230 ml 0.1 M H₂SO₄ in absence and presence of ethylamines in the test cell (using CH instruments, Inc., USA) includes the zinc metal specimens as a working electrode, Ag/AgCl electrode as a reference electrode as well as platinum electrode as an auxiliary electrode and allowed to establish a steady-state open circuit potential (OCP) for approximately 70 min. 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}) [8].

Electrochemical impedance spectroscopy measurements

EIS measurements were made at corrosion potentials over a frequency range of 1 KHz to 100 KHz by a sine wave with a potential perturbation amplitude of 5 mV. The real Z' and imaginary Z'' parts were measured at various frequencies. From the plot of Z' Vs. Z'', the charge transfer resistance (R_{ct}), and double layer capacitance (C_{dl}) were calculated. Impedance measurements were carried out both in the absence and presence of inhibitor.

Results and Discussion

The results were presented in Tables 1 to 4 and in Figure 1 to 10. To assess the effect of corrosion of zinc in sulphuric acid ethylamines were added as an inhibitors.

I.E. has been calculated as follows:

$$\text{I. E.} = \frac{W_u - W_i}{W_u} \times 100 \dots \dots \dots (1)$$

Where, W_u is the weight loss of metal in uninhibited acid and W_i is the weight loss of metal in inhibited acid.

Energy of activation (Ea) has been calculated from the slope of log ρ versus 1/T (ρ = corrosion rate, T = absolute temperature) (Fig. 1) and also with the help of the Arrhenius equation [9].

$$\log \frac{\rho_2}{\rho_1} = \frac{Ea}{2.303R} \left[\left(\frac{1}{T_1} \right) - \left(\frac{1}{T_2} \right) \right] \dots \dots \dots (2)$$

Where, ρ₁ and ρ₂ are the corrosion rate at temperature T₁ and T₂ respectively.

The value of heat of adsorption (Q_{ads}) was calculated by the following equation [10].

$$Q_{ads} = 2.303 R \left[\log \left(\frac{\theta_2}{1 - \theta_2} \right) - \log \left(\frac{\theta_1}{1 - \theta_1} \right) \right] \times \left[\frac{T_1 \times T_2}{T_2 - T_1} \right] \dots \dots \dots (3)$$

Where, θ₁ and θ₂ [θ = (W_u - W_i)/W_u] are the fractions of the metal surface covered by the inhibitor at temperature T₁ and T₂ respectively.

The values of the free energy of adsorption (ΔG_a) were calculated with the help of the following equation [10].

$$\log C = \log \left(\frac{\theta}{1 - \theta} \right) - \log B \dots \dots \dots (4)$$

Where, log B = -1.74 - $\left(\frac{\Delta G_a}{2.303 RT} \right)$ and C is the inhibitor concentration.

The enthalpy of adsorption (ΔH_a) [11] and entropy of adsorption [12] (ΔS_a) were calculated using the equation.

$$\Delta H_a^0 = Ea - RT \dots \dots \dots (5)$$

$$\Delta S_a^0 = \frac{\Delta H - \Delta G}{T} \dots \dots \dots (6)$$

Inhibitor efficiency (I.E.) from I_{corr} was calculated using following equation.

$$\text{I. E.} (\%) = \frac{i_{\text{corr}}(\text{uninh}) - i_{\text{corr}}(\text{inh})}{i_{\text{corr}}(\text{uninh})} \times 100 \dots \dots \dots (7)$$

The charge transfer resistance (R_{ct}) values were calculated from the difference in impedance at lower and higher frequencies. To obtain the double layer capacitance (C_{dl}), the frequency at which the imaginary component of the impedance is maximum was calculated by using the following equation [13].

$$C_{dl} = \frac{1}{2 \pi f R_{ct}} \dots \dots \dots (8)$$

Where f is the frequency at the maximum height of the semicircle on the imaginary axis and R_{ct} is the charge transfer resistance [14].

I.E. from C_{dl} was calculated using following equation.

$$\text{I. E.} (\%) = \frac{C_{dl}(\text{uninh}) - C_{dl}(\text{inh})}{C_{dl}(\text{uninh})} \times 100 \dots \dots \dots (9)$$

Corrosion by plain sulphuric acid: The corrosion rate increases with the increase in concentration of acid. The corrosion rate was 1035.50, 2880.84 and 6179.88 mg dm⁻² d⁻¹ in 0.1, 0.3 and 0.5 M H₂SO₄ concentrations respectively for a period of 24 h at 301 ± 1 K as shown in Table-1.

