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Inhibition of corrosion of aluminium in alkaline medium by glutaric acid in conjunction with zinc sulphate and diethylene triamine penta (Methylene phosphonic acid)

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ABSTRACT

The inhibition efficiency (IE) of glutaric acid (GA) in conjunction with Zn^{2+} *and diethylene triamine penta (methylene phosphonic acid) [DTPMP] on the corrosion of aluminium in 0.5M NaOH was investigated by weight loss and electrochemical techniques. The ternary system GA (1000 ppm) –* Zn^{2+} *(20 ppm) - DTPMP (20ppm) has 93.4% IE. IE values calculated from electrochemical polarization studies were in good agreement with those calculated from weight loss studies.* ΔG *values were calculated from surface coverage values. Effect of temperature on the IE was also studied from which Ea and* ΔH *were computed. Surface morphology studies were done using scanning electron microscopy (SEM) and atomic force microscopy (AFM). The inhibitors act as mixed inhibitors and obey Langmuir adsorption isotherm.*

Key Words: Aluminium corrosion, Mixed type inhibitors, Glutaric acid, Adsorption isotherm, Surface morphology.

INTRODUCTION

Aluminium and its alloys are the second largest used materials next to iron and its alloys due to their lightweight, strength, durability, formability and corrosion resistance. Prevention of corrosion of aluminium has been a subject of numerous studies due to their high technological value and wide range of industrial and house hold applications. Aluminium and its alloys are very good corrosion resistant materials in neutral aqueous solution, due to the formation of passive film. It is well known that pitting corrosion occurs on metals covered with passive films. The Cl^- ions cause the passive film to break down at certain weak spots on the metal surface. During pitting corrosion large parts of the metal surface are covered with a protective film and are in the passive state, while other small parts of the surface are in the active state. Corrosion behaviour of aluminium in various media has been studied. Several inhibitors have been used to control the corrosion of aluminium. To prevent the corrosion of aluminium in acid medium, inhibitors such as imidazoline derivatives [1], Capparis decidua [2] polyethylene glycol and polyvinyl alcohol [3], delonix regia extract [4] and Sansevieria trifasciata extract [5], have been used. In alkaline medium, adipic acid-DTPMP[6], polyvinyl alcohol [7], gongronema latifolium extract [8], bismark brown dye [9], methyl orange [10], [3 – (4 – hydroxy – 3 methoxy – phenyl) – 1 – phenyl – propenone] [11], 1 – (2,4 – dihydroxy phenyl) – 3 - (2 – hydroxyl phenyl) – propenone [12], salicylideneaniline [13] and adathoda vasica leaves extract [14] have been used as corrosion inhibitors to prevent corrosion of aluminium. Corrosion inhibition of aluminium using super hydrophobic films [15], structure and stability of adhesion promoting aminopropyl phosphonate layers at polymer / aluminium oxide interfaces [16], microbially influenced corrosion of aluminium [17], corrosion inhibition of aluminium by rare earth chlorides [18], effects of inhibitors on corrosion behaviour of dissimilar aluminium alloy corrosion using fluorometric methods [19], surface modification for aluminium pigment inhibition [20] and film formed on 6000 series aluminium [21], have been investigated. Because of the voice raised by environmental scientists, several corrosion researchers have started using environmental friendly natural products as corrosion inhibitors [22-26]. Water extracts from leaves of date palm, phoenix dactylifera, henna, Lawsonia inermis, corn and Zea mays, were tested as corrosion inhibitors for steel, aluminium, copper and brass in acid chloride and sodium hydroxide solutions, using weight loss, solution analysis and potential measurements [27]. Priya et al. have studied the corrosion behaviour of aluminium in rain water containing garlic extract [28]. Jain et al. have investigated the electrochemical behaviour of aluminium in acidic media [29]. Several investigators have reported the corrosion inhibition behaviour of carbon steel and copper in acidic medium [30-33].

This paper reports the corrosion behaviour of commercial aluminium metal in 0.5M NaOH solution and the inhibition of corrosion of aluminium by glutaric acid, Zn^{2+} and DTPMP at 30^0, 40^0 and 50^0C. Weight loss method and electrochemical techniques were used to calculate the IE values. Free energy change, activation energy and enthalpy values were computed and discussed. Surface morphology of the metal surface was studied using SEM and AFM. Langmuir adsorption isotherm and El.Awady adsorption isotherm were drawn and analysed.

MATERIALS AND METHODS

Commercial aluminium specimens, of dimensions 1.0x 4.0 x 0.14 cm, containing 99% pure aluminium were polished to a mirror finish, degreased with trichloro ethylene, and used for the weight loss method and for surface examination studies. All the weighings of the aluminium specimens before and after corrosion were carried out using a Denver Electronic balance (TP214) with readability of and 0.1 mg in 200g range. This balance has reproducibility (standard deviation) of 0.1 mg. AR grade $ZnSO_4$, glutaric acid and DTPMP were used to prepare inhibitor combinations in double distilled water.

