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Anti-Corrosive Effect of Tridax Procumbens – Zn^{2+} System Controlling the Corrosion of Carbon Steel

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ABSTRACT
The corrosion inhibition efficiency (IE) of an aqueous extract Tridax Procumbens(TP) in controlling the corrosion of carbon steel aqueous medium containing 60 ppm of chloride ions in absence and presence of Zn^{2+} has been studied by weight loss method. The formulation consisting of 1 ml of Tridax Procumbens extract and 150 ppm of Zn^{2+} offers 96% inhibition efficiency. The synergistic effect exists between Tridax Procumbens and Zn^{2+} system. Polarization study shows that the Tridax Procumbens – Zn^{2+} system function as a cathodic inhibitor. AC impedance spectra reveal that a protective film formed on the surface. The Adsorption equilibrium exhibited better fit to Langmuir isotherm than Freundlich isotherm. FTIR spectra reveal that the protective film consists of Fe^{2+}-Tridax Procumbens and $Zn(OH)_2$.

Key Words : Carbon steel, Tridax Procumbens, Corrosion inhibitor, Inhibition efficiency, protective film.

I. INTRODUCTION

Corrosion is a natural phenomenon involving the reversion from metallic to compound state. The corrosion occurs because of the natural tendency for most metals to return to their natural state. It cannot be avoided, but it can be controlled and prevented using the suitable preventive measures such as alloying, cathodic protection, anodic protection, protective coating and application of inhibitors, etc. Among all these techniques inhibitors reduce the aggressiveness of the corrosive environment and forming a protective layer on the metal surface thereby the metal and alloys are prevented from corrosion One way of protecting metal from corrosion is to use corrosion inhibitors. Corrosion inhibitors are widely used in industry to reduce the corrosion rate of metals and alloys in contact with aggressive environment. Most of the corrosion inhibitors are synthetic chemicals, expensive and very hazardous to environment. Therefore, it is desirable to source for environmentally safe inhibitors [1–4]. It has been shown that natural products of plant origin contain different organic compounds (e.g., alkaloids, tannins, pigments, and organic and amino acids, and most are known to have inhibitive action [5, 6]). The aqueous extracts from different parts of some plants such as Henna, Lawsonia inermis [7], Rosmarinous officinalis L. [8], Carica papaya [9], cordia latifoliaand curcumin [10], date palm, phoenix dactylifera, henna, lawsonia inermis, corn, Zea mays [11], Atropa Belladonna Extract[12] and Nypa Fruticans Wurmb [13] have been found to be good corrosion inhibitors for many metals and alloys. Recently, an excellent review about "natural products as corrosion inhibitors for metals in corrosive media" has been published [14]. So the less toxic inhibitors such as molybdate, sodium gluconate, citrate, amino acids, and green inhibitors like seed, stem, flower of plant extracts are being used as corrosion inhibitors. So the research is focus on Tridax Procumbens as corrosion inhibitors.

The present work is undertaken:
1. To evaluate the inhibition efficiency (IE) of an aqueous extract Tridax Procumbens (TP) in controlling the corrosion of carbon steel in aqueous medium containing 60 ppm of chloride ions in the absence and presence of Zn^{2+}
2. To analysis the protective film formed on the carbon steel by FTIR spectra.

3. To understand the mechanistic aspects of corrosion inhibition by polarization studies and AC impedance analysis
4. To analysis the protective film formed on the carbon steel by Langmuir and Freundlich isotherm
5. To propose a suitable mechanism for corrosion inhibition.

II. EXPRIMENTAL

II.1. Preparation of plant extract

An aqueous extract of Tridax-procumbens was prepared by grinding 50g of Tridax-procumbens with double distilled water, filtering the suspending impurities, and making up to 500ml. The extract was used as corrosion inhibitor in the present study.

II.2. Preparation of the specimen

Carbon steel (0.026%S, 0.06%P, 0.4%Mn, 0.1% C, and the rest Fe) specimen of dimension 1 cm x 4 cm x 0.2 cm were used for weight loss study. Carbon steel rod of the same composition, encapsulated in Teflon was polished to a mirror finish and degreased with trichloroethylene.

