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RESEARCH ARTICLE

Corrosion inhibition of mild carbon steel in well water medium L-TRYPTOPHAN - Zn^{2+} System

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ABSTRACT

The environmentally friendly inhibitor system, L-Tryptophan-Zn^{2+}, was investigated using the weight loss method. A synergistic effect was observed between L-Tryptophan and Zn^{2+} system. The formulation comprising 250 ppm of L-Serine and 50 ppm of Zn^{2+} demonstrated an impressive inhibition efficiency of 91%. UV-Visible spectroscopy revealed the formation of a protective film on the metal surface. FTIR analysis suggested that the Zn^{2+}-L-Tryptophan complex formed at the anodic sites of the metal surface, which inhibited the anodic reaction, while $Zn(OH)_2$ formed at the cathodic sites, controlling the cathodic reaction. A corrosion inhibition mechanism was proposed based on the results from weight loss studies and surface analysis techniques. Synergism parameters were calculated and found to be greater than 1, confirming the synergistic effect between L-Tryptophan and Zn^{2+}. The surface morphology of the compound was examined using SEM and EDAX.

1. INTRODUCTION

Corrosion is a natural process that converts a refined metal into a more chemically stable oxide. It is the gradual deterioration of materials (usually a metal) by chemical or electrochemical reaction with their environment. In the most common use of the word, this means electrochemical oxidation of metal in reaction with an oxidant such as oxygen, hydrogen or hydroxide. Rusting, the formation of iron oxides, is a well-known example of electrochemical corrosion.(1)

Corrosion inhibitors are chemicals that, when added in small amounts to a hostile environment, reduce the rate of attack on a material such as a metal. The corrosion inhibitor slows down the rate at which a metal in that environment corrode.(1) Uniform corrosion, pitting, crevice corrosion, filiform corrosion, galvanic corrosion, environmental cracking, and fretting corrosion.(1) Corrosion prevention refers to the implementation of strategies and techniques aimed at reducing or eliminating the deterioration of materials caused by chemical reactions with the environment. There are effective corrosion prevention methods that can extend the life of metal equipment by up to 250%. Not all corrosion prevention methods are equal.(2) An inhibitor is a chemical compound that effectively reduces the corrosion rate of metal when added in small concentration or otherwise mildly aggressive medium or environment. Inhibitors are classified as anodic, cathodic or mixed inhibitors. Inhibitors reduces corrosion by interfering with one or more of the corrosion reactions and affecting the corrosion process as a whole. As most corrosion inhibitors have a certain amount of toxicity, care must be taken to ensure that their application confirms to prevaile environmental and health regulations. Economic consideration also governs their selection.(3)

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Amino acids contain amino group and carboxyl group. They contain electron rich nitrogen atom and oxygen atom. These electrons can be released to the metal surface and thus corrosion of metals can be prevented. Corrosion of many metals have prevented by amino acids in acidic, basic, and neutral medium. Usually, weight loss method and electrochemical studies have been employed to evaluate the corrosion inhibition efficiency of amino acids. Adsorption of amino acids on metal surface obey Frumkin, Langmuir, Temkin and Freundlich adsorption isotherms. The protective film formed on metal surface in presence of amino acids have been analyzed by FTIR spectra, SEM, EDAX and XRD pattern.

Amino acids have amino group (-NH2) and carboxyl group (-COOH). Lone pair of electrons are available on nitrogen atom and oxygen atom. Hence coordination of amino acids with metal atoms can take place through both these atoms. When there is flow of electrons from these electron rich centres, to electron deficient centres of the metal, corrosion rate will be reduced. Hence amino acids are widely used as, corrosion inhibitors [1-26].