Determination of inhibition efficiency (IE)

The weighed specimens in triplicate were suspended by means of glass hooks in 150ml beakers containing 100 ml of various test solutions in 0.5M NaOH medium. After one hour of immersion, the specimens were taken out, washed in running water, dried and weighed. From the change in weight of the specimens, the corrosion inhibition efficiency (IE) was calculated using the equation:

$$IE = 100\ [1-(W_2 / W_1]\ \%$$

Where W_1 = Corrosion rate in the absence of inhibitor, and
W_2 = Corrosion rate in the presence of inhibitor.

Corrosion Rate (C_R) is calculated by using the following relationship, in mmpy unit.

$$\mathbf{C_R} = \frac{\text{Weight loss (mg) x 87.6}}{\text{Immersion Time (h) x density of the metal (g/cm}^3\text{) x area of specimen (cm}^2\text{)}}$$

$$\textbf{Surface coverage } (\boldsymbol{\theta}) = \frac{(W_1 - W_2)}{W_1}$$

Where W_1 = Corrosion rate in the absence of the inhibitor (blank)
W_2 = Corrosion rate in the presence of the inhibitor.

The effect of temperature on the IE was studied at three different temperatures, namely, 30^0C, 40^0C and 50^0C for one hour duration. **Activation energy (Ea)** values were calculated using Arrhenius equation and **Enthalpy change (ΔH)** values were calculated using the equation:

$$\Delta H = Ea - RT.$$

The free energy change of adsorption, ΔG^0_{ads}, is calculated using the equation:

$$\Delta G^0_{ads} = -\ RT \ln (55.5 \times K_{ads})$$

Where 55.5 is the molar concentration of water in solution and $K_{ads} = \theta / C\ (1-\theta)$.

Electrochemical Polarization measurements

A conventional three electrode cell consisting of Aluminium as working electrode, Platinum as counter electrode and a saturated calomel electrode as reference electrode was used for potentiodynamic polarization studies. Cyclic potentiodynamic polarization studies were carried out in VSP electrochemical analyzer and the data were analyzed by EC – Lab for windows V 9.43(Software), Internet server V 9.40 (firmware) and command interpretor V 9.42 (firmware) % I.E. was calculated by using the formula.

$$\text{IE} \quad \% = \frac{(I_0 - I_{inh})}{I_0} \text{ x } 100$$

Where, I_0 = Corrosion current in the absence of inhibitor.
I_{inh} = Corrosion current in the presence of inhibitor.

AC impedance spectroscopic studies were also carried out, using the same electrochemical analyser.

Surface Morphology studies

Scanning Electron microscopy (SEM) and Atomic Force Microscopy (AFM) were used for surface morphology studies.

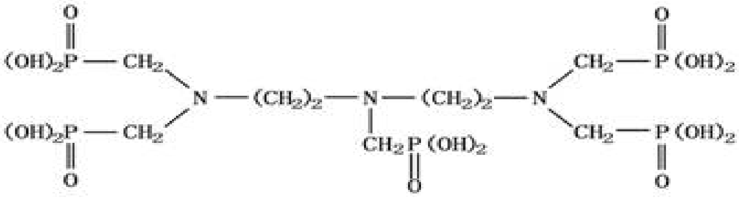
SEM images of polished Aluminium metal, Corroded Metal and Corrosion inhibited metal surfaces are recorded with VEGA 3 TESCAN microscope and compared.

Atomic force microscopy (also known as scanning force microscopy) is a powerful technique for the gathering of roughness statistics from a variety of surfaces [34]. AFM is becoming a well accepted method of roughness investigation [35-40].

All AFM images were obtained on a pico SPM 2100 AFM instrument operating in contact mode in air.

Structure of inhibitors

DTPMP



Glutaric acid

$HOOC–CH_2–CH_2{}^{-}CH_2–COOH$.

RESULTS AND DISCUSSION

Weight loss study

The percentage inhibition efficiencies (IE%) of Zn^{2+} ions, DTPMP and GA separately and in combination, have been evaluated in 0.5 NaOH medium, by weight loss technique at 30^0C and the results are summarized in Table 1 $_{(a)}$ to 1 $_{(c)}$.

Table $1_{(a)}$ illustrates the effect of concentration of GA on the IE at 30^0C. It is observed that 1000 ppm of GA has maximum IE 46%.

Table $1_{(b)}$ depicts the influence of Zn^{2+} and GA on the IE, which increases from 42% to 57.2% during increase of GA from 100ppm to 1000 ppm.

Table $1_{(C)}$ explains the combined effect of DTPMP, Zn^{2+} and GA on the IE. At an optimum constant concentration of 20ppm of DTPMP, an increasing trend of IE is demonstrated during the increase of concentration of GA, which reaches nearly 93.4% at a concentration of 1000ppm of GA.

