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The Corrosion Inhibition of Carbon Steel by Lauric Acid –Zinc Ion System in Neutral Aqueous Media

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Abstract

Lauric acid (LA) in the presence of Zn^{2+} ions is used to function as the corrosion inhibitors to control corrosion of carbon steel. The co-operative effect between LA and Zn^{2+} has been investigated by corrosion mass-loss technique. It is observed that the formulation consisting 25ppm Zn^{2+} and 175ppm LA in 60ppm Cl^- neutral aqueous environment, as the immersion period increases in the growth of protective film on the metal surface also increases. Polarization study indicates that LA an Zn^{2+} system acts as a cathodic inhibitor; UV-visible spectral study indicates that were possibility formation of complexes of LA and Zn^{2+} with carbon steel iron. FTIR spectral study confirmed the formation of protective film on the metal surface. The adsorption of this inhibitor is also found to obey Langmuir adsorption isotherm. The inhibitor system has 80% biocidal efficiencies.

Keywords: *Alloys; adsorption; mass-loss study; polarization study; FTIR study.*

1. INTRODUCTION

Carbon steel has many industrial applications because of its easy availability, low cost, uncomplicated fabrication into water pipe lines, cooling water systems (Saha *et al*. 1955), boilers etc. Corrosion, scale and fouling problems can appear when water containing chloride is used as thermal fluid. This problem can occur jointly, reducing the thermal efficiency of the cooling circuit with the significant social-economic repercussions. To avoid or minimize these problems, water used in cooling circuit is treated with inhibitive formulations. Inhibitors are generally used in these processes to control the metal dissolution. Organic compounds are recognized as the effective inhibitor of the corrosion of many metals and alloys. The efficiency of this inhibitor is closely associate with the chemical adsorption (Spinelli *et al*. 1990; Oliver *et al*. 1992; Vendrame *et al*. 1988; Melloo *et al*. 2001; Lucho *et al*. 2002). Most of the well-known inhibitors are organic compounds containing N, S, and O atoms, and multiple bond in the molecule which are adsorbed on the metal surface and heterocyclic compounds (Martinez *et al*. 2002; Manov *et al*. 2002). This adsorption phenomenon is influenced by nature and surface charge of metal, by the type of aggressive electrolyte and by chemical structure of inhibitors.

The aim of this paper is to study the co-operative effect of LA and Zn^{2+} ion. The electrochemical behavior of carbon steel in neutral aqueous environment containing 60ppm Cl^- in the absence and presence of inhibitor, to investigate the influence of immersion period on the protective efficiency of the system; to study the possibility of formulation of complexes; to analysis the protective film by FTIR spectra; and to propose the mechanism of co-operative effect based on the above result.

2. MATERIAL AND METHODS

2.1. Preparation of Specimen

Carbon steel strip specimens of size 1.0cm×4.0cm×0.2cm containing 0.026%S, 0.06%P, 0.4%Mn,0.1%C,and the remainder iron, were used for mass-loss measurement, strips were polished to a

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mirror finish, degreased with acetone, and used for the mass- loss method and surface examination studies.

2.2. Preparation of Solution

The aggressive chloride solution used were prepared using AR grade NaCl. Appropriate concentrations of solutions were prepared using bidistilled water. The concentration range of the employed was 10-175ppm.

2.3. Mass-loss Method

The mass-loss of rectangular carbon steel immersed in 60ppm Cl^- neutral aqueous environment, with and without addition of different concentrated Zn^{2+} and LA was determined for 7 days at a temperature of 30^{0C}, in air atmosphere the masses of specimens before and after the immersion period were determined using a digital balance (model AUY 220 SHIMADZU). The corrosion products were cleaned with Clarke's solution. The co-operative effect (CE) was then calculated using the equation

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

Where W_1 is the mass loss value in the absence of inhibitor and W_2 is the mass loss value in the presence of inhibitor. Corrosion rate (in mils per year) was calculated using the formula

CR=534W/DAT

W=mass loss in milligrams

D=density of specimen =7.87gcm^{-3}

A=area of specimen=10.4cm^2

T=exposure time in hours=168hrs.