II.3. Weight loss method

Carbon steel specimens in triplicate were immersed in 100 ml of distilled water containing 60 ppm of chloride ions with test solution containing various concentrations of the inhibitors, in the absence and presence of and Zn^{2+} ions, for a period of three days. The weight of the specimens before and after immersion was determined using Shimadzu balance, AY62 model. The corrosion products were cleansed with Clarke's solution[15]. From the change in weight of the specimens, corrosion rates were calculated with the help of the following relationship:

where

CR - corrosion rate
Δm - loss in weight (mg)
A - surface area of the specimen (dm^2)
t - period of immersion (days)]

The inhibition efficiency (*IE*) was then calculated using the equation

$$IE = 100 \left(1 - \frac{W_2}{W_1} \right) \qquad (2)$$

Where W_1 and W_2 are the corrosion rates in the absence and presence of the inhibitor, respectively.

II.4.Polarization study

Polarization studies were carried out in on H&CH electrochemical work station impedance analyzer model CHI 660A. A three electrode cell assembly was used. The working electrode was carbon steel. A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode. According to the stern-Geary equation, the steps of the linear polarization plot are substituted to get corrosion current

$$I_{corr} = b_a \times b_c / 2.303 (b_a + b_c) R_p$$

Where R_p is polarization resistance.

II.5. AC impedance study

The instrument used polarization was also used for AC impedance study. The cell set up was the same as that used for polarization measurements. The real part and imaginary part of the cell impedance were measured in ohms at various frequencies. The values of charge transfer resistance(R_t) and the double layer capacitance(C_{dl}) were calculated.

$$R_t = (R_s + R_t) - R_s$$

Where, R_s = solution resistance

$$C_{dl} = ½ \pi R_t f_{max}$$

Where, f_{max} = maximum frequency

II.6. Surface examination

The carbon steel specimens were immersed in various test solutions for a period of Three days, taken out and dried. The nature of the film formed on the surface of metal specimens was analyzed by FTIR spectroscopic and adsorption studies.

II.7. Surface coverage area

$$1/\theta = \theta/1-\theta, \ 1/C-\theta$$
$$C/\theta = C/1-\theta/\theta.B$$

Where, θ =surface coverage area of metal ion solution at equilibrium,
C = equilibrium concentration of the metal ion,
$1-\theta$ = rate of adsorption,
B = constant related to the energy of adsorption.

II.7.1. Langmuir Adsorption isotherm model

The Langmuir model was developed based on assumption of the formation of a monolayer of the metal ion solution onto the surface of the Tridax Procumbens, It has also been assumed that surface sites are completely heterogeneous. The study of Langmuir isotherm is essential in assessing the adsorption efficiency of the Tridax Procumbens. In this regard the Langmuir isotherm is important, through the restriction and the limitation if this model has been well recognized.

II.7.2. Freundlich Adsorption isotherm Model

The Freundlich isotherm is the earliest known relationship describing the sorption equation. The fairly satisfactory empirical isotherm can be used for non-ideal sorption that involves heterogeneous surface energy system and is expressed by the following equation

$$IE = K_F TP^{1/n}$$

Where K_F is roughly an indicator of the adsorption capacity and $1/n$ is the adsorption intensity. The essential characteristics of Langmuir and freundlich isotherm can be expressed in terms of dimension less constant, separate ion factor or equilibrium parameter R_L, which is defined by $R_L = C_o / 1+bC_o$ is the initial metal ion concentration and b is the Langmuir constant the parameter indicates the shape of isotherm as follows.

R_L	Types of isotherm
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

The applicability of Langmuir and Freundlich model to then chosen metal ion solution system was studied in the present work.

II.8. FTIR spectra

FTIR spectra were recorded in a Perkin – Elmer 1600 spectrophotometer. The film was carefully removed, mixed thoroughly with KBr made in to pellets and FTIR spectra were recorded.