The principal application areas for inhibitors are,

i. Industrial water systems - for cooling, processing, boiler and condenser systems.
ii. Natural water for human use in 6 to 8.5 pH range.
iii. In aqueous solutions of acids used for metal cleaning
iv. In non- aqueous media and in the mineral oil industry during production of crude and its subsequent refining and processing.
v. For the protection of metallic components e.g., machinery tools, electronic hardware etc., [2]

2. MATERIALS AND METHODS

2.1. Weight loss method

The weight losses are found by keeping identical specimens for a constant time and temperature in the solution under study. Inhibition efficiency (%) is calculated by using the formula.

$$IE(\%)=100\ [1 - W2/W1]\ \%$$

Where W1 is the weight loss in the absence of inhibitor

W2 is the weight loss in the presence of inhibitor.[10]

2.2. FTIR Spectra

The carbon steel specimens immersed in various test solutions for one day were taken out and dried. The film formed on the metal surface was carefully removed and thoroughly mixed with KBr, so as to make it uniform throughout. The FTIR spectra were recorded in a Perkin- Elmer 1600 spectrophotometer. [10]

2.3. Ultraviolet and visible spectroscopy

While interaction with infrared light causes molecules to undergo vibrational transitions, the shorter wavelength, higher energy radiation in the UV (200-400nm) and visible (400-700nm) range of the electromagnetic spectrum causes many organic molecules to undergo electronic transitions. What this means is that when the energy from UV or visible light is absorbed by a molecule, one of its electrons jumps from a lower energy to a higher energy molecular orbital.[6]

2.4. Surface Morphology methods

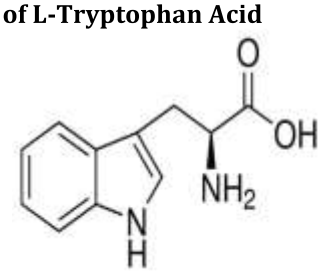
Interfacial tension measurements, double layer capacity measurements, radio tracer technique, X- ray photoelectron spectroscopy, soft X-ray spectro microscopy, auger electron spectroscopy, electron microscopy, magnetic susceptibility, XRD, SEM, ESCA, AFM methods have also been useful to study the mechanism of some inhibitor.[6]

2.5. Characteristics of inhibitor

L-Tryptophan Acid

Molecular weight = 204.23 g/mol
Molecular Formula = $C_{11}H_{12}N_2O_2$

Structure of L-Tryptophan Acid



Glutamic acid is an essential amino acid. L-Tryptophan Acid is an antioxidant. It may help to protect the body from damage caused by ionizing radiation. It also prevents liver damage from acetaminophen poisoning. L-Tryptophan is the L-enantiomer of glutamic acid. It has a role as a nutraceutical, a micronutrient, an antidote to paracetamol poisoning, a human metabolite and a mouse metabolite. It is used in the biosynthesis of proteins. In plants and microorganisms, tryptophan acid biosynthesis belongs to the aspartate family, along with threonine and lysine. The main backbone of glutamic acid is derived from aspartic acid, while the sulphur may come from cysteine or hydrogen sulfide.

2.6. Determination of corrosion rate

The weighed specimens in triplicate were suspended by means of glass or plastic hooks in 100 ml beakers containing 100 ml of various test solutions. After 7 days of immersion, the specimens were taken out, dried and washed. From the change in weight of the specimens, corrosion rates were calculated using the following relationship.
Corrosion rate = Loss in weight (mg)Surface area of the specimen dm2 × Period of immersion (days)
The corrosion rate is expressed in mdd units [mdd = mgm/(dm2)(day)]
Corrosion inhibition efficiency (I.E) was then calculated using the equation given below.

IE=100 (1-W2W1)%

Where W1 = Corrosion rate in the absence of inhibitor
W2 = Corrosion rate in the presence of inhibitor

2.7. Synergism Parameters (S_I)

The synergism parameters (SI) were calculated using the relation as stated below[]

$S_i = 1+ \theta_{1+2}/1-,\theta_{1+2}$

Where $\theta_{1+2} = (\theta_1 + \theta_2) - (\theta_1 - \theta_2)$
θ1+2' = Combined inhibition efficiency of substance 1 and substance 2

If the resultant value of SI is greater than 1, then the result confirms the synergistic effect between the inhibitor and the additives.

3. MATERIALS AND METHODS

3.1. Preparation of mild carbon steel specimen

The mild carbon steel specimen used for the experiment is in the composition following composition.