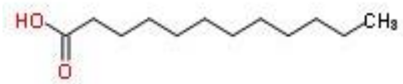


Fig. 1: Molecular structure of Lauric acid

2.4. Surface Examination Study

The carbon steel specimens were immersed in various test solutions for a period of one day. After one day, the specimens were taken out and dried. The nature of the film formed on the surface of the metal specimen was analyzed by various surface analysis techniques.

2.4.1. Potentiodynamic Polarization Study

For Potentiodynamic polarization studies, mild steel strips of same composition, coated with commercially available lacquer (lakame) with an exposed area 10 cm^2 were used and the experiments were carried out at temperature (28 ± 2 ºc). Equilibrium time leading to study state of the specimens was 30 min. scan rate in Potentiodynamic experiment was 1mV/s. This study was carried out using CHI 660A electrochemical impedance analyzer model a three electrode cell assembly was used. The angular specimen of carbon steel with one face of the electrode.

A saturated calomel electrode (SCE) was used as reference electrode. A rectangular platinum foil was used as the counter electrode. Polarization curves were recorded after doing IR compensation. The result such as Tafel slopes, I_{corr} and E_{corr} values were calculated.

$$CE= (I_0-I_{inh}) / I_0 \times 100$$

Where I_0 and I_{inh} are corrosion current densities in the absence and presence of inhibitor, respectively.

2.4.2. Fourier transforms infrared spectra

These spectra were recorded in a perkin-elmer-1600 spectrophotometer using KBr pellets. The FTIR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr and making the pellet.

2.4.3. UV-visible absorbance spectra

The instrument model lambda 35 was used for recording UV-visible absorbance spectra.

2.5. Determination of biocidal efficiency of the system

The biocidal efficiency of LA in the presence of 25ppmZn^{2+} and 60ppm Cl^- formulation were determined.

The Zobell medium was prepared by dissolving 5g peptone, 1g yeast extract, 0.1g Potassium dihydrogen phosphate and 15g agar-agar in one litre of triple-distilled water. The medium was sterilized by applying 15 pounds per square inch for 15 minutes in an autoclave.

The various concentrations of LA, Viz., 10ppm, 25ppm, 50ppm, 75ppm, 125ppm, 150ppm and 175ppm were added to the formulation consisting of the inhibitor system, the polished and de-greased carbon steel specimens in triplicate were immersed in these environments for a period of seven days. After seven days, 1ml each of the test solution from environments was pipetted out into sterilized petri dishes each containing about 20ml of the sterilized Zobell medium. The petri dishes were then kept in a sterilized environment inside the laminar flow system fabricated and supplied by CEEERI – Pilani, for 24hours. The total viable hetero tropic bacterial colonies were counted using a bacterial colony counter.

The CE of the formulation consisting of the inhibitor in the presence of various concentrations of LA was determined in the same way as mass loss study.

3. RESULTS AND DISCUSSION

The CE% of LA-Zn^{2+} systems are given in Table 1. It was found that the CE% increases as the concentration of LA increases. Similarly, increase of concentration of Zn^{2+} also increases CE%. A CE exists between LA and $Zn^{2+.}$ For example, 175ppm of LA alone has 28% CE and 25ppm of Zn^{2+}alone has 23% CE. However, interestingly the formulation consisting of 175ppm LA and 25ppm Zn^{2+} has 94% CE. The above formulation shows better CE than the individual inhibitor (Thangavelu *et al.* 2011).

Table1 gives the values for CE% obtained from mass loss method and the corresponding corrosion rates of LA-Zn^{2+} systems for 7 days

Table1: The corrosion co-operative efficiencies and the corresponding corrosion rates of LA-Zn^{2+} systems.

Conc. of lauric acid (ppm)	CR=534W/DAT (mpy)×10^{-4}	CE (%)	Surface coverage θ
Blank	8.6990	-	-
10	3.3398	61.60	0 .6160
25	2.8737	66.96	0.6696
50	2.0194	76.78	0.7678
75	1.9417	77.67	0.7767
125	1.5534	82.21	0.8221
150	1.3203	84.82	0.8482
175	0.5436	93.75	0.9375

3.1. Synergism parameter(S_I)

The synergism parameters or CE parameters are indications of CE. Synergistic effect is existing between inhibitors. The synergistic effect existing between Zn^{2+} of concentrations 15ppm and 25ppm and various concentrations of LA is found to be greater than 1. The results are given in Table2.

