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Corrosion inhibition by arginine – Zn^{2+} system

INTRODUCTION

Amino acids form a class of non-toxic organic compounds which are completely soluble in aqueous media (neutral, acidic or basic medium) and produced with high purity at low cost. These properties would justify their use as corrosion inhibitors. The literature presents some studies involving amino acids on the corrosion prevention of iron [1], steel [2-4], aluminium [5, 6], nickel [7] and copper [8-12]. The electrochemical behaviour of cysteine has been studied by cyclic voltametry using solid electrode [13,14]. The adsorption of amino acid on carbon steel in acidic environment have been investigated by Akiyama and Nobe [15].

The effect of cysteine on the anodic dissolution of copper in sulfuric acid medium have been investigated by Matos et.al. [16]. Copper dissolution behaviour in EDTA and glycine was first examined by producing potential-pH diagrams for the copper-water-EDTA and copper-water-glycine systems [17]. Aksu and Doyle have investigated the electrochemistry of copper in aqueous glycine solutions I the absence of H_2O_2 by comparing the polarization behaviour with the appropriate potential-pH diagram for the copper-water-glycine system [18].

Naturally occurring organic substances as corrosion inhibitors for mild steel in acid have been investigated. Potentiodynamic cathodic and anodic polarization technique was used to study the effect of some common amino acids concentration on the corrosion inhibition of mild steel in H_2O_2 [19]. Inhibition of the corrosion of iron in hydrochloric acid solutions by amino acids was studied. Twenty-two different common amino acids and four related compounds were used. The best results were obtained with 3,4-diiodotyrosine, with an inhibition efficiency of 87%. The best common amino acid was tryptophan with an inhibition efficiency of 80%. Hydroxyproline, cystine, and cysteine acted as corrosion accelerators. Definite trends were observed which were related to the molecular structure. In general, amino acids with longer hydrocarbon chains showed greater inhibition. Additional amino group or groups which increased electron density on the alpha amino group also increased the inhibition efficiency [20].

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The present work is undertaken,

(i) to evaluate the inhibition efficiency of Arginine, in controlling corrosion of carbon steel in an aqueous solution containing 60 ppm of Cl^- by weight loss method.
(ii) to correlate the ligand – transporting efficiency of Zn^{2+} and the corrosion inhibition efficiency of Arginine.
(iii) to study the mechanistic aspects of corrosion inhibition by electrochemical studies such as polarization study, AC impedance spectra.
(iv) to analyze the protective film formed on the metal surface by FTIR spectroscopy and
(v) to propose suitable mechanism of corrosion inhibition based on the above results.

METHODS AND MATERIALS

Preparation of Specimens

Carbon steel (0.026% S, 0.06% P, 0.4% Mn, 0.1% C and the rest Fe) specimen encapsulated in Teflon was polished to a mirror finish and degreased with trichloroethylene. The surface area of the exposed metal surface was 0.5 cm^2.

Weight loss method

Three carbon steel specimens were immersed in 100 ml of the solution containing 60 ppm of Cl^- and various concentrations of the inhibitor in the absence and presence of Zn^{2+}, for a period of 3 days. The weight of the specimen before and after immersion was determined using Shimadzu balance AY62. Inhibition efficiency (IE) was calculated from the relationship IE = (1-W_2/W_1) x 100, where W_1 = corrosion rate in the absence of inhibitor, and W_2 = corrosion rate in the presence of the inhibitor.

Surface examination study

The carbon steel specimens were immersed in various test solution for a period of 3 days. After 3 days, the specimens were taken out and dried. The film formed on the surface of the metal specimens was analysed by surface analysis technique.

FTIR spectra

These spectra were recorded with the Perkin – Elmer - 1600 spectrophotometer. The FTIR spectrum of the protective film was recorded by carefully removing the film mixed it with KBr and making the pellet.

Electrochemical study

The surface of the carbon steel electrode used for this study was 0.5 cm^2.

Potentiodynamic polarization study

Polarization study was carried out in an H and CH electrochemical work station Impedance Analyzer Model CHI 660A provided with iR compensation facility, using a three electrode cell assembly. Carbon steel was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. After having done iR compensation, polarization study was carried out at a sweep rate of 0.01 V/Sec. The corrosion parameters such as linear polarization resistance (LPR), corrosion potential E_{corr}, corrosion current I_{corr} and Tafel slopes (b_a and b_c) were measured.