III. RESULTS AND DISCUSSION
III.1. Analysis of Results of Mass Loss Method

The corrosion inhibition efficiency of carbon steel immersed in an aqueous solution containing 60 ppm of chloride ions in the absence and presence of inhibitor systems are given in Table 1 and Table 2. It is seen from Table 1. that the aqueous extract of Tridax Procumbens (TP) alone is good inhibitor. As concentration TP increases, IE slightly increases. That is at higher concentrations, TP accelerates corrosion. For example 1 ml of TP shows -1% IE; But addition of 150 ppm of Zn^{2+} with Tridax Procumbens (TP) shows 96% IE. This suggests that a synergistic effect exists between TP – Zn^{2+} system[16].

Table 1. Corrosion inhibition efficiency (IE) of

S.No.	TP Extract ml	Zn^{2+} 0 (ppm) IE%	Zn^{2+} 50 (ppm)) IE%	Zn^{2+} 100 (ppm) IE%	Zn^{2+} 150 (ppm) IE%
1	0	-	3	5	9
2	1	-1	-23	16	96
3	2	07	01	40	81
4	3	08	05	37	75
5	4	17	07	47	88
6	5	14	44	65	89
7	6	26	54	71	93

carbon steels in 60 ppm Chloride solution in the presence of inhibitor obtained by weight loss method. Inhibitor system : TP- Zn^{2+} system.

Table 2. Corrosion inhibition efficiency (IE) of

S.No.	TP Extract ml	Zn^{2+} (ppm)	IE %
1	0	-	-
2	1	150	96
3	2	150	96
4	3	150	96
5	4	150	96
6	5	150	96
7	6	150	96

carbon steels in 60 ppm Chloride solution in the presence of inhibitor obtained by weight loss method. Inhibitor system : TP- Zn^{2+} system.

III.2. Influence of TP on the inhibition efficiency with Zn^{2+} system

The influence of TP on the inhibition efficiency with Zn^{2+} system is given Table 2. When various concentration of TP added to the TP –Zn^{2+} system, the inhibition efficiency does not altered. The Zn^{2+} - TP systems are much transported towards the metal surface, hence protective film is stable.

III.3. Polarization curves

The polarization curves of carbon steel immersed in various test solution are shown in Figure 1. The corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes (b_a and b_c) and Linear polarization resistance (LPR) given in the Table 3.
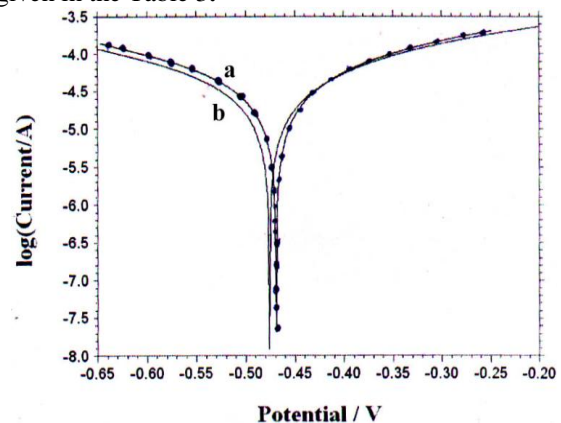

Figure 1 polarization curves of carbon steel immersed in various test solution
(a) Aqueous solution contains 60 ppm Cl^-
(b) 150 ppm of Zn^{2+} + 1 ml of TP

Table 3. Corrosion parameter of carbon steel immersed in 60 ppm Chloride solution in the absence and presence of inhibitors

P (ml)	Zn^{2+} ppm	E_{corr} mv vs SCE	b_a mv/ decade	b_c mv/ decade	LPR Ω cm^2	I_{corr} A/ cm^2
0	0	-469	529	494	1317	3.22×10^{-5}
1	150	-476	531	494	1509	2.81×10^{-5}

When carbon steel immersed in aqueous solution containing 60 ppm Cl⁻ ions, the corrosion potential is -469 mv vs SCE. The formulation consisting of 150 ppm of Zn^{2+} and 1 ml of TP has corrosion potential of -476 mv vs SCE. The corrosion potential shift is very small. The Tafel slopes b_a and b_c for the system are almost equal (531mv/decade and 494 mv/decade). These results suggest that TP - Zn^{2+} system formulation functions as mixed inhibitor but it is controlled predominately as the cathodic reaction.