Table 1(a)

Zn^{2+} = NIL, DTPMP = NIL Temp 30°C			
Concentration of GA (ppm)	**Corrosion Rate C_R (mmpy)**	**IE%**	**Surface Coverage θ_1**
0(Blank)	309.8	_	_
100	247	20.2	0.202
200	233	24.5	0.243
300	220	29.0	0.290
400	207	32.8	0.325
600	193.7	37.4	0.374
800	179	41.9	0.419
1000	166	46.0	0.460

Table 1 (b)

Zn^{2+} = 20 ppm, DTPMP- NIL Temp. 30°C			
Concentration of GA (ppm)	**Corrosion Rate C_R (mmpy)**	**IE %**	**Surface Coverage θ 2**
0 (Blank)	-	-	-
100	179.8	42	0.42
200	169.6	45.2	0.452
300	164.0	47.1	0.471
400	157	49.3	0.493
600	145	53.2	0.532
800	139.5	55.0	0.55
1000	132.5	57.2	0.572

Table 1 (c)

Zn^{2+} = 20 ppm ; Temp. 30°C				
Concentration (ppm)		**Corrosion Rate C_R (mmpy)**	**IE %**	**Surface Coverage θ_3**
DTPMP	**GA**			
20	100	118.1	61.9	0.619
20	200	103.4	66.6	0.666
20	300	98.1	68.3	0.683
20	400	75.3	75.7	0.757
20	600	51.5	83.4	0.834
20	800	30.8	90.1	0.901
20	1000	20.3	93.4	0.934

Electrochemical Studies

The results of the potentiodynamic polarization studies for the blank and various environments containing the inhibitor systems are summarized in Table $2_{(a)}$. IE values calculated from the polarization data are in good agreement with those obtained from weight loss data. The Tafel curves in figures 1(a) to 1(d) illustrate that the nature of the curves remains almost the same, even after the addition of inhibitors. However, on increasing the concentrations of the inhibitors, the curves are gradually shifted towards lower current Density. The corrosion potential values also are shifted towards negative direction, indicating the decrease in the rate of the cathodic reaction. Further, the decrease in the anodic and cathodic Tafel slopes βa and βc are related to the decrease in both the cathodic and anodic currents. The inhibitors act as mixed inhibitors.

Table 2 (a) - Potentiodynamic Polarization Data.

Zn^{2+} 20ppm; DTPMP 20 ppm				**Respective Figure**
GA (ppm)	**-Ecorr (V)**	**Icorr (mA)**	**IE %**	
0 (Blank)	1.58	7.556	-	1(a)
400	1.670	1.9	74.8	1(b)
800	1.658	0.78	89.7	1(c)
1000	1.600	0.48	93.7	1(d)

Table 2 (b) – Impedance Data.

Zn^{2+} 20 ppm; DTPMP – 20 ppm			**Respective Figure**
GA (ppm)	**R_{ct} (Ω cm^2)**	**C_{dl} ($\mu F/cm^2$)**	
0 (Blank)	0.876	56.3	2(a)
1000	1.74	0.1892	2(b)

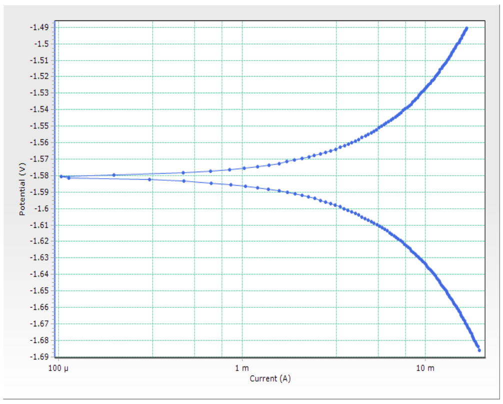


Figure 1(a) Polarization graph (Tafel)

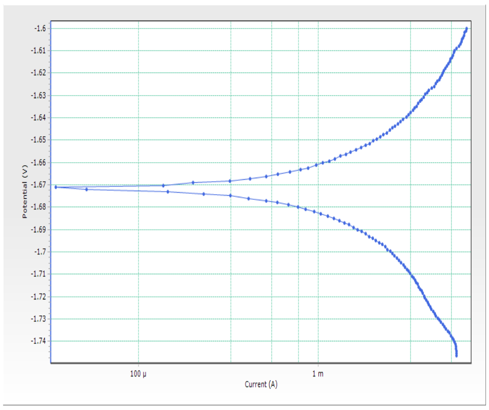


Figure 1 (b) Polarization graph (Tafel)

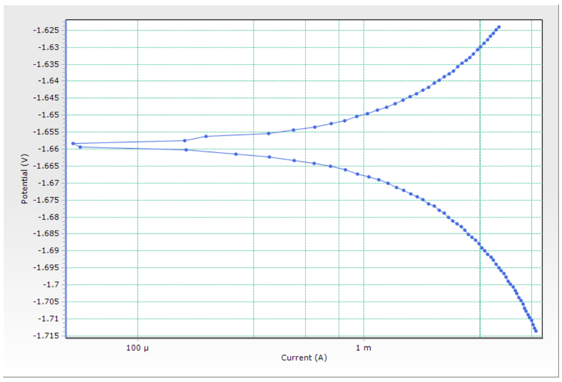


Figure 1(c) Polarization graph (Tafel)