Alternating current impedance spectra

AC impedance spectra were recorded in the same instrument used for polarization study, using the same type of three electrode cell assembly. The real part (Z') and imaginary part (Z") of the cell impedance were measured in ohms for various frequencies. The charge transfer resistance (R_t) and double layer capacitance (C_{dl}) values were calculated.

Cyclic voltammetry

Cyclic voltammetry was carried out in an H and CH electrochemical work station Impedance Analyzer model CHI 660 provided with iR compensation facility, using a three electrode cell assembly. Carbon steel was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. The graph between (V) vs current (cyclic voltammetry) was drawn.

RESULTS AND DISCUSSION

Analysis of results of weight loss method

Corrosion rates of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^-, in the absence and presence of Arginine and Zn^{2+}, obtained by weight loss method are given in Tables 1 to 3. The inhibition efficiencies are also given in these Tables. The corrosion rates of carbon steel as a function of concentrations of inhibitor systems are shown in Fig 1.

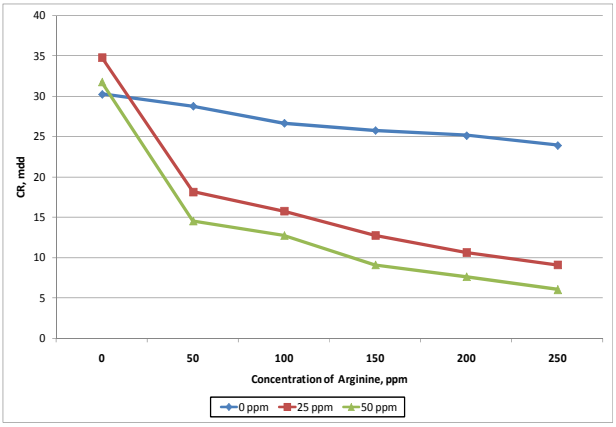

Fig 1: Corrosion rates of carbon steel as a function of concentration of Arginine

It is observed that when carbon steel is immersed in aqueous solution, in the presence of 60 ppm of Cl^-, the corrosion rate is 30.28 mdd. Upon addition of various concentrations of Arginine, the corrosion rate slowly decreases. The inhibition efficiency gradually increases from 5% to 21%. 250 ppm of Arginine has 21% IE.

Table 1 - Corrosion rates (CR) of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- and the inhibition efficiencies (IE) obtained by weight loss method. Inhibitor: Arginine + Zn^{2+}

Cl^- ppm	Arginine ppm	Zn^{2+} ppm	CR mdd	IE %
60	0	0	30.28	-
60	50	0	28.77	5
60	100	0	26.64	12
60	150	0	25.74	15
60	200	0	25.13	17
60	250	0	23.92	21

Influence of Zn^{2+} on the inhibition efficiency of Arginine

The influence of a divalent metal ion, Zn^{2+}, on the efficiency of Arginine, in controlling corrosion of carbon steel, is given Tables 2 and 3. It is observed that in the presence of 25 ppm of Zn^{2+}, the IE of Arginine slightly improves. The divalent Zn^{2+} ion forms a complex with Arginine, diffuses towards the metal surface and form Fe^{2+} - Arginine complex releasing Zn^{2+}.

Table 2 - Corrosion rates (CR) of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- and the inhibition efficiencies (IE) obtained by weight loss method. Inhibitor: Arginine + Zn^{2+}

Cl^- ppm	Arginine ppm	Zn^{2+} ppm	CR mdd	IE %
60	0	0	30.28	-
60	0	25	34.82	-15
60	50	25	18.17	40
60	100	25	15.75	48
60	150	25	12.72	58
60	200	25	10.60	65
60	250	25	9.08	70

Table 3 - Corrosion rates (CR) of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- and the inhibition efficiencies (IE) obtained by weight loss method: Inhibitor: Arginine + Zn^{2+}

Cl^- ppm	Arginine ppm	Zn^{2+} ppm	CR mdd	IE %
60	0	0	30.28	-
60	0	50	31.79	-5
60	50	50	14.53	52
60	100	50	12.72	58
60	150	50	9.08	70
60	200	50	7.57	75
60	250	50	6.06	80

In the presence of 50 ppm of Zn^{2+}, the inhibition efficiency of Arginine still increases. This is due to the fact that more Arginine is transported towards metal surface increases the formation of Fe^{2+}-Arginine complex and hence an increase in the IE is noticed. Similar observation has been made by Rajendran et.al., when they studied the inhibition efficiency of phosphonic acid in the presence of Zn^{2+} [21].