Effect of inhibitor concentration: At constant acid concentration, the I.E. of the ethylamines increases with the inhibitor concentration, e.g. in case of ethylamine in 0.1 M H₂SO₄ the I.E. was found to be 65.68, 71.46, 87.47 and 91.03%, in case of diethylamine 69.52, 80.12, 90.70 and 93.26 and for triethylamine found to be 68.07, 77.36, 89.21 and 92.75% correspond to 30, 40, 50 and 60 mM inhibitor concentration respectively [Table- 1].

Effect of acid concentration: At constant inhibitor concentration, the I.E. increases with the increase in acid concentration. At 50 mM inhibitor concentration, the I.E. of ethylamine was 87.47, 91.15 and 93.46%, for diethylamine it is 90.70, 92.63, 94.84% and for triethylamine it was 89.21, 91.68 and 94.08% with respect to 0.1, 0.3 and 0.5 M acid concentration respectively [Table-1].

Effect of temperature: To determine the effect of temperature on corrosion, corrosion loss was measured in 0.1 M sulphuric acid containing 30, 40, 50 and 60 mM inhibitor concentration at solution temperature of 313, 323 and 333 K for an immersion period for 3 h. As the temperature is increases, corrosion loss increases while percentage of I.E. decreases (Table-2). Corrosion loss (CL) increases with temperature may be due to the desorption of the adsorbed molecules inhibitor and/or aggressive at higher temperature and thus exposing the fresh metal surface to further attack [15]. which results in

intensification of the kinetic of electrochemical reaction^[16] and thus explains the higher corrosion rate at elevated temperature. The addition of ethylamines in corrosive media indicates that as the temperature increases I.E. decreases, e.g. in 0.1 M H₂SO₄ at 60 mM inhibitor concentration, the I.E. for ethylamine was 81.20, 77.30 and 74.54% at 313, 323 and 333 K respectively.

Energy of activation (E_a): The mean 'E_a' Value from equation (2) for Zinc in 0.1 M H₂SO₄ was 14.25 kJ mol⁻¹. A comparison of the values of the E_a for the corrosion process in inhibited (in a range of 22.15 to 49.69 kJ mol⁻¹) which indicates that the "E_a" values were higher in inhibited than in uninhibited acid (Table-2). The higher values of mean E_a, indicates physical adsorption of the inhibitors on metal surface^[17]. The value of E_a calculated from the slop of Arrhenius plot (Fig. 1) and using equation 2 were almost similar.

Heat of adsorption (Q_{ads}): From Table 2 it was evident in all cases Q_{ads} values were negative range from -11.67 to -61.44 kJ mol⁻¹. As the temperature increases values of Q_{ads} decreases (become more negative). The negative Q_{ads} values shows that the adsorption and hence the I.E. decreases with rise in temperature^[18].

Adsorption isotherm: The graph of log [θ / (1 - θ)] vs. log C for aliphatic amines (ethylamine, diethylamine and triethylamine) in 0.1 M H₂SO₄ was depicted in Figure 2. A straight line in all the inhibitors studied. A straight line was obtained proving the fact that the adsorption of these compounds on the zinc surface obeys the Langmuir adsorption isotherm.

Free energy of adsorption (ΔG^o_a): The values of mean ΔG^o_a calculated from Table- 2. In all cases mean ΔG^o_a values were negative and lie in the range of -20.64 (ethylamine) to -22.64 (triethylamine). The negatives values of (ΔG^o_a) indicated the spontaneous adsorption of the inhibitor. This is usually characteristic of strong interaction with metal surface. ΔG^o_a values were more positive than -40 kJmol⁻¹ indicating that inhibitor is physically adsorbed on the metal surface^[11].

Enthalpy of adsorption (ΔH^o_{ads}): The enthalpy of adsorption (ΔH^o_{ads}) were positive as 17.56 kJ mol⁻¹ (ethylamine) to 61.25 kJ mol⁻¹ (diethylamine) indicating the endothermic nature of the reaction¹¹ suggesting that higher temperature favors the corrosion process.

Entropy of adsorption (ΔS^o_{ads}): The entropy (ΔS^o_{ads}) values were positive as 0.12 kJ mol⁻¹K⁻¹ (ethylamine) to 0.98 kJ mol⁻¹K⁻¹ (diethylamine) confirming that the corrosion process is entropically favourable^[12].