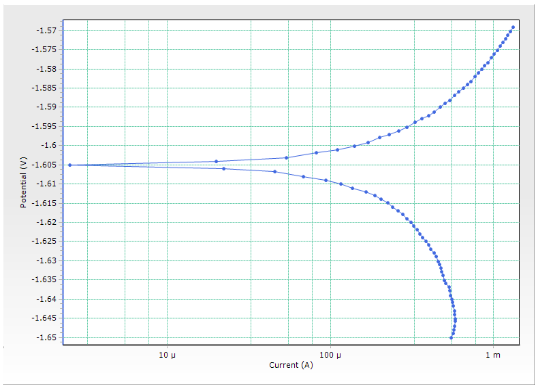


Figure 1 (d) Polarization graph (Tafel)

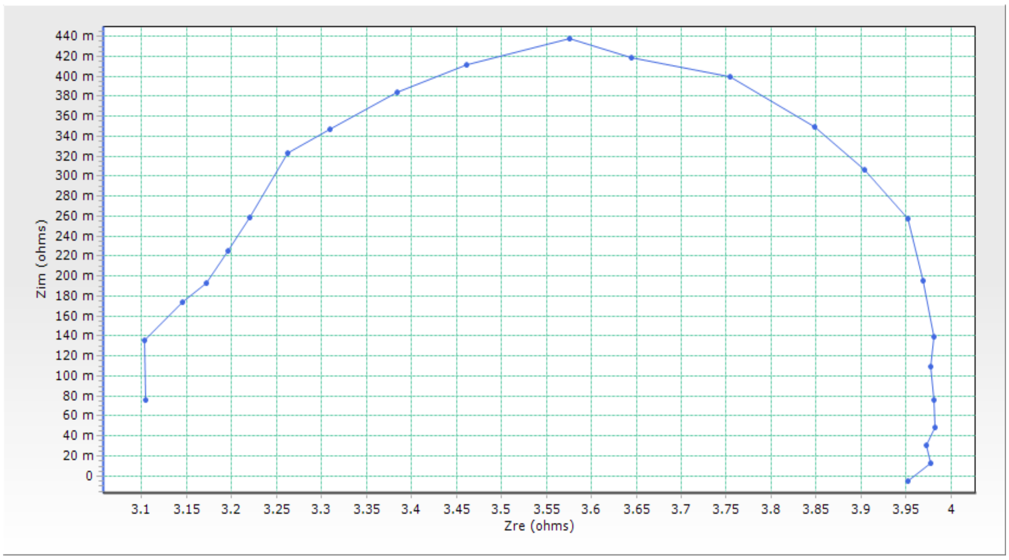


Figure 2 (a) Impedance graph (Nyquist)

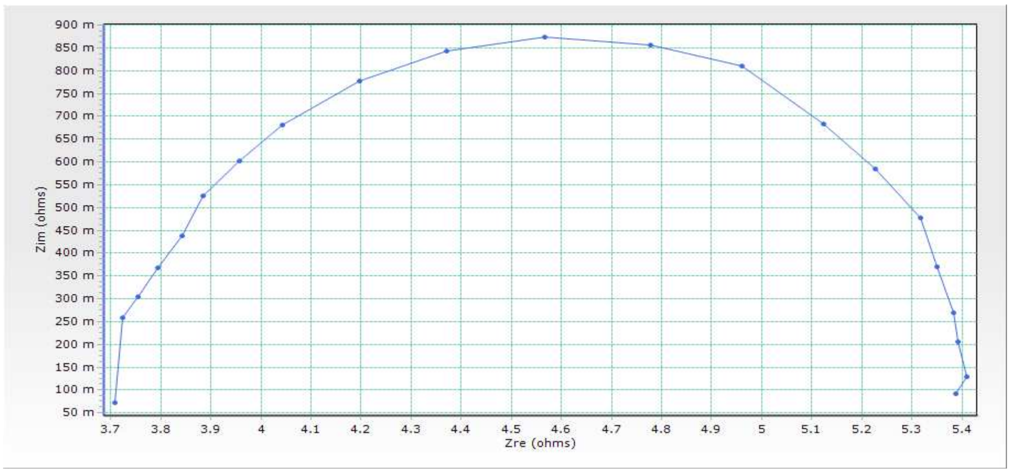


Figure 2 (b) Impedance graph (Nyquist)

The impedance Nyquist graphs are given fig 2(a) and 2(b). The R_{ct} and C_{dl} values tabulated in Table 3 (b) confirm the adsorption of the inhibitor system and formation of the layer of the inhibitor on the metal surface.

Effect of temperature on inhibition efficiency

The effects of the inhibitor systems containing 20ppm DTPMP, 20ppm Zn^{2+} and various concentrations of GA were studied through weight loss measurement at the elevated temperatures of 40^0C and 50^0C also and the results are presented in Table 3(a). The IE values decrease with the rise of temperature.