Synergism parameters

Synergism parameters were calculated using the relation

$$S_I = 1\text{-}I_{1+2} / 1\text{-}I'_{1+2}$$

Where $I_{1+2} = (I_1+I_2) - (I_1 x I_2)$

I_1=inhibition efficiency of substance 1

I_2=inhibition efficiency of substance 2

Where I'_{1+2}= combined inhibition efficiency of substance 1 & 2

Synergism parameters are indications of synergistic effect existing between two inhibitors [22]. The values of synergism parameters [Tables 4 and 5] are greater than one, indicating synergistic effect existing between Zn^{2+} and various concentrations of Arginine.

Table 4 - Synergism parameters derived from inhibition efficiencies of Arginine – Zn^{2+} system.

Arginine ppm	Zn^{2+}		S_I
	0 ppm	25 ppm	
0	0	-15	0
50	5	40	1.64
100	12	48	3.74
150	15	58	3.93
200	17	65	4.00
250	21	70	4.64

Table 5 - Synergism parameters derived from inhibition efficiencies of Arginine – Zn^{2+} system.

Arginine ppm	Zn^{2+}		S_I
	0 ppm	50 ppm	
0	0	-5	0
50	5	52	0.47
100	12	58	1.16
150	15	70	1.22
200	17	75	1.30
250	21	80	1.52

Analysis of Variance (ANOVA)

To investigate whether, the influence of Zn^{2+} on the inhibition efficiencies of Arginine is statistically significant, F- test was carried out [23]. The results are given Tables 6 and 7.

In Table 6, the influence of 25 ppm of Zn^{2+} on the inhibition efficiencies of 50, 100, 150, 200 and 250 ppm of Arginine is investigated. The obtained F – value 47.82 is statistically significant, since it is greater than the critical F – value 5.32 for 1, 8 degrees of freedom at 0.05 level of significance. Therefore, it is concluded that the influence of 25 ppm Zn^{2+} on the inhibition efficiencies of various concentrations of Arginine is statistically significant.

Table 6 - Distribution of F-value between the inhibition efficiencies of various concentrations of Arginine (0 ppm Zn^{2+}) and the inhibition efficiencies of Arginine in the presence of 25 ppm Zn^{2+}

Source of variance	Sum of squares	Degrees of freedom	Mean square	F	Level of significance of F
Between	4452.1	1	4452.1	47.82	p>0.05
Within	744.8	8	93.1		

In Table 7, the influence of 50 ppm of Zn^{2+} on the inhibition efficiencies of 50, 100, 150, 200 and 250 ppm of Arginine is investigated. The obtained F – value 81.18 is statistically significant. Since it is greater than the critical F – value 5.32 for 1, 8 degrees of freedom at 0.05 level of significance. Therefore, it is concluded that the influence of 50 ppm Zn^{2+} on the inhibition efficiencies of various concentrations of Arginine is statistically significant.

Table 7 - Distribution of F-value between the inhibition efficiencies of various concentrations of Arginine (0 ppm Zn^{2+}) and the inhibition efficiencies of Arginine in the presence of 50 ppm Zn^{2+}

Source of variance	Sum of squares	Degrees of freedom	Mean square	F	Level of significance of F
Between	7022.5	1	7022.5	81.18	p>0.05
Within	692	8	86.5		

Analysis of polarization curves

The potentio dynamic polarization curves of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- are shown in Fig. 2.