Potentiodynamic Polarization Measurements: Potentiodynamic polarization curve of zinc in 0.1 M H₂SO₄ in absence and presence of 60 mM ethylamines were shown in Fig.-3 to Fig-5. Electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}) anodic Tafel slope (β_a), cathodic Tafel slope (β_c) and percentage inhibition efficiency (I.E.) were given in Table 3. The curves show polarization of both, the cathodes as well as anodes. (Fig. 3-6) I.E. calculated from corrosion current obtained by the extrapolation of the cathodic and anodic Tafel lines are given in Table 3. In almost all the cases, the I.E. from Tafel plots

agree well (within ± 4%) with the values obtained from weight loss data.

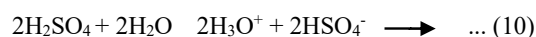
Electrochemical impedance spectroscopy (EIS) measurements: Corrosion of zinc in 0.1 M H₂SO₄ in presence of ethylamines was investigated by ESI at room temperature. Nyquist curves for zinc obtain in absence and presence of 60 mM ethylamines as inhibitors were shown in figure 7-10 and Table-4.

The addition of inhibitor increase R_{ct} value while C_{dl} decrease. This decrease in C_{dl} value was due to the adsorption of inhibitor on metal surface. The results suggest that the inhibitor acts by the formation of a protective layer on the surface, which modifies the metal/solution interface.

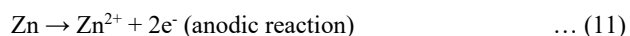
It was observed that the impedance diagram was almost semicircular in appearance, but not perfect semicircle. The difference has been attributed to frequency dispersion. The semicircle nature of the plots indicates that the corrosion of zinc is mainly controlled by charge transfer process.

Mechanism of corrosion

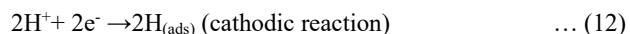
Sulphuric acid acts as strong acid. The probable chemical reaction taking place in sulphuric acid is as under. It undergoes hydrolysis in aqueous solution.



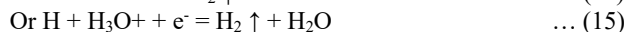
Generally, zinc dissolves in sulphuric acid solution due to somewhat hydrogen type of attack, the reaction taking place at the microelectrodes of the corrosion cell being represented as,



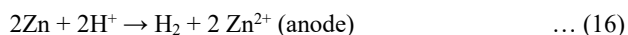
Reduction reaction is indicated by a decrease in valence or the consumption of electrons.



H₂ gas is liberated by any of the two following reactions:

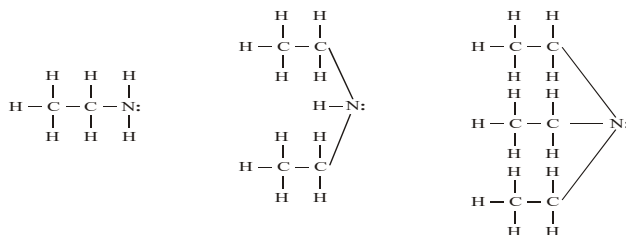


The following secondary reactions can also take place in acid solution:



Corrosive attack of sulphuric acid on zinc which may be mainly due to the formation of HSO₄⁻ during the ionization of acid. However, inhibitors prevent the auto-catalytic cycle of the formation of HSO₄⁻ which results into inhibitor of the corrosion of zinc in sulfuric acid.

In 0.1 M sulphuric acid, generally at all inhibitor concentration the orders of I.E. of these three ethylamines were in the order: diethylamine > triethylamine > ethylamine. In case of all these three inhibitors studied diethylamine was found to be most effective as it show 93.26, 94.84 and 96.12% I.E. in 0.1, 0.3 and 0.5 M sulphuric acid at 60 mM inhibitor concentration respectively. It was evident that ethylamine shows 65.68, 71.46, 87.47 and 91.03% I.E. while diethylamine shows 69.52, 80.12, 90.70 and 93.26% I.E. in 0.1 M sulphuric acid at 30, 40, 50 and 60 mM inhibitor concentration.

Ethylamine
pka = 10.63Diethylamine
pka = 10.80Triethylamine
pka = 10.72

+I effect (low) +I effect (medium) +I effect (high) It was evident that ethylamine show lowest I.E. in all acid concentration. While diethylamine shows higher IE in all acid concentration.

As pka value increases, decreases its acidity hence increases its basicity so I.E. increases. The number of ethyl group while lone pair (l.p.) of electron remains same in all these three inhibitors. The number of the functional groups is assumed [19] to reduce the protective properties owing to steric hindrance but the protection could be improved by functional groups acting adsorption centers.