$$S_I=(1-I_{1+2})/(1-I'_{1+2})$$

Where

$I_{1+2}=(I_1+I_2)-(I_1 \times I_2)$

I_1 = surface coverage of inhibitors (LA)

I_2 = surface coverage of inhibitors (Zn^{2+})

I'_{1+2}= combined surface coverage of inhibitors (LA) and (Zn^{2+})

Surface coverage = (CE/100) (%)

I_2 for Zn^{2+}(15ppm) = 0.11 and I_2 for Zn^{2+}(25ppm) = 0.23

Table 2: Synergism parameter (S_I)

Conc. of Lauric acid (ppm)	Zn^{2+} (15ppm)			Zn^{2+} (25ppm)	
	I_1	I'_{1+2}	S_I	I'_{1+2}	S_I
10	0.22	0.57	1.6144	0.61	1.5640
25	0.28	0.60	1.602	0.66	1.6779
50	0.31	0.64	1.7058	0.76	2.2881
75	0.35	0.67	1.7530	0.77	2.2413
125	0.38	0.69	1.7800	0.82	2.6835
150	0.41	0.70	1.7503	0.84	2.9927
175	0.44	0.76	2.0766	0.93	6.8992

3.2. Analysis of variance (ANOVA-F-test)

To investigate whether the CE existing between two inhibitors statistically significant, F-test was used. If F-value was above 0.0500 for 1, 12degrees of freedom, the CE proved below 0.0500 for 1, 12 degrees of freedom, it was statistically insignificant at a 0.05 level of significance. The results are given in Table 3.

Table 3: Distribution of F value between the inhibition efficiencies of the influences of LA-Zn^{2+} (15) ppm

Source of variance	Sum of square	Degree of Freedom	Mean square	F	Level of P
Between	3584	1	3584	71.476	0.050
Within	601.714	12	50.143		

Table 3a: Distribution of F value between the inhibition efficiencies of the influences of LA-Zn^{2+} (25 ppm)

Source of variance	Sum of square	Degree of Freedom	Mean square	F	Level of P
Between	6428	1	6428	72.500	0.050
Within	1062.85	12	88.57		

The obtained F-value of 71.476 for 15ppm of Zn^{2+} was statistically significant. Therefore, it was concluded that the influence of 15ppm of Zn^{2+} on the inhibition efficiencies of various concentrations of LA was statistically significant.

The obtained F-value of 72.581 for 25ppm of Zn^{2+} was statistically significant. Therefore, it was concluded that the influence of 25ppm of Zn^{2+} on the inhibition efficiencies of various concentrations of LA was statistically significant.

3.4. Influence of immersion period on the CE of LA (175ppm)-Zn^{2+} (25ppm) system

The influence of immersion period on the CE of LA (175ppm)-Zn^{2+} (25ppm) is shown in Table 4.

Table 4: Influence of immersion period on the CE of LA (175ppm) –Zn^{2+} (25ppm) system

Immersion period, Days	Blank (CR/ mpy) × 10^{-4}	60ppmCl+LA175ppm + Zn^{2+}25ppm (CR/ mpy) × 10^{-4}	CE %
1	2.6097	0.5436	98
3	1.7941	0.4433	97
5	1.2178	0.2609	95
7	9.8641	0.5825	94

It was found that, as the immersion period increases, the CE decreases due to the fact that, as the period of immersion increases, the protective film Fe^{2+}-LA complex formed on the metal surface is broken by the continuous attack of other ions present in the environment. A similar behavior was observed corrosion prevention of carbon steel.

3.5. Analysis of Potentiodynamic polarization study

The Potentiodynamic polarization curves of carbon steel immersed in various environments are given in Fig 2a, 2b, 2c, 2d. The corrosion parameters are given in Table 5. When carbon steel is immersed in aqueous environment containing 60ppm Cl^- , the corrosion potential (E_{corr}) is -383.98mV and the corrosion current is 46.8μA. when 25ppm Zn^{2+} 25ppm LA and 25ppm Zn^{2+} , 50ppm LA and 25ppm Zn^{2+} ,150ppmLA are added, the corrosion potential shifts to the cathodic region (-383.98mV to -446.41mV, -460.82mV, -471.48mV). The corrosion current decreases to 18.2μA, 13.8μA, 8.2μA. the shift to cathodic region indicates that in the presence of LA-Zn^{2+} system, cathodic reaction namely, dissolution of iron is controlled and also CE were calculated and these values are good agreement with mass-loss measurements.