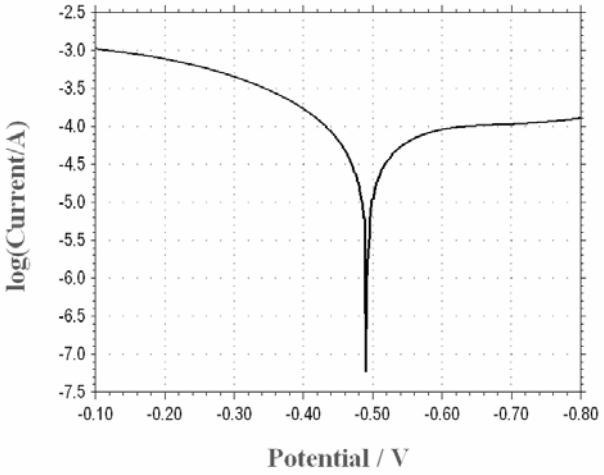

Fig.2: Polarization curves of carbon steel immersed in various test solutions : a) Cl^- 60 ppm

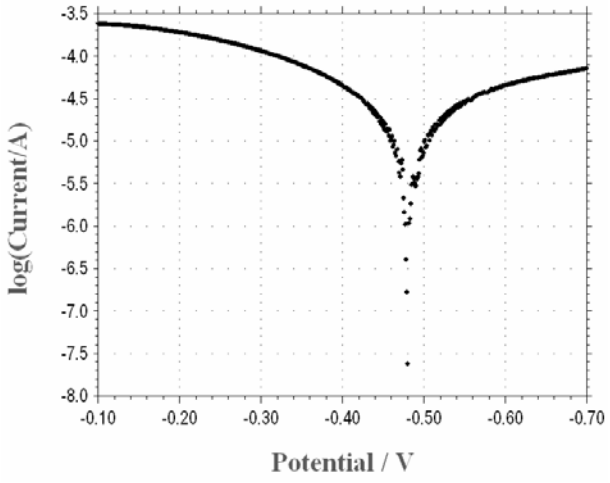

Fig.2: Polarization curves of carbon steel immersed in various test solutions : b) Cl^- 60 ppm + Arginine 250 ppm + Zn^{2+} 50 ppm

The corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes (b_a,b_c) and linear polarization resistance (LPR) are given in Table 8.

When carbon steel is immersed in an aqueous solution containing 60 ppm of Cl^-, the corrosion potential is -490 mV vs SCE. When the inhibitors are added (250 ppm of Arginine and 50 ppm of Zn^{2+}), the corrosion potential shifts to the anodic side (-480 mV vs SCE). This shift is very small, further the LPR value increases from 7.2 x 10^2 ohm cm^2 to 2.1 x 10^3 ohm cm^2 and the corrosion current decreases from 8.2 x 10^{-5} A/cm^2 to 2.7 x 10^{-5} A/cm^2. This result suggests that a protective film (Fe^{2+} - Arginine complex) is formed on the metal surface. This protects the metal from corrosion. Arginine – Zn^{2+} system functions as a mixed type inhibitor.

Table 8 - Corrosion parameters of Carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- obtained by polarization study. Inhibitor: Arginine + Zn^{2+}

Cl^- ppm	Arginine ppm	Zn^{2+} ppm	E_{corr} mV vs SCE	b_c mV/decade	b_a mV/decade	LPR ohm cm^2	I_{corr} A/cm^2
60	0	0	-490	506	188	7.2 x 10^2	8.2 x 10^{-5}
60	250	50	-480	296	210	21 x 10^2	2.7 x 10^{-5}

Analysis of AC impedance spectra

The AC impedance spectra of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- in the absence and presence of inhibitors are shown in Fig. 3.

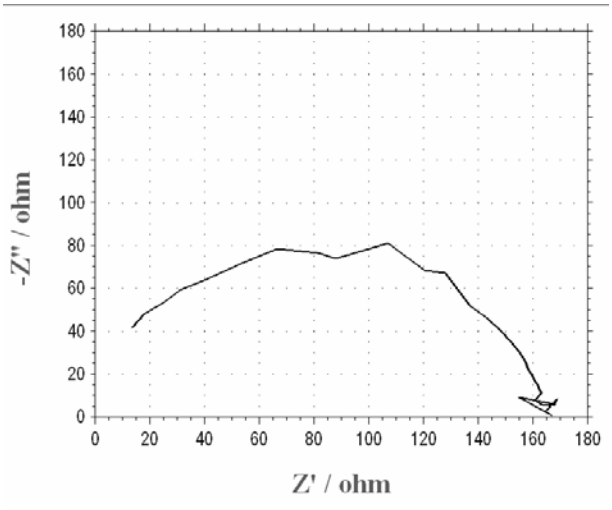

Fig.3: AC impedance spectra of carbon steel immersed in various test solutions, Nyquist plots: a) Cl^- 60 ppm