Table 1: Effect of H₂SO₄ concentration on corrosion rate (CR) and inhibitor efficiency (I.E.) on zinc metal having different concentration of ethylamines.

Inhibitor	Inhibitor concentration (mM)	Acid concentration					
		0.1 M		0.3 M		0.5 M	
		CR (mg dm ⁻² d ⁻¹)	I. E.(%)	CR (mg dm ⁻² d ⁻¹)	I. E.(%)	CR (mg dm ⁻² d ⁻¹)	I. E. (%)
I	II	III	IV	V	VI	VII	VIII
Blank	-	1035.5	-	2880.84	-	6179.88	-
Ethylamine	30	355.38	65.68	887.87	69.18	1689.57	72.66
	40	295.53	71.46	670.24	76.04	1366.98	77.88
	50	129.74	87.47	254.95	91.15	404.16	93.46
	60	92.88	91.03	210.00	92.71	310.84	94.97
Diethylamine	30	311.62	69.52	829.97	71.19	1472.04	76.18
	40	205.85	80.12	447.39	84.47	832.42	86.53
	50	96.30	90.70	212.31	92.63	318.88	94.84
	60	69.79	93.26	148.65	94.84	239.77	96.12
Triethylamine	30	330.63	68.07	847.54	70.58	1519.01	75.42
	40	234.43	77.36	538.14	81.32	1050.57	83.00
	50	111.73	89.21	239.68	91.68	365.84	94.08
	60	75.07	92.75	170.54	94.08	266.97	95.68

Table 2: Effect of temperature on the Corrosion Loss (CL), Energy of activation (E_a), Heat of adsorption (Q_{ads}), Free energy of adsorption (ΔG°_a) for zinc in 0.1 M sulphuric acid at various concentrations. Effective area of specimen: 0.3013 dm² Immersion period: 3 h

Inhibitor	Inhibitor Conc. (mM)	Temperature (K)						Mean E _a kJ mol ⁻¹	'E _a ' from Arrhenius Plot	Heat of adsorption, Q _{ads} (kJ mol ⁻¹)	
		313		323		333				313-323 K	323- 333 K
		CL (mg/dm ²)	I.E. (%)	CL (mg/dm ²)	I.E. (%)	CL (mg/dm ²)	I.E. (%)				
Blank	-	1324.26	-	1549.95	-	1838.69	-	14.25	14.06	-	-
Ethylamine	30	514.33	61.16	653.83	57.81	856.28	53.42	22.15	21.83	-11.67	-16.25
	40	431.46	64.41	600.73	61.24	823.09	55.23	27.99	27.68	-22.68	-22.12
	50	328.57	75.18	477.92	69.16	867.02	52.84	31.97	31.61	-25.28	-22.99
	60	248.92	81.12	351.80	77.30	467.97	74.54	27.30	27.07	-19.55	-13.53
Diethylamine	30	481.24	63.65	680.38	56.10	906.07	50.72	27.55	27.11	-26.50	-19.34
	40	345.17	73.93	484.56	68.73	713.57	61.19	31.46	31.11	-21.41	-29.71
	50	169.26	87.21	361.78	76.65	537.67	70.75	49.64	49.64	-61.44	-27.14
	60	112.84	91.47	212.41	86.29	345.17	81.22	48.30	47.96	-47.79	-33.56
Triethylamine	30	514.43	61.15	690.34	55.46	925.98	49.63	25.49	25.19	-19.71	-18.28
	40	355.12	73.18	491.20	68.30	713.57	61.19	30.33	29.89	-19.86	-27.92
	50	265.51	79.97	381.67	75.37	527.71	71.29	29.74	29.44	-22.26	-18.69
	60	165.94	87.46	255.55	83.51	398.27	78.33	37.99	37.51	-26.88	-30.17

Table 3: Polarization data and inhibition efficiency (IE %) of ethanolamine for zinc in 0.1 M sulphuric acid

System	E _{corr} (V)	I _{corr} (μA/cm ²)	Tafel slope (mV/decade)		B (V)	IE (%)	
			+β _a	-β _c		Weight Loss	By polarization
Blank	-1.094	2.232	0.4133	0.2191	0.0622	-	-
Ethylamine	-1.050	0.5312	0.1283	0.1814	0.0326	91.03	76.20
Diethylamine	-1.050	0.1761	0.1225	0.1939	0.0326	93.26	92.91
Triethylamine	-1.063	0.1653	0.1091	0.1684	0.0287	92.75	92.59

Effective area of specimen: 1 cm²

Inhibitor concentration: 60 mM

B_a = Anodic Tafel constant, β_c = Cathodic Tafel constant,

CD = Corrosion current density from interception of anodic and cathodic lines.