Table 3(a). Effect of temperature on IE

Concentration of GA (ppm)	Zn^{2+} = DTPMP = 20 ppm					
	IE % at 30^0C	C_R at 30^0C	IE% at 40^0C	C_R at 40^0 C	IE% at 50^0C	C_R at 50^0 C
Blank	-	310	-	727	-	942
100	61.9	118	50.6	360	43.2	535
200	66.6	103	56.0	319	49.2	479
300	68.3	98	60.8	285	55.7	417
400	75.7	75	68.0	233	62.1	357
600	83.4	51.5	76.2	173	68.9	293
800	90.1	30.8	80.3	143	74.3	242
1000	93.4	20.3	83.2	135	77.3	214

Activation energy (Ea) values were calculated from corrosion rate data at different temperatures and from the Ea values, Enthalpy change (ΔH) values for the corrosion process were calculated and given in Table 3(b).

Table 3 (b) Activation Energy and Enthalpy Changes

Process	Average Ea (KJ/mol)	Average ΔH (KJ / mol)
Corrosion in the absence of Inhibitors	44.77	42.17
Corrosion in the Presence of inhibitors (GA =1000ppm)	94.84	92.24

More positive the values of Ea and ΔH, more is the endothermic nature of the corrosion process and therefore, less spontaneous is the corrosion process. In other words, higher values Ea and ΔH indicate slowing down of corrosion process. i.e. Corrosion is decreased in the presence of inhibitors (Refer Table 3b).

Adsorption Isotherms

The surface coverage values (θ) calculated from weight loss measurements were fitted into the Langmuir adsorption isotherm.

The Langmuir adsorption isotherm model has the form $C/\theta = 1/K_{ads} + C$.

Where C is the concentration, K_{ads} is the equilibrium Constant of adsorption process. A plot of C/θ Vs C is linear (Fig. 4a). The slope deviates from unity indicating that there is attraction or repulsion in the adsorbed layer of inhibitor on the aluminium surface [41-42]

El.Awady adsorption isotherm is expressed as:

$$\log(\Theta/1-\Theta)=\log K+ y \log C.$$

Here, the $K_{ads,}$ the equilibrium constant of adsorption process is calculated by the relation $K_{ads}=k^{1/y}$. A plot of log (Θ/1- Θ) Vs log C is linear but deviates from linear nature at lower concentrations (Fig. 4b) and therefore it is concluded that the inhibition process follows Langmuir adsorption isotherm rather than El. Awady isotherm.

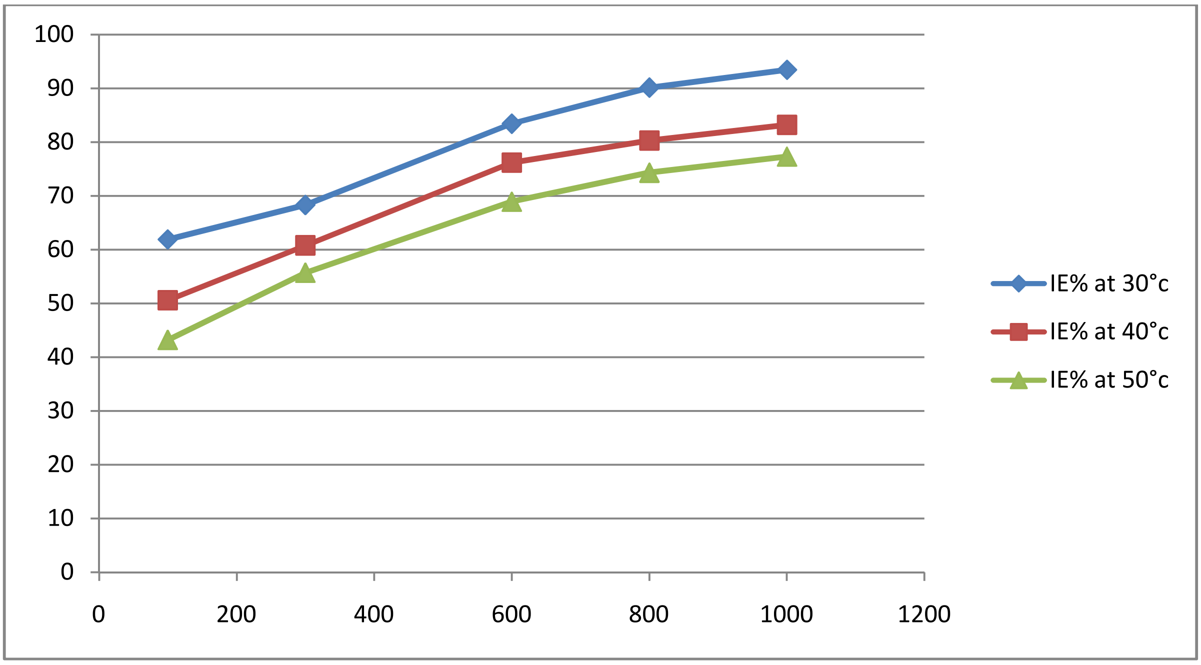


Fig. 3 Effect of concentration of inhibitor and temp. on IE% [IE% Vs GA (ppm)]