Table 1 : Composition of mild carbon steel

Elements	**C**	**Mn**	**P**	**Si**	**S**	**Cr**	**Ni**	**Mo**	**Fe**
Composition (%)	**0.017**	**0.196**	**0.009**	**0.007**	**0.014**	**0.043**	**0.013**	**0.015**	**99.686**

Mild carbon steel specimen of the above composition was analysed by using vacuum emission spectrometer DV-4 (supplied by BAIRD Corporation of India) and of the dimensions 1.0 cm × 4.0 cm × 0.2 cm were polished to mirror finish using emery sheets, washed with distilled water, dried and were used for the weight loss and surface examination studies.

3.2. Chemicals used

L-Tryptophan
Zinc sulphate
Ferrous sulphate

3.3. Well Water

For the present study, well water of The American College, Madurai was used. Corrosion behaviour of mild carbon steel in this water was evaluated.

Table 2: Water Parameters

S.No	**Parameters**	**Test Method**	**Unit**	**Range**
1	PH	IS 3025 part 11- 1983	-	7.93
2	Total Hardness as $CaCO_3$	IS 3025 Part 21- 2009	mg/L	620
3	Total Alkalinity	IS 3025 Part 23- 1986	mg/L	540

4	Chloride as Cl	IS 3025 part 32- 1988	mg/L	600
5	Sulphate as SO_4	IS 3025 part 24- 1986	mg/L	99
6	Nitrate as NO_3	APHA. 23rd Edition 2017- 4500 NO_3 B	mg/L	15
7	Fluoride as F	APHA. 23rd Edition 2017 - 4500 - F- D	mg/L	Less than 0.2

3.4. Preparation of stock solution

1g of L - Tryptophan Acid was dissolved in well water and made up to 100ml in a standard measuring flask. 1ml of this solution was diluted to 100ml to get 100ppm of L - Tryptophan Acid.

Zinc sulphate solution

Exactly 1.1g of Zinc sulphate was dissolved in well water and made up to 250ml in a standard measuring flask.

The details regarding the preparation of various environments used for weight loss method in the present study is given in the Table 1,2, 3.

Table 3: L- Tryptophan Acid and $ZnSO_4$

S.No	L- Tryptophan Acid (ppm)	$ZnSO_4$ Solution (ppm)	Total volume made up to 100 ml with well water
1	-	-	100
2	50	0	100
3	100	0	100
4	150	0	100
5	200	0	100
6	250	0	100

Table 4: L- Tryptophan Acid and $ZnSO_4$ (10 ppm)

S.No	L- Tryptophan Acid (ppm)	$ZnSO_4$ solution (ppm)	Total volume made up to 100 ml with well water
1	-	-	100
2	50	10	100
3	100	10	100
4	150	10	100
5	200	10	100
6	250	10	100

Table 5: L - Tryptophan Acid and $ZnSO_4$ (50 ppm)

S.No	L- Tryptophan Acid (ppm)	$ZnSO_4$ solution (ppm)	Total volume made up to 100 ml with well water
1	-	-	100
2	50	50	100
3	100	50	100
4	150	50	100
5	200	50	100
6	250	50	100

The environment chosen for the surface examination studies his is given in the Table 6. Among the several inhibitor combinations, the one which offered the highest inhibition efficiency was chosen as the environment.

Table 6: Preparation of environment for surface examination studies

S.No	Inhibitor	Environment Chosen
1	**L-** Tryptophan Acid	Well Water + L- Tryptophan Acid (250ppm) + Zn^{2+}(50 ppm)

4. RESULT AND DISCUSSION

4.1. Analysis of results of the weight loss method

Inhibition efficiency (IE%) of L-trytophan -Zn^{2+} systems in controlling corrosion of carbon steel immersed in well water in the presence and absence of inhibitor system (Immersion period = 7 days) are given in the table 6-8. It is observed that L-Tryptophan alone has poor inhibition efficiency. In the presence of various concentrations of Zn^{2+}(10 and 50 ppm) the IE of L-tryptophan increases. A synergistic effect exists between L - tryptophan and Zn^{2+}.(16)

Table 6: Corrosion rates (CR) of mild steel immersed in well water in the presence and absence of inhibitor system at various concentrations and the inhibition efficiencies (IE) obtained by weight loss method.