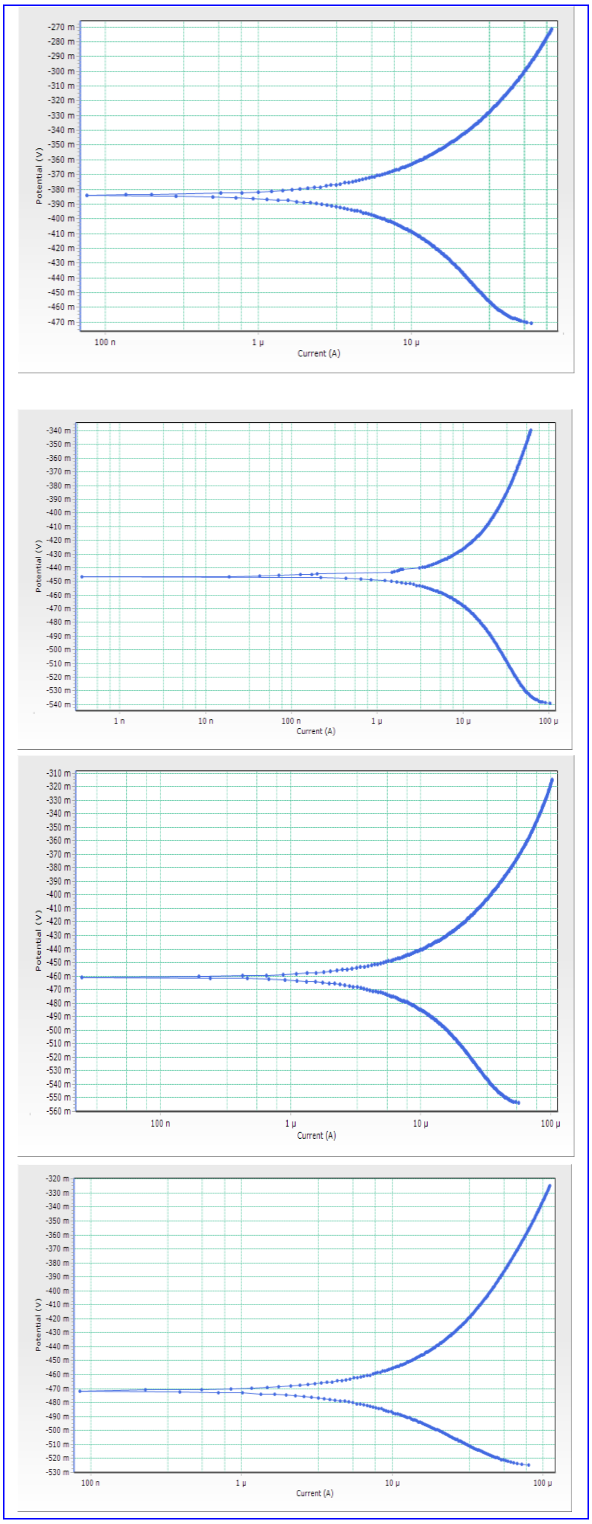


Fig. 2: Poarization graph (Tafel)

Table 5: Corrosion Parameters of Carbon Steel in Neutral Aqueous Environment in Presence and absence of inhibitor obtained by the polarization Study

Conc. of Zn^{2+} (ppm)	Conc. of LA (ppm)	E_{corr} (mV)	βa (mV)	βc (mV)	I_{corr} (μA)	CE %
-	-	-383.9	139.88	184.2	46.8	-
25	25	-446.4	111.32	99.91	18.2	61
25	50	-460.8	228.88	388.74	13.8	71
25	150	-471.4	192.58	117.08	8.2	83

3.6. Analysis of UV-visible absorbance spectra

UV- visible absorption spectra can be used to confirm the possibility of formation protective film on the metal surface (Thangavelu *et al.* 2011; Rajendran *et al.* 2012). UV- visible absorption spectrum of LA, Zn^{2+} and 60ppm Cl^- is shown in fig.3a. The peak appears at 203nm (1.46) (the peak intensity is given in parentheses) Fe^{2+}-LA-Zn^{2+} complex in solution was prepared by mixing LA solution and Fe^{2+} ion (as $FeSO_4.7H_2O + H_2O$ freshly prepared). Its UV-visible absorption is shown in fig.3b. Peak appears at 215(0.238)nm. The increase of absorbance suggests that there is a possibility of formation of Fe^{2+}-LA complex.