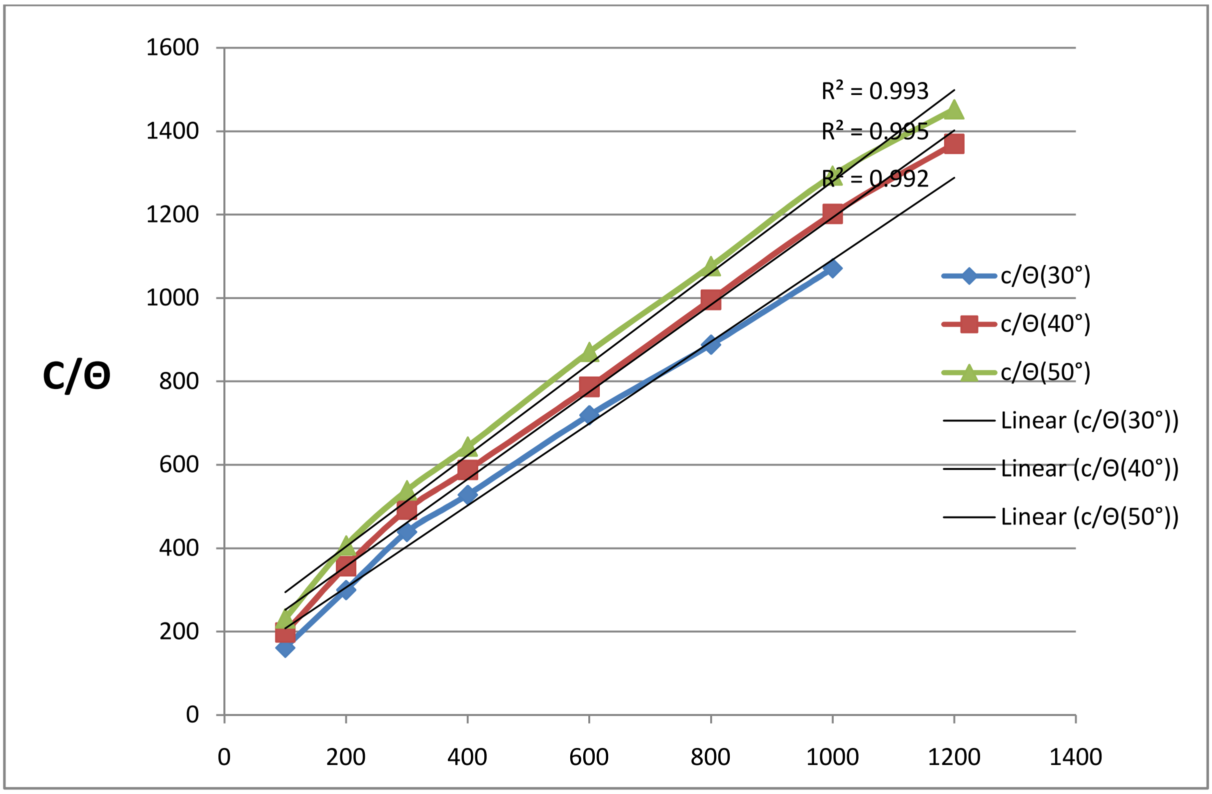


Fig. 4(a) LANGMUIR ADSORPTION ISOTHERM [C/Θ Vs C]