Table 4: EIS parameters for the corrosion of zinc in 0.1 M H₂SO₄ in the absence and presence of ethyl amines

System	R _{ct} (Ω cm ²)	C _{dl} (μF cm ⁻²)	IE (%) calculated from	
			EIS method	Weight loss method
Blank	49.5	151.84	-	-
Ethylamine	187.0	11.20	92.62	91.03
Diethylamine	290.0	4.90	96.77	93.26
Triethylamine	320.0	3.31	97.55	92.75

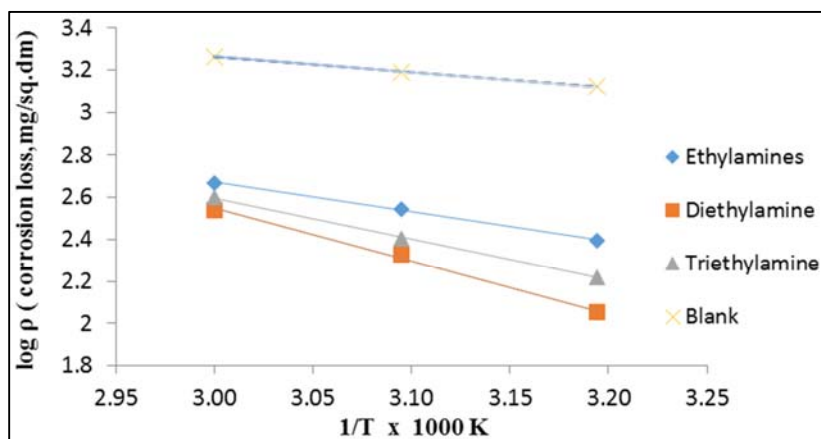


Fig. 1: Arrhenius plots for corrosion of zinc in 0.1 M sulphuric acid in absence and presence of 60 mM inhibitor concentration.

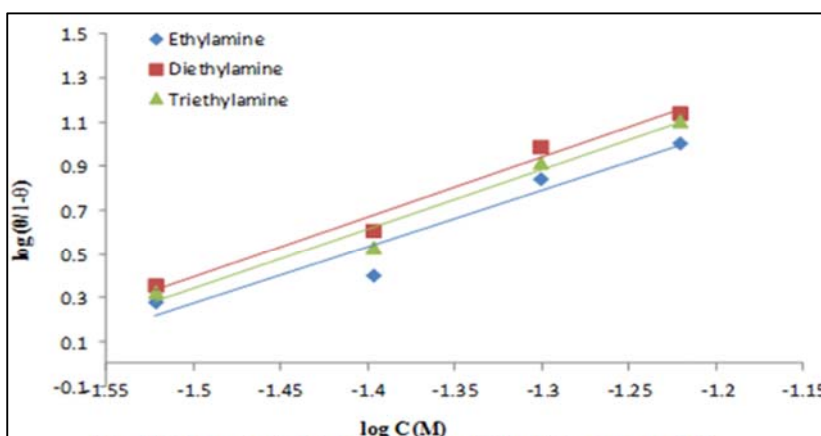


Fig. 2: Plot of $\log [\theta/(1-\theta)]$ versus $\log C$ for ethylamines in 0.1 M sulphuric acid concentration

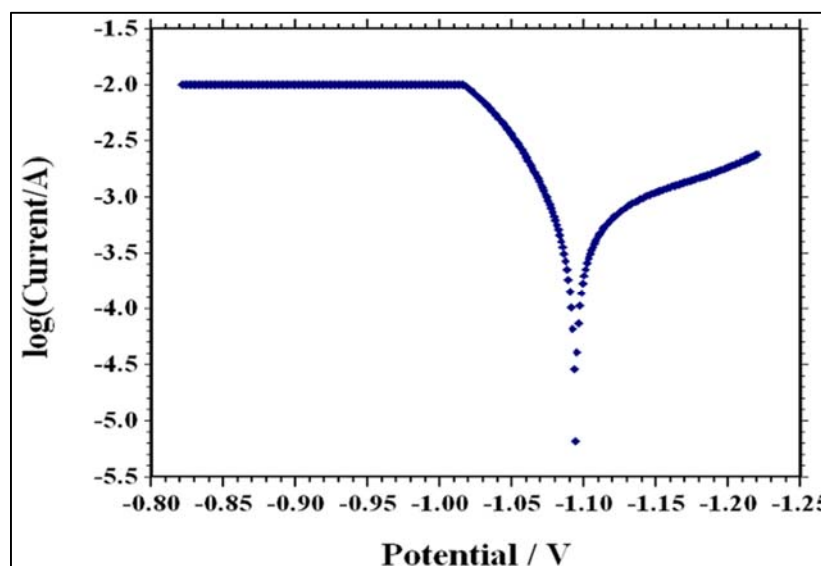


Fig 3: Polarization for corrosion of zinc in 0.1 M Sulphuric acid.