The formation of protecting film on the metal surface is further supported by the fact that the linear polarization resistance (LPR) value increases from 1317 Ω cm^2 to 1509 Ω cm^2. The corrosion current decreases from 3.22×10^{-5} A/cm^2 to 2.81×10^{-5} A/cm^2. Thus the polarization study confirms the formation of protecting film on the metal surface[17]

III.4. Analysis of AC impedance spectra

The AC impedance spectra of carbon steel immersed in various test solution and shown in Figure 2. The AC impedance parameters namely, charge transfer resistance(R_t) and the double layer capacitance(C_{dl}) are given in the Table 4.
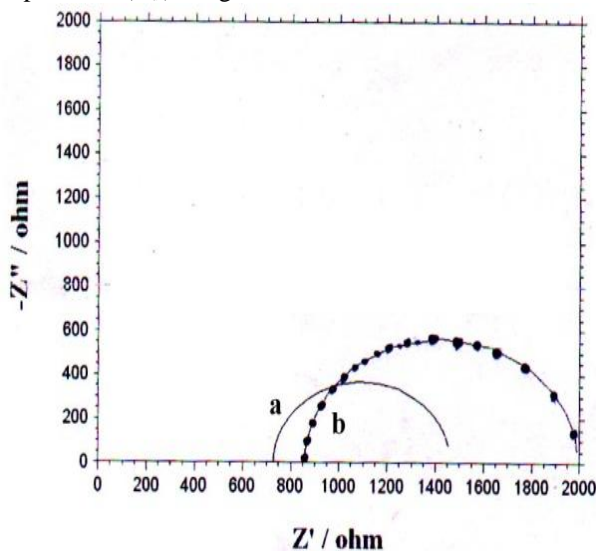

Figure 2. AC impedance of carbon steel immersed in various test solution

(a) Aqueous solution contains 60 ppm Cl⁻
(b) 150 ppm of Zn^{2+} + 1 ml of TP

When carbon steel immersed in aqueous solution containing 60 ppm Cl⁻ ions, the R_t value is found to be 729 Ωcm^2. The C_{dl} value is 5.828×10^{-7} F/cm^2. When 150 ppm of Zn^{2+} and 1 ml of TP are added, the R_t value increased from 729 to 1130 Ωcm^2 and the C_{dl} value has decreased from 5.828×10^{-7} to 2.443×10^{-7} F/cm^2. The increase in R_t values obtained from impedance studies justify the good performance of a compound as an inhibition in aqueous medium. This behaviour means that the film obtained act as a barrier to the corrosion process that clearly proves the formation of the film[18].

Table 4. AC impedance parameters of carbon steel immersed in 60 ppm Chloride solution in the absence and presence of inhibitors.

TP (ml)	Zn^{2+} ppm	R_t Ω cm^2	C_{dl} F/cm^2
0	0	729	5.828×10^{-7}
1	150	1130	2.443×10^{-7}

III.5. Langmuir adsorption isotherm Model

The Langmuir adsorption model is based on the assumption that maximum adsorption corresponds to the saturated monolayer of Langmuir equation can be described by

$$C/\theta = 1/\theta.B + (1/\theta)$$

Where C is the equilibrium concentration of the metal ion solution, θ is the surface coverage area of the metal ion, B is the Langmuir constant related to adsorption capacity and rate of adsorption, respectively. The linear plot of specific adsorption C/θ against the equilibrium concentration (C) Figure 3. Shows that the adsorptions obey the Langmuir model. The Langmuir constant B were determined from the slope and intercept of the plot and are presented in Table 5. The R^2 values (0.9566) suggest that the Langmuir isotherm provides a good fit to the isotherm data
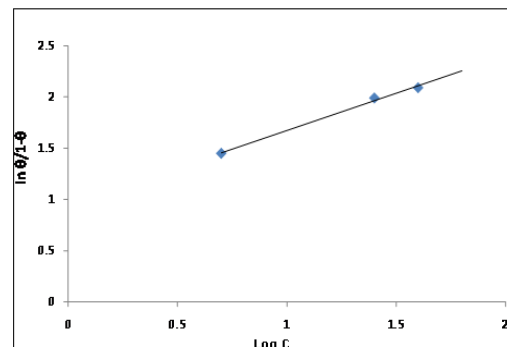

Figure 3.Langmuir isotherm for Zn^{2+} Solution on to Tridax Procumbens

The essential characteristics if the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor R_L given by the equation

$$R_L = 1/1 + bc_o$$

Where C_0 is the highest initial concentration of Tridax Procumbens and b is Langmuir constant.The parameter R_L, indicates the nature of shape of isotherm accordingly.