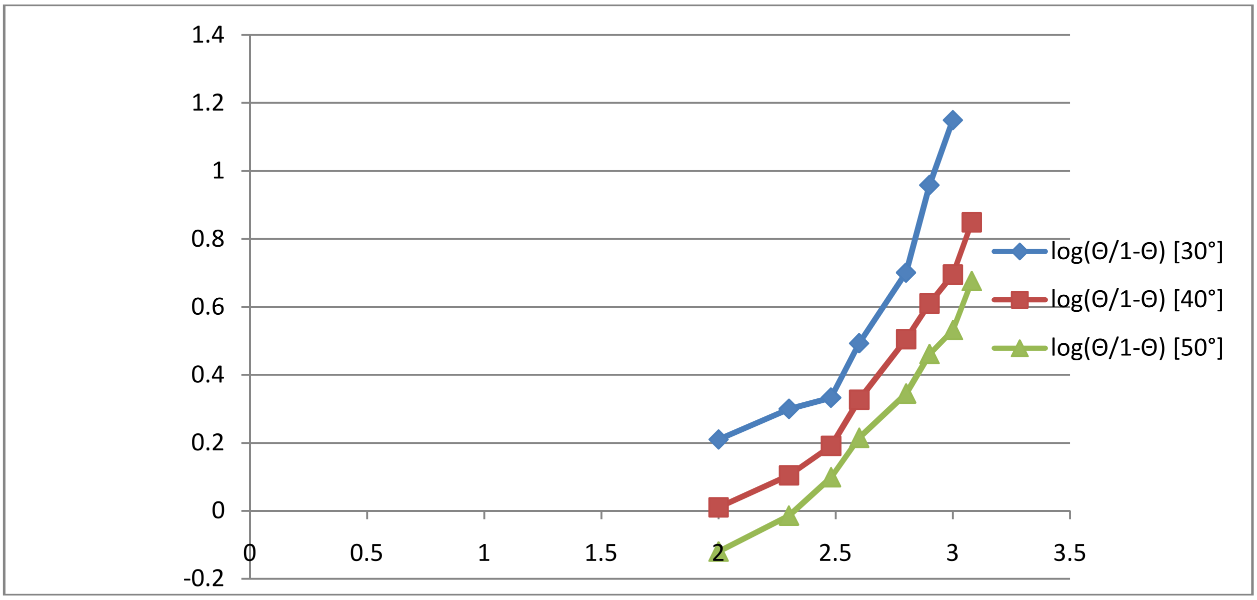


Fig 4(b) El.AWADY ADSORPTION ISOTHERM [log (Θ/1-Θ) Vs log C]

From the K_{ads} values, calculated using the θ values for various concentrations of inhibitors at different temperatures, ΔG^0ads values were also calculated and tabulated in Table 4.

Table: 4. Free energy of adsorption (ΔG^o_{ads}) values (KJ / mol^{-1})

GA (ppm)	← Additives added to GA →				
	DTPMP = Nil; Zn^{2+} = Nil	DTPMP = Nil; Zn^{2+} = 20 (ppm)	DTPMP = 20 ppm ; Zn^{2+} = 20 ppm.		
	at 30°C	at 30°C	at 30°C	at 40°C	at 50°C
100	-25.04	- 27.41	- 29.45	-29.25	- 29.19
200	-23.64	- 25.99	- 28.22	-27.97	-27.84
300	-23.61	- 25.16	- 28.39	-27.43	-27.35
400	-22.93	- 24.66	- 28.59	-27.90	-27.69
600	-22.41	- 24.03	- 28.78	-27.21	-27.01
800	-22.16	- 23.49	- 28.55	-27.40	27.35
1000	-22.02	-23.15	-29.10	-27.32	-27.19

The values of ΔG^0_{ads} are negative and range from – 22.02 to – 29.45 KJ mol^{-1}. The negative values indicate that the adsorption of the inhibitors on the aluminium surface is spontaneous.

The ΔG^0_{ads} values decrease with the increase of temperature suggesting that there is a decrease in adsorption of the inhibitors as the temperature increase from 30 to 50°C.

In general, the values of ΔG^0_{ads} around – 20 KJ mol^{-1} correspond to physisorption while those above -40 KJ mol^{-1} correspond to chemisorption. [43, 44].

Analysis of SEM images

The SEM images of the aluminium metal surface immersed in 0.5M NaOH solution and in the combined inhibitor system are given in Fig 5(a) & 5(b), respectively.

A comparative examination of these images, clearly suggest that the surface of Aluminium specimen is smoothened to a very large extent in the presence of the combined inhibitor system. This smoothening might be due to the adsorption of the inhibitor molecules on it and thus the surface is fully covered.

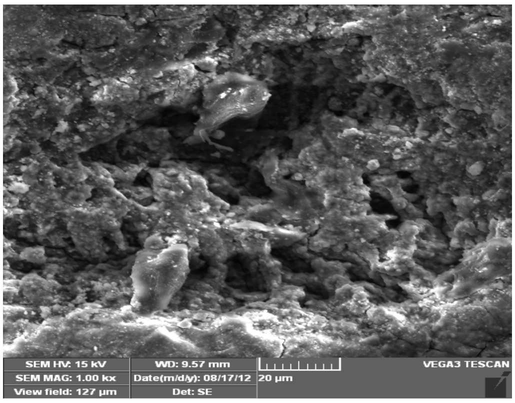


Figure 5(a) SEM - Al surface in 0.5 NaOH (blank)

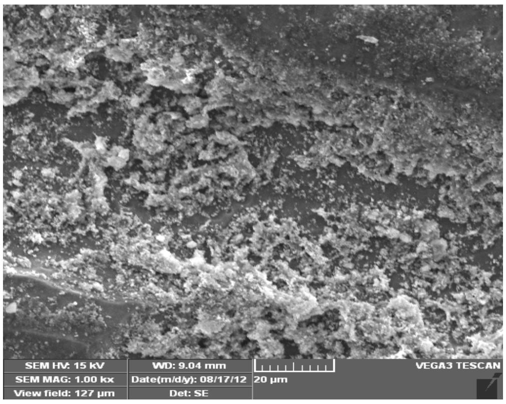
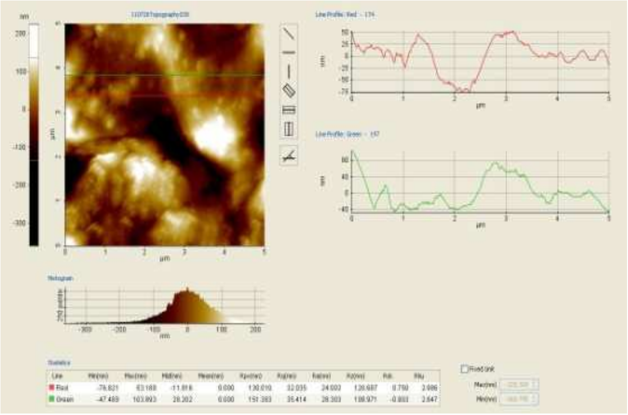