The AC impedance parameters such as charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in Table 9. In the presence of inhibitors (250 ppm of Arginine and 50 ppm of Zn^{2+}), the R_t value increases and C_{dl} value decreases. This indicates that a protective film is formed on the metal surface. The corresponding Bode plots are shown in Fig. 3 (c) and (d). It is observed that in the absence of inhibitors the real impedance value [log(Z/ohm)] is 2.23. In the presence of inhibitors this value increases to 2.76.

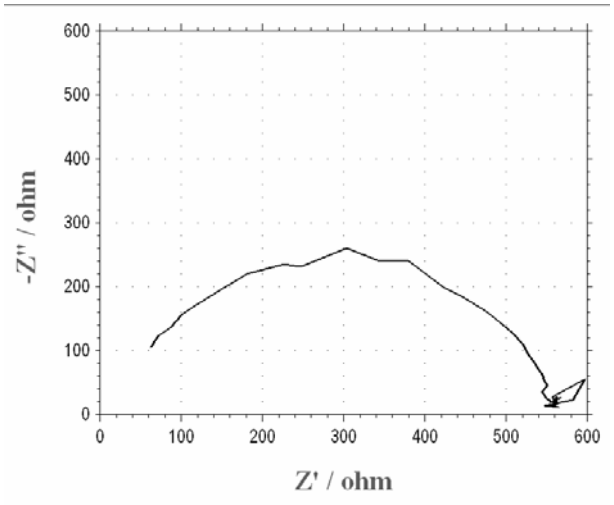

Fig.3: AC impedance spectra of carbon steel immersed in various test solutions, Nyquist plots: b) Cl^- 60 ppm + Arginine 250 ppm + Zn^{2+} 50 ppm

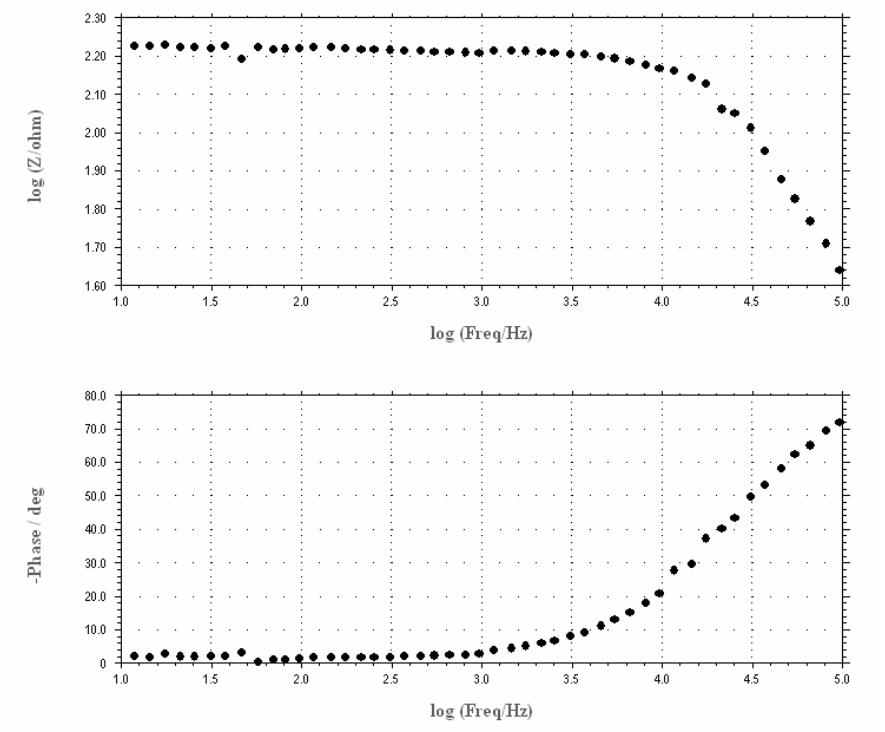

Fig.3: AC impedance spectra of carbon steel immersed in various test solutions, Bode plots: c: Cl^- 60 ppm