Table 5. Langmuir Isotherm for Zn^{2+} Solution on Tridax Procumbens

Zn^{2+} ppm	TP (ml)	ln C	$\square$	$\square$ /$\square$	ln $\square$/$\square$	CR
150	1	0.0	0.96	24.00	3.18	0.90
150	2	0.7	0.81	4.26	1.45	05
150	3	1.1	0.75	3.00	1.10	07
150	4	1.4	0.88	7.33	1.99	03
150	5	1.6	0.89	8.09	2.09	03
150	6	1.8	0.93	13.29	2.59	01

III.6. Freundlich adsorption isotherm Model

The Freundlich isotherm (Table 6) is the earliest known relationship describing the sorption equation.

Table 6.Freundlich isotherm for Zn^{2+} solution on Tridax Procumbens

Zn^{2+} (ppm)	TP (ml)	IE %	CR (mdd)	log TP	log IE %
150	1	96	0.90	0.00	1.98
150	2	81	05	0.30	1.91
150	3	75	07	0.48	1.88
150	4	88	03	0.60	1.94
150	5	89	03	0.70	1.95
150	6	93	01	0.78	1.97

The fairly satisfactory empirical isotherm can be used for non-ideal sorption that involves heterogeneous surface energy system and is expressed by the following equation

$$IE = K_F TP^{1/n}$$

Where K_F is roughly an indicator of the adsorption capacity and 1/n is the adsorption intensity. In general as the K_F value increases the adsorption capacity of Tridax Procumbens. Value n>1 represent favourable adsorption condition[19]. The Linear form of equation is given below

$$\log IE\% = \log K_F + (1/n) \log TP$$

Values of K_F and n are calculated from the intercept and slope of the plot Figure 4. and are listed in Table 7. The R^2 value (0.0372) is lower than Langmuir isotherm.
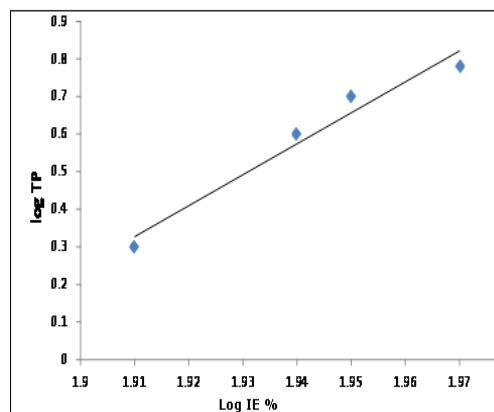

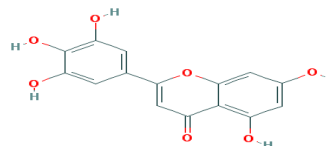Figure 4.Freundlich isotherm for Zn^{2+} solution on to Tridax Procumbens

Table 7. Langmuir and Freundlich isotherm constant and correlation Co-efficient for Adsorption of Zn^{2+} Solution Concentration

Langmuir isotherm	
K_F	0.000
1/n	2.000
R^2	0.9566
R_L	0.1851
Freundlich isotherm	
K_F	0.030
1/n	8.000
R^2	0.0372
R_L	0.1424

The best equilibrium model is determined based linear square regression correlation co-efficient R^2 from Figure 5, It was observed that the equilibrium sorption data were very best fit isotherm expression conform the monolayer coverage process of Tridax Procumbens on to Zn^{2+}.

III.7. Analysis of FTIR spectra

The active component present in Tridax Procumbens is shown in scheme 1.