Inhibitor system : L-Tryptophan Acid Zn^{2+}(0 ppm)
Immersion period. : 7 days
pH : 5.5 - 7.0

L- Tryptophan Acid (ppm)	Zn^{2+} ppm	CR (mdd)	IE (%)
0	0	35.06	-
50	0	19.0	13.9
100	0	17.4	22.0

150	0	15.2	31.1
200	0	14.0	36
250	0	12.6	42.9

Table 7: Corrosion rates (CR) of mild steel immersed in well water in the presence and absence of inhibitor system at various concentrations and the inhibition efficiencies (IE) obtained by weight loss method.

Inhibitor system : L- Tryptophan Acid Zn^{2+} (10 ppm)
Immersion period : 7 days
pH : 5.5 - 7.0

L- Tryptophan Acid (ppm)	Zn^{2+}ppm	CR(mdd)	IE(%)
0	10	35.06	-
50	10	10.11	61
100	10	4.5	82
150	10	4.3	83
200	10	3.63	86
250	10	2.38	90

Table 8: Corrosion rates (CR) of mild steel immersed in well water in the presence and absence of inhibitor system at various concentrations and the inhibition efficiencies (IE) obtained by weight loss method.

Inhibitor system : L-Tryptophan Acid Zn^{2+}(50 ppm)
Immersion period : 7 days
pH : 5.5 - 7.0

L-Tryptophan Acid. (ppm)	Zn^{2+}ppm	CR (mdd)	IE (%)
0	0	35.06	-
50	50	20.07	51
100	50	18.18	60
150	50	11.68	74
200	50	5.19	88
250	50	3.89	91

4.2. Synergism parameter (S_i)

Synergism parameter (SI) has been used to know the synergistic effect existing between two inhibitors [9 - 14]. Synergism parameter (SI) can be calculated using the following relationship.

$$SI= 1-\theta_{1+2} / 1- \theta^1_{1+2}$$

Where

Θ = surface coverage

θ_{1+2} = (θ1+θ2) - (θ1θ2)

θ_1 = surface coverage by L-Tryptophan

θ_2 = surface coverage by Zn^{2+}

$\theta`_{1+2}$ = surface coverage by both L-tryptophan and Zn^{2+}

and where θ=IE/100%

The synergism parameters of L-Tryptophan -Zn^{2+}system were given in Table 7 and Table 8 corresponding to various concentration of Zn^{2+}ion. For different concentrations of inhibitors, SI approaches 1 when no interaction between the inhibitor, compounds exist. When SI >1, it points to synergistic effects. In the case of SI < 1, it is an indication that the synergistic effect is not significant. From table 9, it is observed that value of synergism parameters (SI) calculated from surface coverage were found to be one and above. This indicates that the synergistic effect exists between L-tryptophan and Zn^{2+}. Thus the enhancement of the inhibition efficiency caused by the addition of Zn^{2+} ions to L-Tryptophan is due to the synergistic effect. (15)

Table 9. Inhibition efficiencies and synergism parameters for various concentrations of L-Tryptophan Acid and Zn^{2+} (10 ppm) system.

L-Tryptophan Acid (ppm)	Inhibition efficiency IE(%)	Surface coverage (	Zn^{2+} (Ppm)	IE %	Surface coverage	Combined IE% I_{1+2}	Combined surface coverage	Synergism parameters (S_i)
50	13.9	0.13	10	10	0.10	61	0.61	1.22
100	22.0	0.22	10	10	0.10	56	0.56	1.54
150	31.12	0.31	10	10	0.10	63	0.63	1.59
200	36.	0.36	10	10	0.10	69	0.69	1.74
250	42.9	0.42	10	10	0.10	90	0.90	1.88

Table 10: Inhibition efficiencies and synergism parameters for various concentrations of L-Glutamic acid-Zn2+ (50 ppm) system.