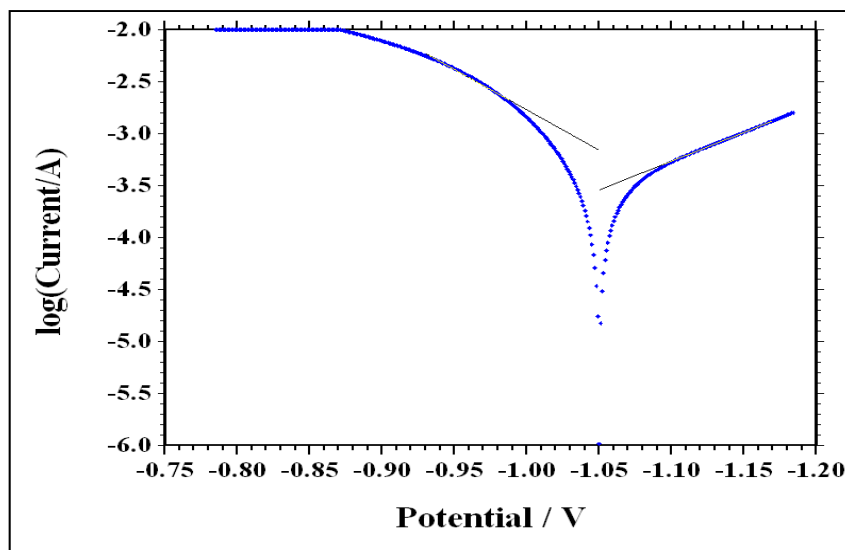


Fig 4: Polarization curve for corrosion of zinc in 0.1 M Sulphuric acid containing 60 mM Ethylamine.

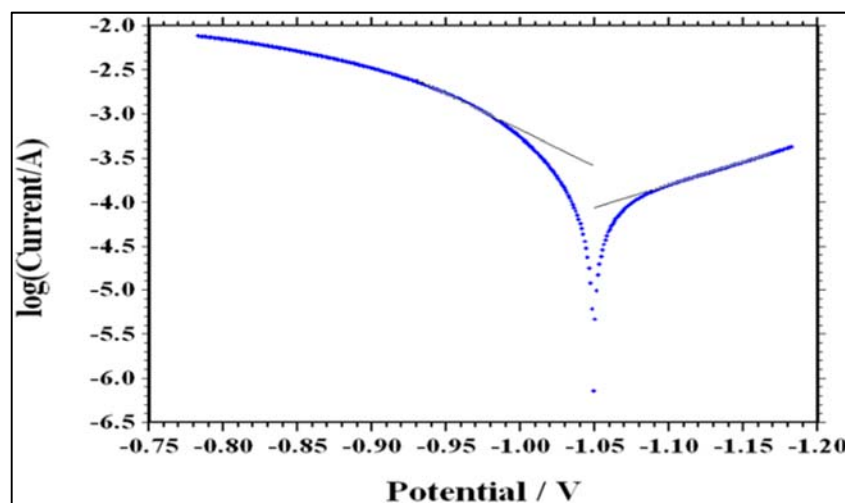


Fig 5: Polarization curve for corrosion of zinc in 0.1 M Sulphuric acid containing diethylamine.