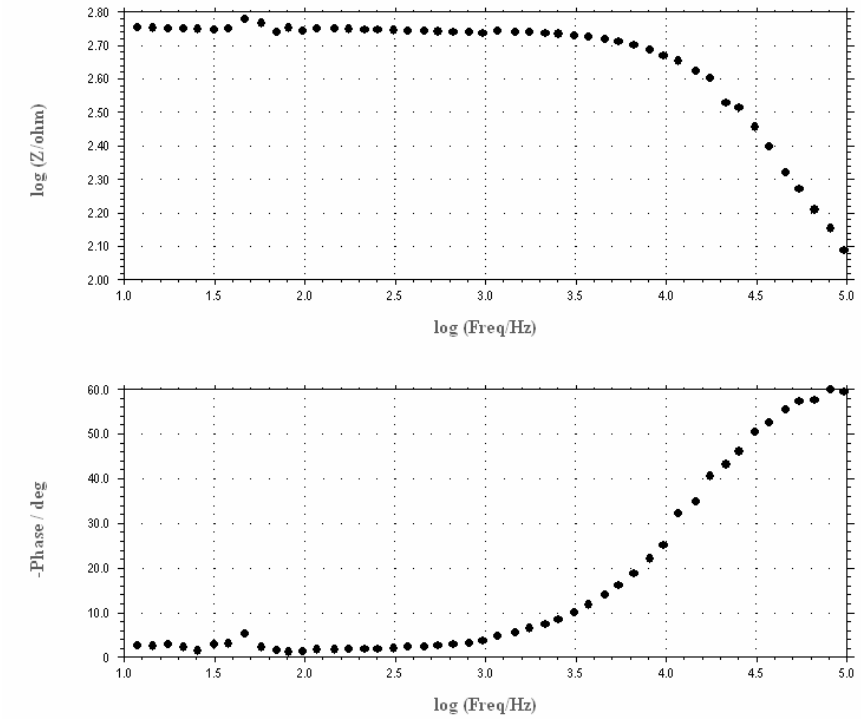

Fig.3: AC impedance spectra of carbon steel immersed in various test solutions, Bode plots : d) Cl^- 60 ppm + Arginine 250 ppm + Zn^{2+} 50 ppm

The AC impedance parameters such as charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in Table 9. In the presence of inhibitors (250 ppm of Arginine and 50 ppm of Zn^{2+}), the R_t value increases and C_{dl} value decreases. This indicates that a protective film is formed on the metal surface. The corresponding Bode plots are shown in Fig. 3 (c) and (d). It is observed that in the absence of inhibitors the real impedance value [log(Z/ohm)] is 2.23. In the presence of inhibitors this value increases to 2.76.

Table 9 - AC impedance parameters of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^- obtained by AC impedance spectra. Inhibitor: Arginine + Zn^{2+}

Cl^- ppm	Argi-nine ppm	Zn^{2+} ppm	R_t ohm cm^2	C_{dl1} F/cm^2	Impedance value log[Z/ohm]
60	0	0	153	5.9×10^{-8}	2.23
60	250	50	495	1.8×10^{-8}	2.76

Analysis of Cyclic Voltammograms

The cyclic voltammograms of carbon steel immersed in various test solutions are shown in Fig.4. There is no indication of any redox couple. No characteristic peaks were observed.