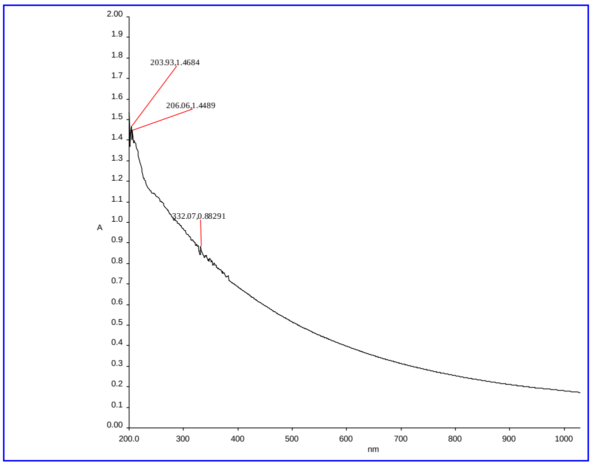


Fig. 3a: UV-visible Adsorption Spectrum of LA + Zn^{2+}+60ppmCl^-

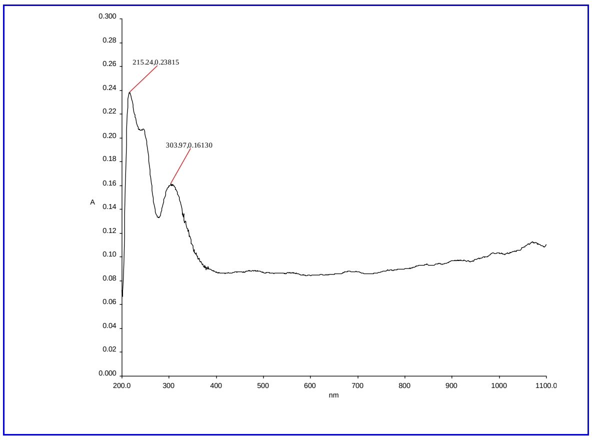


Fig. 3b: UV-visible Spectrum of 175ppm LA+ 25ppmZn^{2+} +60ppm Cl^-+100ppmFe^{2+}

3.7. Analysis of FTIR spectra

Earlier researchers have confirmed that FTIR spectrometer is a powerful instrument that can be used to determine the type of bonding for organic inhibitor adsorbed on the metal surface (Lalitha *et al.* 2005).

FTIR spectrum of pure LA shown in fig.4a.The FTIR spectrum of film formed on the metal surface after immersion in the neutral aqueous environment containing 60ppm Cl^-, 175ppm LA, and 25ppm Zn^{2+} for 7days is shown in fig.4b. The C=O stretching frequency has shifted from 1633cm^{-1} to 1631cm^{-1} due to the oxygen atoms C=O group has coordinated with Fe^{2+} formed on metal surface, resulting the formation of Fe^{2+} -LA complex on the anodic sites of the metal surface. The peak at 1402cm^{-1} is due to Zn-O stretching. The stretching frequency at appears at 3432cm^{-1} due to –OH. Therefore it is concluded that $Zn(OH)_2$ is formed on cathodic sites of metal surface (Silverstein *et al.* 2007).