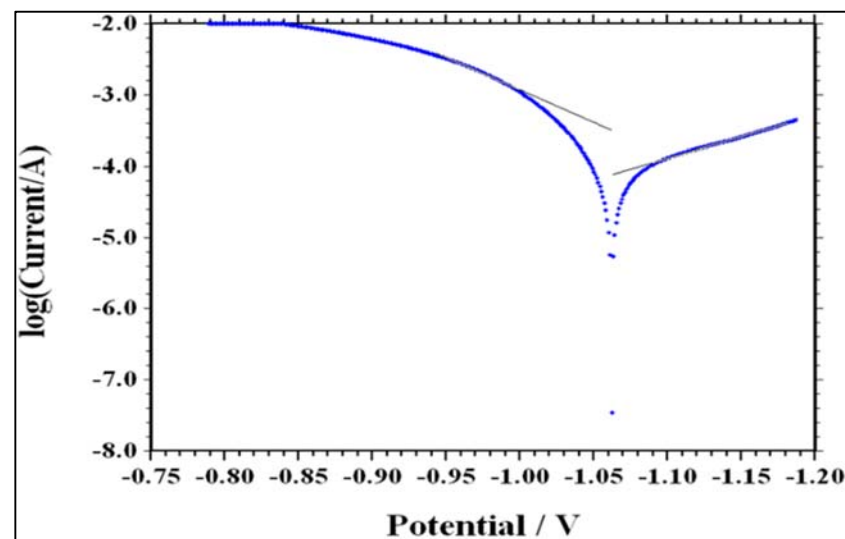


Fig 6: Polarization curve for corrosion of zinc in 0.1 M Sulphuric acid containing triethylamine.

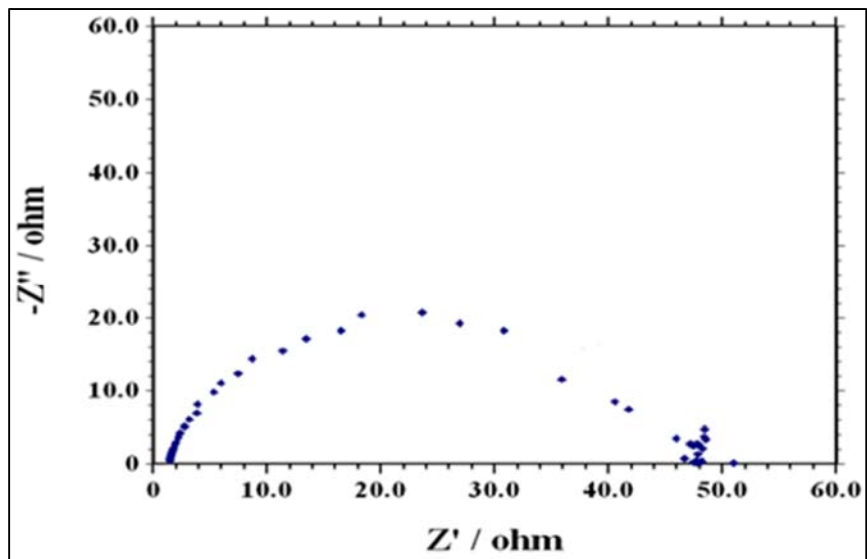


Fig 7: Nyquist plot for corrosion of zinc in 0.1 M Sulphuric acid without inhibitor.

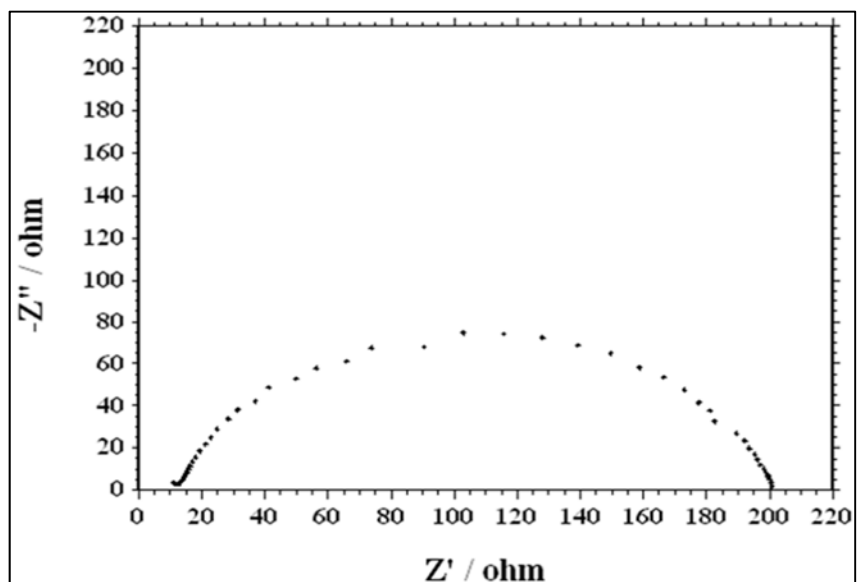


Fig 8: Nyquist plot for corrosion of zinc in 0.1 M Sulphuric acid containing 60 mM ethylamine.