L-Tryptophan Acid (ppm)	Inhibition Efficiency IE%	Surface coverage (θ_1)	Zn^{2+} (ppm)	IE %	Surface coverage (θ_2)	Combined IE% I_{1+2}	Combined surface coverage	Synergism parameters (S_i)
50	13.9	0.13	50	15	0.15	51	0.51	1.46
100	22.0	0.22	50	15	0.15	60	0.60	1.57
150	31.1	0.31	50	15	0.15	74	0.74	2.07
200	36	0.36	50	15	0.15	88	0.88	4.08
250	42.9	0.42	50	15	0.15	91	0.91	4.77

4.3. UV- Visible Absorption Spectroscopy

UV-visible absorption spectrum of an aqueous solution containing tryptophan and Fe^{2+}(freshly) prepared $FeSO_4$. $7H_2O$ is shown in Fig. 1

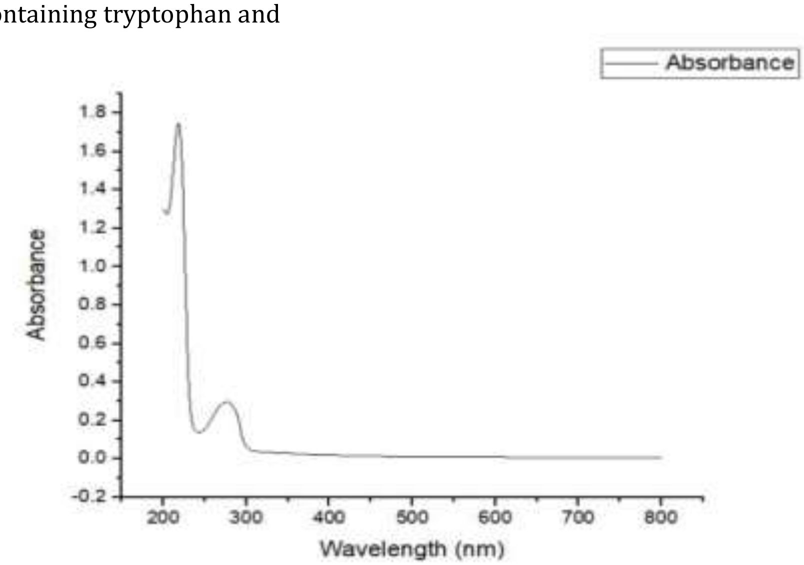


Fig .1. A peak appears at 280 nm. This peak is due to the formation of Fe^{2+} L- Tryptophan complex formed in solution.

4.4. Analysis of FTIR spectra

The FTIR spectrum of the film formed on the metal surface after immersion in the well water, 200 ppm of L-tryptophan and 50 ppm $Zn^{2+}$is shown in Figure 2. The -C=O stretching frequency of carboxyl group appears at 288.79 cm-1. The -CN stretching frequency appears at 1666.88 cm-1. The -NH stretching frequency appears at 3466.74 cm-1. [16] The C-S stretching frequency is at 806.98 cm-1. This observation suggests that L-tryptophan has coordinated with Fe^{2+} through the oxygen atom of the carboxyl group and nitrogen atom of the amine group resulting in the formation of Fe^{2+}-L-tryptophan acid complex on the anodic sites of the metal surface. The peak at 521.8 cm-1 corresponds to Zn-O stretching. The peak at 3404.95cm-1 is due to -OH stretching. This confirms that $Zn(OH)_2$is formed on the cathodic sites of metal surface . Thus the FTIR spectral study leads to the conclusion that the protective film consist of Fe^{2+}-L-tryptophan complex and $Zn(OH)_2$