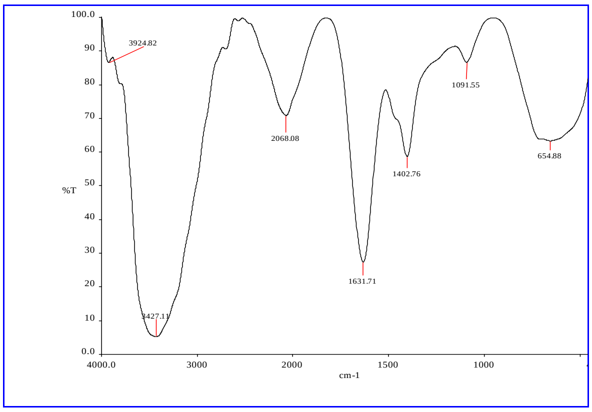


Fig. 4a: FTIR spectrum of pure LA

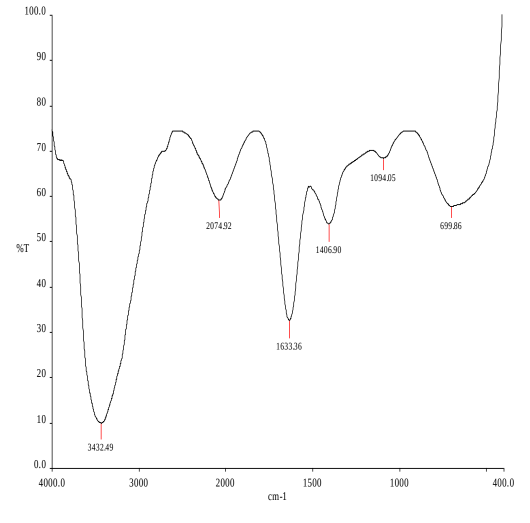


Fig. 4b: FTIR spectrum of 175ppmLA + 25ppm Zn^{2+} + 60ppm Cl^-

3.8. Adsorption isotherm

The adsorption of LA on the metal surface can occur either directly on the basis of donor-acceptor interaction between the π-electrons of the aliphatic carboxylic acid and the vacant d-orbital's of iron surface atoms or the interaction of LA with already adsorbed chloride or sulfate ions. The specific adsorption anions having the smaller degree of hydration, such as chloride ions, are expected to be more pronounced. Being specifically adsorbed they create an excess negative charge towards the solution and favour more adsorption of the cations.

Strong adsorption of organic molecules is not always a direct combination of the molecule with the metal surface. In some cases, the adsorption can occur through already adsorbed chloride or sulfate ions, which interfere with the adsorbed organic molecules. The molecular structure of the organic compound is important in synergistic inhibition.

The greatest synergistic inhibition is to be expected for an anion-cations pair in which both ions have appreciable tendencies toward covalent binding. The lesser interference by sulfate ions may lead to lower adsorption and inhibition of corrosion (Murukawa *et al*. 1964).

The degree of surface coverage (θ) for different concentrations of inhibitors in neutral aqueous media have been evaluated from mass loss measured using this equation.

$$\theta = W_{\theta=0} - W_{\theta} / W_{\theta=0} - W_{\theta=1}$$

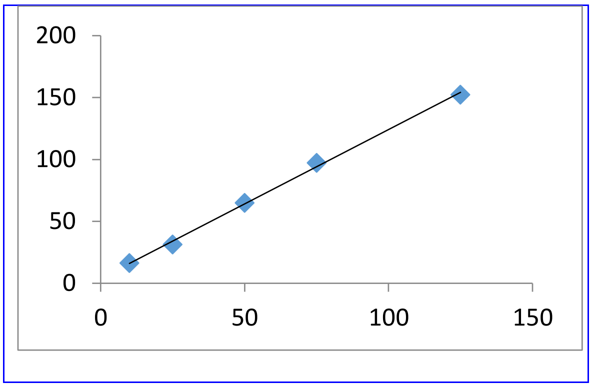
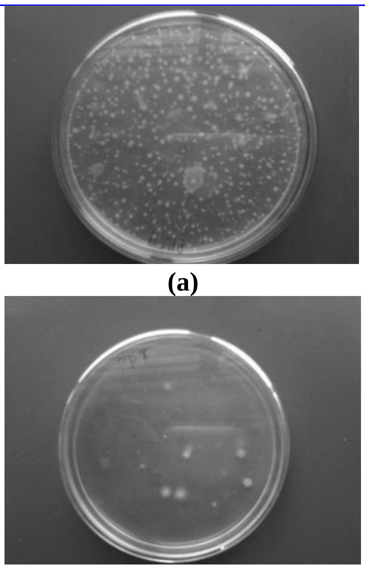


Fig. 5: Langmuir adsorption plots for carbon steel in 60ppm Cl^- containing different concentration of LA-Zn^{2+} : mass-loss measurements

The surface coverage values (θ) were tested graphically for fitting a suitable adsorption isotherm. The plots of C_{inh}/θ versus C_{inh} yields a straight line, providing that the adsorption of the LA-Zn^{2+}solutions on the carbon steel surface obeys the Langmuir isotherm.