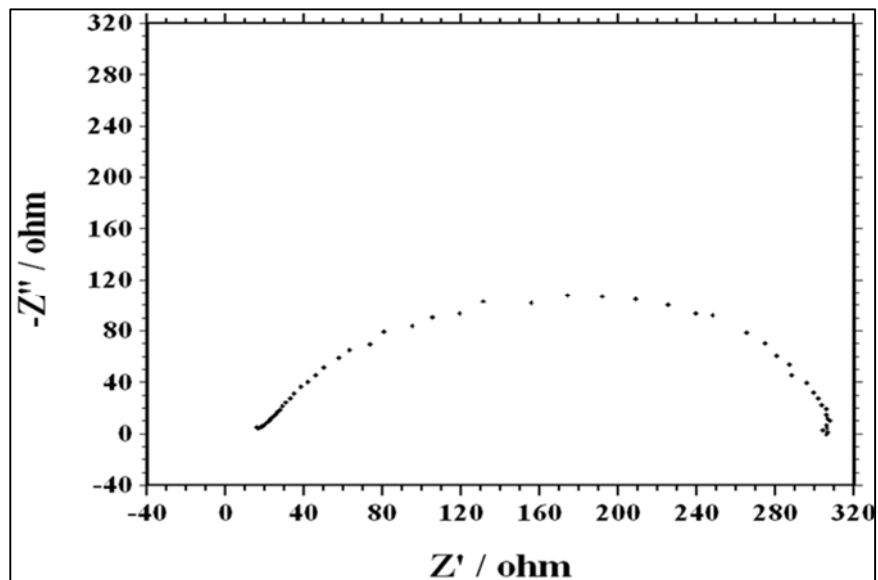


Fig 9: Nyquist plot for corrosion of zinc in 0.1 M Sulphuric acid containing 60 mM diethylamine.

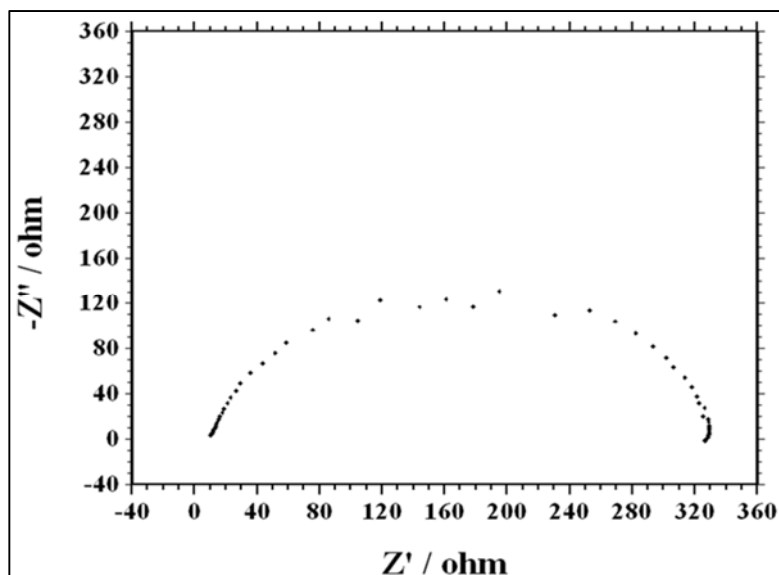


Fig 10: Nyquist plot for corrosion of zinc in 0.1 M Sulphuric acid containing 60 mM triethylamine.

Conclusion

- As the acid concentration increase the corrosion rate increases.
- At constant inhibitor concentration, the I.E. of all inhibitors increases as the concentration of acid increases. At all concentration of acid, as the inhibitor concentration increases I.E. increases and corrosion rate decreases.
- At 60 mM inhibitor concentration and 0.1 M acid concentration diethylamine shows better I.E. while ethylamine shows less effective.
- As the temperature increases corrosion rate increases while I.E. decreases in plain acid.
- In all cases, the value of heat of adsorption (Q_{ads}) and the value of free energy of adsorption (ΔG_a^0) are negative. Value of change of enthalpy (ΔH_a^0) and entropy of adsorption (ΔS_a^0) were positive.
- Mean value of 'Ea' in inhibited acid were higher than the value of 'Ea' in acid only, which shows that physical adsorption of the inhibitor molecule.
- Plot of $\log(\theta/1-\theta)$ Vs $\log C$ (inhibitor concentration) shows straight line, which indicate that the inhibitors cover both the anodic and cathodic regions through general adsorption following Langmuir isotherm.
- Results obtained from polarization method and EIS methods were in good agreement with weight loss method.

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