Scheme 1

A few drops of an aqueous extract of aqueous extract of Tridax Procumbens(TP) was dried on a glass plate. A solid mass was obtained. Its spectrum is shown in figure 5a. The peak at 3385 cm^{-1} corresponds to -OH Stretching frequency, the peak at 1651 cm^{-1} corresponds to >C=O Stretching frequency.
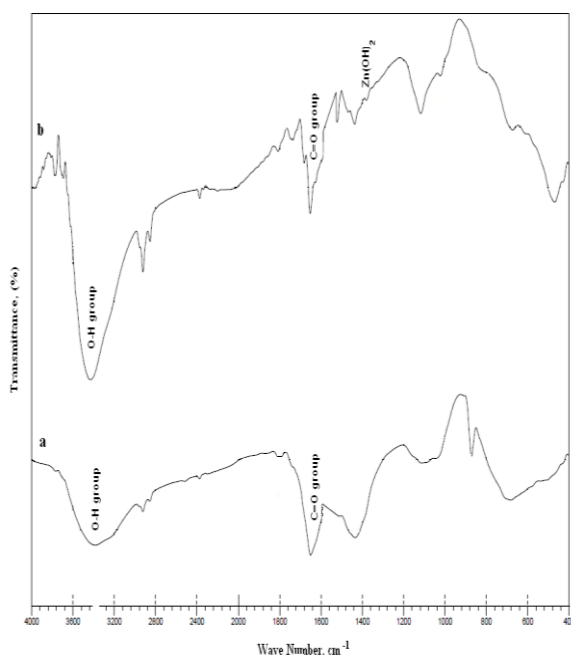
Figure 5. FTIR spectrum of various test solution
    (a) Pure TP
    (b) Film formed on carbon steel after immersion of test solution containing 150 ppm of Zn^{2+} + 1ml TP

FTIR spectrum of thin film formed on the metal surface, after the immersion in 150 ppm of Zn^{2+} and 1 ml of TP solution in shown in figure 5b. The –OH stretching frequency shifted from 3385 cm^{-1} to 3426 cm^{-1}, and the >C=O stretching frequency shifted from 1651 cm^{-1} to 1653 cm^{-1}.This indicates that the oxygen atom of carbonyl group has coordinated with Fe^{2+}-TP complex on the anodic sites of the metal surface. The peak at 1381 cm^{-1} due to the $Zn(OH)_2$ formed on the cathodic sites. The FTIR spectrum confirmed that the protective film consist of Fe^{2+}-TP complex and $Zn(OH)_2$ on the metal surface[20,21].

IV. MECHANISM

The weight – loss study reveals that the formulation consisting of 150 ppm of Zn^{2+} and 1ml of Tridax Procumbens has 96 % inhibition efficiency. The FTIR spectrum reveals that the protective film consist of Fe^{2+} - TP complex and $Zn(OH)_2$.

In order to explain the above observations, the following mechanism of corrosion inhibition is proposed[21].
1. When the environment consisting of 150ppm of Zn^{2+} and 1ml of TP is prepared, there is a formation of Zn^{2+} -TP complex.
2. When Carbon steel is introduced in this solution there is diffusion of Zinc complex towards the metal surface.

3. On the metal surface Zinc complex is converted into iron complex on the anodic site.
$$Zn^2\text{-TP} + Fe^{2+} \rightarrow Fe^{2+}\text{-TP} + Zn^{2+}$$
4. The released Zn^{2+} combined with OH- to form $Zn(OH)_2$ on the cathodic Sites.
$$Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2\downarrow$$
5. Thus protective film consists of Fe^{2+} - TP and $Zn(OH)_2$.

V. CONCLUSION

The present study leads to the following conclusions:
- The formulation consisting of 1 ml Tridax Procumbens extract and 150 ppm of Zn^{2+} offers 96% inhibition efficiency;
- The synergistic effect exists between Tridax Procumbens – Zn^{2+} system;
- Polarization study reveals that this formulation controls the cathodic reaction predominantly;
- AC impedance spectra reveal that a protective film is formed on the metal surface;
- The value of the separation factor R_L, indicated the Tridax Procumbens system was favourable adsorption.
- FTIR spectra reveal that the protective film consists of Fe^{2+} - Tridax Procumbens complex and $Zn(OH)_2$

VI. ACKNOWLEDGEMENT

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