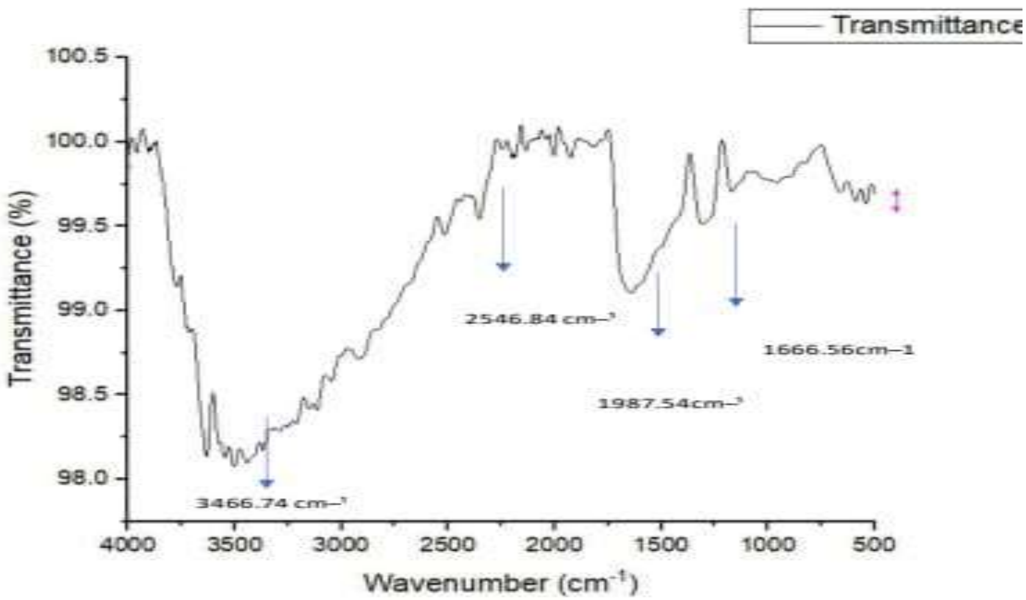


Fig.2 FT_IR Spectrum of L-tryptophan and 50 ppm Zn^{2+}

4.5. SEM Analysis of Metal Surface

SEM provides a pictorial representation of the surface. To understand the 00nature of the surface film in the absence and presence of inhibitors and the extent of corrosion of carbon steel, the SEM micrographs of the surface are examined. The SEM images of different magnification of carbon steel specimen immersed in well water for 7 days in the absence and presence of inhibitor system are shown in Figure 3 (a, b, c, d, e, f) respectively. The SEM micrographs of polished carbon steel surface (control) in Figure 3 (a,b) shows the smooth surface of the metal. This shows the absence of any corrosion products (or) inhibitor complex formed on the metal surface. The SEM micrographs of carbon steel surface immersed in well water (Figure 3 (c, d)) show the roughness of the metal surface which indicates the highly corroded area of carbon steel in well water. However in Figure 3 (e, f) indicate that in the presence of inhibitor (250 ppm L-L- tryptophan and 50 ppm Zn^{2+}) the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The metal surface almost free from corrosion is due to the formation of insoluble complex on the surface of the metal. In the presence of L-Tryptophan and Zn2+, the surface is covered by a thin layer inhibitors which effectively controls the dissolution of carbon steel.

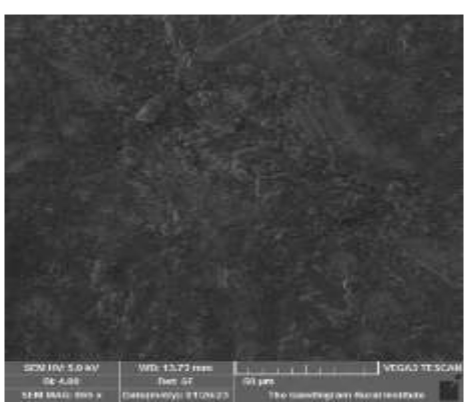


Fig (a).

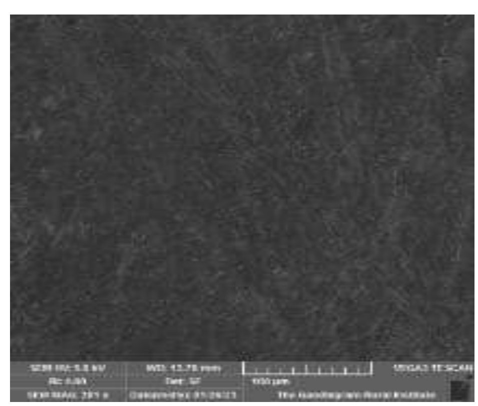


Fig (b)

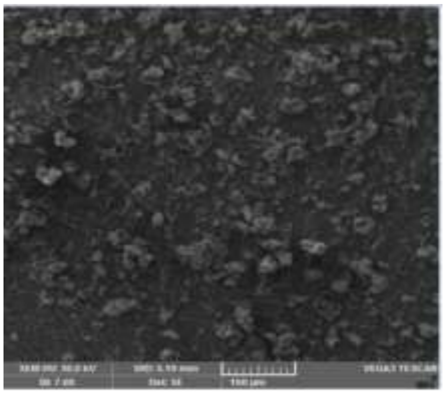


Fig (c)