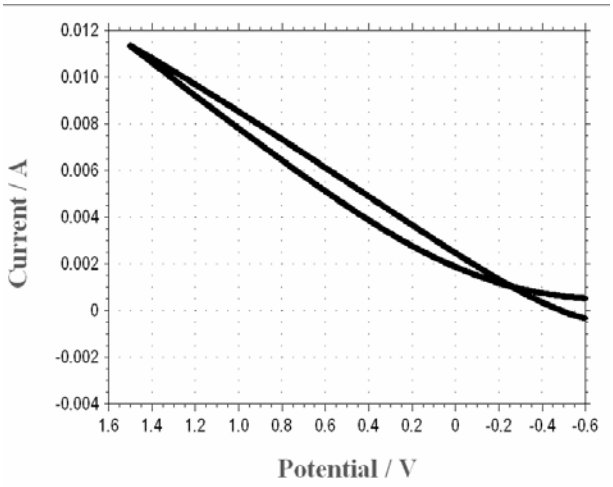

Fig.4: Cyclic voltammograms of carbon steel immersed in various test solutions: a) Cl^- 60 ppm

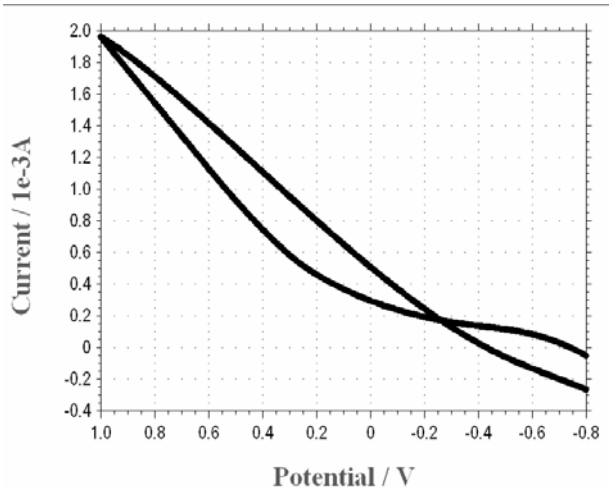

Fig.4: Cyclic voltammograms of carbon steel immersed in various test solutions. b) Cl^- 60 ppm + Arginine 250 ppm + Zn^{2+} 50 ppm

Analysis of FTIR spectra

The FTIR spectrum of pure Arginine (KBr) is shown in Fig. 5 (a). The C=O stretching frequency appears at 1671 cm^{-1}. The C-N stretching frequency appears at 1132 cm^{-1} [24]. The FTIR spectrum of the film formed on the metal surface after immersion in the solution containing 60 ppm of Cl^-, 250 ppm of Arginine and 50 ppm of Zn^{2+} is shown in Fig. 5 (b). It is observed that the C=O stretching frequency has shifted from 1671 cm^{-1} to 1660 cm^{-1}; the C-N stretching frequency has shifted from 1132 cm^{-1} to 1120 cm^{-1}. This suggests that Arginine has coordinated with Fe^{2+}, through the electron of oxygen atom of C=O group and N-atom of the –C-NH_2 group resulting in the formation of Fe^{2+} - Arginine complex on the metal surface. The peak at 1350 cm^{-1} is due to $Zn(OH)_2$. Thus analysis of FTIR spectra leads to the conclusion that the protective film consist of Fe^{2+} - Arginine complex and $Zn(OH)_2$ [25,26].

Mechanism of corrosion inhibition

Weight loss study reveals that the formulation consisting of 250 ppm Arginine and 50 ppm of Zn^{2+} has 80% inhibition efficiency in controlling corrosion of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^-. Synergism parameters suggest that a synergistic effect exists between Arginine and Zn^{2+}. Polarization study reveals that this system functions as mixed type inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. FTIR spectra reveal that the protection film consists of Fe^{2+} - Arginine complex.

In order to explain the above fact in a holistic way the following mechanism of corrosion inhibition is proposed.

- When the formulation consisting of 250 ppm of Arginine and 50 ppm of Zn^{2+} is prepared, there is formation of Zn^{2+} - Arginine complex in solution.
- When carbon steel metal is immersed in this solution, there is diffusion of Zn^{2+} - Arginine complex towards the metal surface.
- On the metal surface, it is converted to Fe^{2+} - Arginine complex. Zn^{2+} is released.

$$Zn^{2+} \text{ - Arginine} + Fe^{2+} \longrightarrow Fe^{2+} \text{ - Arginine} + Zn^{2+}$$

$$Zn^{2+} + 2OH^- \longrightarrow Zn(OH)_2\downarrow$$

- Thus the protective film consists of Fe^{2+}- Arginine complex and $Zn(OH)_2$.