3.9. Biocidal efficiency

It is observed that the systems studied have excellent biocidal efficiency. The formulation consisting of 60ppm Cl^- 25ppm Zn^{2+} and 175ppm LA has 94% CE and 80% biocidal efficiency, which is shown in the Table(6). The control and inhibitor system are shown in fig.5a and fig.5b. In control system, more number of bacterial colony are since compared with inhibitor system.



(a)

(b)

Fig. 5: a) Control, b) After inhibition

Table 6: Biocidal efficiency of LA on 25ppm Zn^{2+} +60ppm Cl^- system

Conc. of Lauric Acid (ppm)	Bulk bacterial Count×10^8/ml	Biocidal Efficiency	CE %
Control	30	-	-
10	18	38	61.60
25	17	41	66.96
50	14	52	76.78
75	12	58	77.67
125	9	69	82.21
150	8	72	84.82
175	6	80	93.75

4. MECHANISM OF INHIBITION

From the results obtained from electrochemical and mass-loss measurements, it was concluded that LA inhibited the corrosion of carbon steel in aqueous environment by adsorption at carbon steel / solution interface.

It is general assumption that the adsorption organic inhibition at the metal surface interface is the first step in the mechanism of the inhibitor action. Organic molecules may be adsorbed on the metal surface in four types namely,

1) Electrostatic interaction between the charged molecules and the charged metal,
2) Interaction of unshared electron pair in the molecule with the metal,
3) Interaction of π electrons with the metal and ,
4) A combination of types (i-iii) (Schweinsberg *et al*. 1988; Shorky *et al*. 1998).

The inhibition of active dissolution of the metal is due to the adsorption of the inhibitor molecules on the metal surface by forming a protective film. The inhibitor molecules can be adsorbed on the metal surface through electron transfer from the adsorbed species to the vacant electron orbital of low energy in the metal to form a co-ordinate type link.

The inhibition efficiency depends on many factors (Bentiss *et al.* 2009) including the number of adsorption centers, mode of interaction with metal surface, molecular size and structure.

It is well known that iron has co-ordination affinity towards nitrogen, sulphur and oxygen bearing ligands (Ali *et al*. 2008; Lowmunkhong *et al*. 2009). Hence, adsorption on iron can be attributed to co-ordination through group, hetero atom (N and S) and π –electrons of aromatic ring, molecular size and mode of interaction with the metal surface and the formation of metallic complexes. The unshared and π –electrons interact with d-orbital of Fe to provide a protective film. The inhibitive properties of such compound depend on the electron densities around the active centre; the inhibition mechanism of the inhibitor is a combination of surface blockage and electrostatic repulsion between adsorbed substance layer and chloride ions (Badr *et al*. 2009). The adsorption density of inhibitor depends on the inhibitor concentration. The inhibitor of these reactions would obviously depend on the degree of the surface coverage of the metal with the inhibitor molecules, on the other. The order of the increasing inhibition, the molecular size of the inhibitor and consequently the number of adsorption centers plays an important role in the enhancement of the protection of carbon steel against corrosion.

5. CONCLUSION

The corrosion behaviour of carbon steel was investigated in aqueous environment with and without addition of various concentration of LA using mass-loss, Potentiodynamic polarization and UV-Visible absorbance spectra, FTIR spectra. The main conclusions are;

1. The formulation consisting 60ppmCl^-, 25ppm Zn^{2+} and 175ppm LA has 94% CE.
2. There is a synergism between LA and Zn^{2+}.
3. ANOVA Test indicates that test was statistically significant.
4. Polarization curves indicated that LA is a cathodic inhibitor. The inhibition efficiencies obtained from polarization were in good agreement.
5. UV-visible spectral study indicates that there was a possibility of formation of complexes.
6. The FTIR analysis showed that the inhibition of carbon steel corrosion occurred due to the formation of protective film on the metal surface adsorption of constituent of LA.
7. The adsorption of LA obeyed Langmuir adsorption isotherm.
8. The formulation consisting of 60ppm Cl^- , 25ppm Zn^{2+} and 175ppm LA has 80% biocidal efficiency.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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