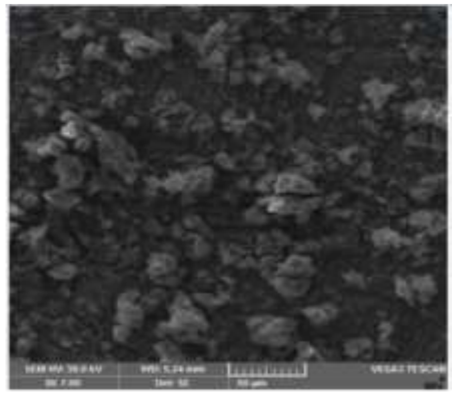


Fig (d)

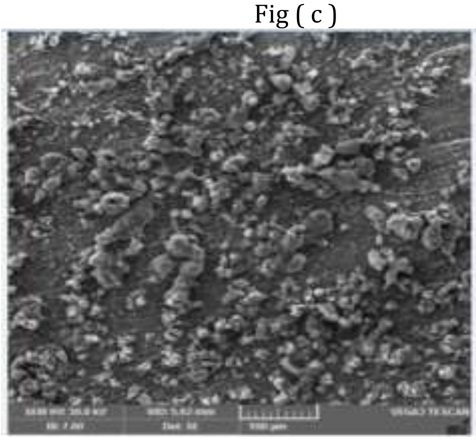


Fig (e).

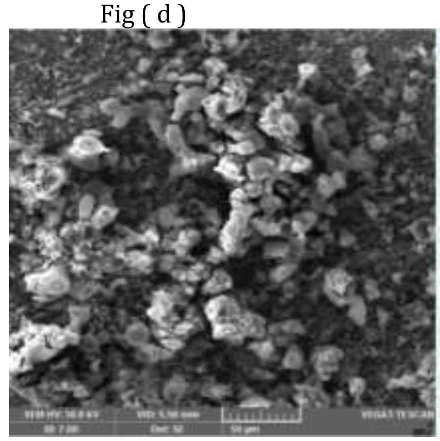


Fig (f)

Figure 3. SEM Analysis of
(a) Polished mild Carbon Steel; Reference Sample (100m)
(b) Polished mild Carbon Steel; Reference Sample (50m)
(c) Mild Carbon Steel immersed in well water; blank (100m)
(d) Mild Carbon Steel immersed in well water; blank (50m)
(e) Mild Carbon Steel immersed in well water +250 ppm L-Tryptophan +50 ppm Zn^{2+} (100m)

4.6. Energy dispersive analysis of X-rays (EDAX)

The EDAX survey spectra were used to determine the elements present on the metal surface before and after exposure to the inhibitor solution. The objective of this section was to confirm the results obtained from chemical and electrochemical measurements that a protective surface film of inhibitor is formed on the metal surface. To achieve this, EDAX examinations of the metal surface were performed in the presence of inhibitors system. They show the characteristic peaks of some of the elements constituting the mild carbon steel sample. The EDAX spectrum of carbon steel immersed in well water containing 250 ppm of L-Tryptophan and 50 ppm of Zn^{2+} is shown in Figure 4. It shows the characteristic lines for the existence of N, and Zn. In addition, the intense C and O signals. The appearance of the N, and Zn signal and this enhancement in C and O signal is due to the presence of inhibitor. These data show that metal surface is covered the N, O, C and Zn atoms. This layer is undoubtedly due to the inhibitor system. Figure 4 shows that the Fe peaks observed in the presence of inhibitor. The suppression of the Fe peaks occurs because of the overlying inhibitor film. This observation indicates the existence of an adsorbed layer of inhibitor that protects steel against corrosion. These results suggest that N, O, and C of L-Tryptophan has coordinated with Fe^{2+}, resulting in the formation of Fe^{2+}-L-Tryptophan acid complex on the anodic sites of metal surface and presence of Zn atoms are precipitated as Zn (OH)2 on the cathodic sites of metal surface.