Figure 5(b) SEM- Al surface in the presence of inhibitors



Statistics

Line	Min(nm)	Max(nm)	Mid(nm)	Mean(nm)	Rpv(nm)	Rq(nm)	Ra(nm)	Rz(nm)	Rsk	Rku
🟥 Red	-76.831	63.188	-11.816	0.000	130.010	32.035	24.003	120.687	0.790	2.086
🟩 Green	-47.489	103.893	28.202	0.000	151.383	35.414	28.303	108.971	-0.803	2.647

Figure 6(a) AFM image

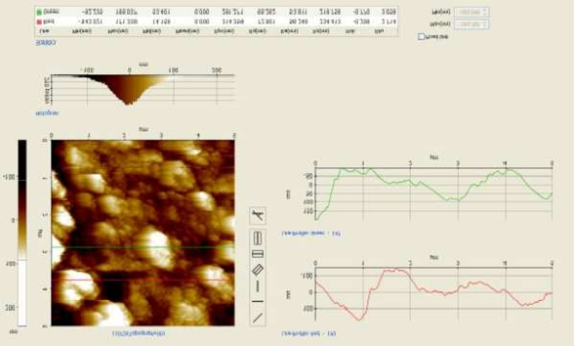
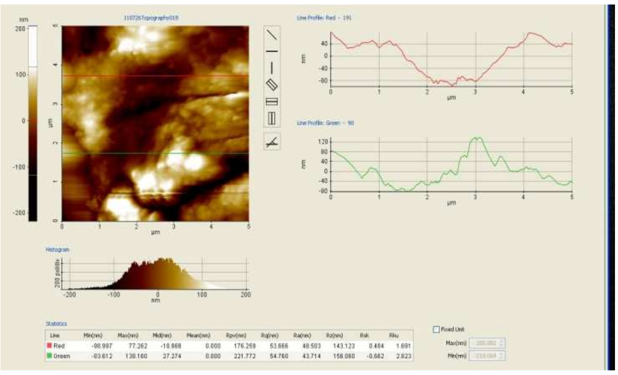


Figure 6 (b) AFM image



Statistics

Line	Min(nm)	Max(nm)	Mid(nm)	Mean(nm)	Rpv(nm)	Rq(nm)	Ra(nm)	Rz(nm)	Rsk	Rku
🟥 Red	-98.997	77.262	-10.868	0.000	176.259	53.666	48.503	143.123	0.484	1.691
🟩 Green	-83.612	138.160	27.274	0.000	221.772	54.760	43.714	158.060	-0.662	2.823

Figure 6 (c) AFM image

Analysis of AFM images

Fig 6(a), 6(b) and 6(c) show the AFM images of polished metal, corroded surface in the absence of inhibitors and the smoothened surface in the presence of inhibitors, respectively. Table 5 gives the corresponding roughness and peak-to-valley height values.

Table-5 - AFM roughness data

Sample	RMS(Rq) Roughness (nm)	Average roughness(Ra) (nm)	Maximum peak-to-valley height (P-V) (nm)	Respective Figure
Polished Al metal	32	24	130	5 (a)
Al immersed in 0.5M NaOH solution	72.6	56.2	314	5 (b)
Al immersed in combined inhibitor system	41	33	182	5 (c)

A comparative view of the above roughness table clearly demonstrates that the surface of the metal is smoothened due to the adsorption layer formed.

CONCLUSION

i. GA has a maximum of 46% IE at 30^{0}C for alumiuium in 0.5M NaOH medium. In the presence of 20ppm Zn^{2+}ions, the IE of GA shifts to a maximum of 57.2% at 1000 ppm concentration. The ternary system consisting of 20ppm DTPMP, 20ppm Zn^{2+} and 1000ppm GA has a remarkably high IE, 93.4%.
ii. The IE% decreases with increase of temperature to 40^{0}C and 50^{0}C.
iii. The IE Values calculated from electrochemical polarization studies are in good agreement with those of weight loss studies. Electrochemical polarization studies show that the inhibitor system acts as a mixed inhibitor.
iv. Observations made from SEM, AFM and Langmuir adsorption isotherm suggest that the corrosion inhibition occurs through the adsorption of inhibitors on the surface of aluminium.
v. The negative values of ΔG indicate that the adsorption of the inhibitors on the aluminium surface is spontaneous. Ea and ΔH values of the corrosion process also support this observation.

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