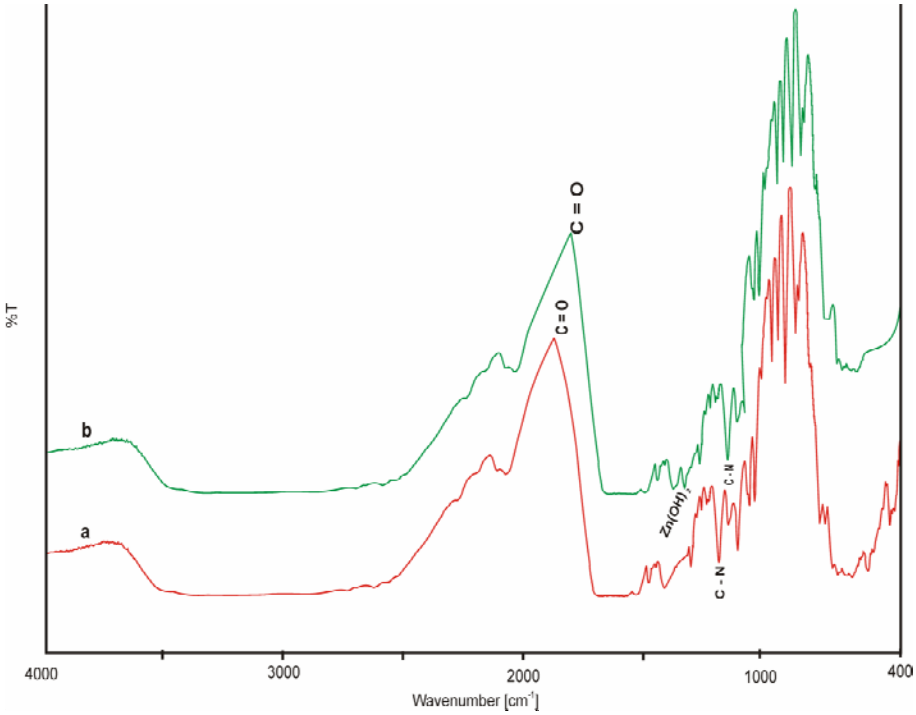

Fig. 5: FTIR spectra (KBr): a) pure Arginine, b) film formed on metal surface after immersion in test solution containing 60 ppm Cl^- + 250 ppm Arginine + 50 ppm Zn^{2+}

CONCLUSIONS

The inhibition efficiency of Arginine –Zn^{2+} system in controlling corrosion of carbon steel in an aqueous solution containing 60 ppm of Cl^-, has been evaluated by weight loss method. The present study leads to the following conclusion.

- Weight loss study reveals that the formulation consisting of 250 ppm Arginine and 50 ppm of Zn^{2+} has 80% inhibition efficiency in controlling corrosion of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^-.
- Synergism parameters suggest that a synergistic effect exists between Arginine and Zn^{2+}.
- Polarization study reveals that this system functions as a mixed type inhibitor.
- AC impedance spectra reveal that a protective film is formed on the metal surface.
- FTIR spectra reveal that the protection film consists of Fe^{3+} - Arginine complex.
- As the amount of Zn^{2+} ion available for transport of the ligand increases, the inhibition efficiency increases.

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ABSTRACT

CORROSION INHIBITION BY ARGININE – Zn^{2+} SYSTEM

The inhibition efficiency of arginine in controlling corrosion of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^-, has been evaluated by weight loss method in the absence and presence of Zn^{2+}. Weight loss study reveals that the formulation consisting of 250 ppm Arginine and 50 ppm of Zn^{2+} has 80% inhibition efficiency in controlling corrosion of carbon steel immersed in an aqueous solution containing 60 ppm of Cl^-. Synergism parameters suggest that a synergistic effect exists between Arginine and Zn^{2+}. Polarization study reveals that this system functions as mixed type inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. FTIR spectra reveal that the protection film consists of Fe^{2+} - Arginine complex. As the amount of Zn^{2+} ion available for transport of the ligand increases, the inhibition efficiency increases.

Key words : *Corrosion inhibition, amino acid, F-Test, synergistic effect, synergism parameter*