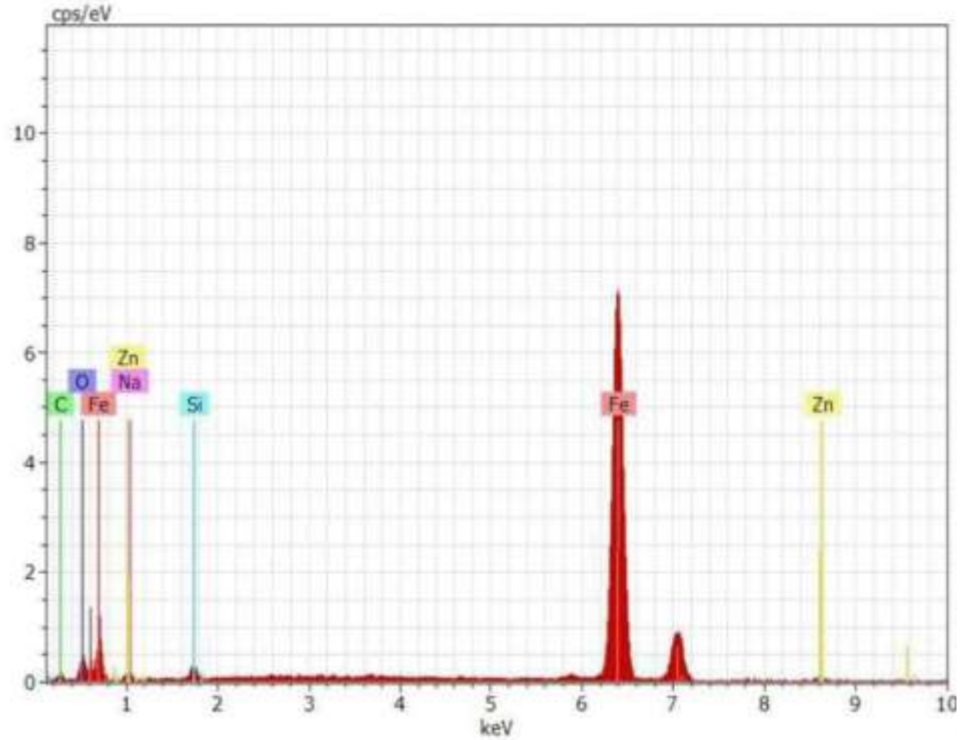


Figure 4. EDAX spectrum of carbon steel immersed in well water containing 250 ppm of L-Tryptophan and 50 ppm of Zn^{2+}

4.6. Mechanism of corrosion inhibition

The results of the weight-loss study show that the formulation consisting of 250 ppm L-Tryptophan and 50 ppm of Zn^{2+} has 91 % IE in controlling corrosion of carbon steel in well water. A synergistic effect exists between Zn^{2+} and L-trytophan FTIR spectra reveals that the protective film consists of Fe-L-tryptophan complex and $Zn(OH)_2$. In order to explain these facts the following mechanism of corrosion inhibition is proposed When the solution containing well water, 50 ppm Zn^{2+} and 250 ppm of L-trytophan acid is prepared, there is formation of Zn^{2+}-L-tryptophan complex in solution. When mild carbon steel is immersed in this solution, the Zn^{2+}L-tryptophan complex diffuses from the bulk of the solution towards metal surface. Zn^{2+}-L- tryptophan complex diffuses from the bulk solution to the surface of the metal and is converted into a Fe^{2+}-L-tryptophan complex, which is more stable than Zn^{2+}-L-tryptophan . On the metal surface Zn^{2+}-L-tryptophan complex is converted in to Fe^{2+}-L-tryptophan acid on the anodic sites. $Zn^{2+}$is released.

Zn^{2+}-L-tryptophan+ Fe^{2+} /Fe^{2+}-L-tryptophan+ Zn^{2+}

The released Zn2+ combines with OH^- to form $Zn(OH)_2$ on the cathodic sites.

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

Thus the protective film consists of Fe2+-L-tryptophan complex and $Zn(OH)_2$.[16]

5. CONCLUSION

A Synergistic effect exists between L-tryptophan and Zn^{2+}in controlling corrosion of carbon steel immersed in well water. The formation consisting of 250 ppm of L- Tryptophan and 50 ppm of Zn^{2+} provided 91% IE. The formation of immersion period on the above inhibitor systems has been studied. Polarization study reveals that this formation functions as a mixed type of inhibitor system. The protective film on carbon steel is studied by UV and IR spectrum studies. The morphology of the metal is studied by SEM, EDAX images.

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