

# Synergistic Effect of *Allium cepa*-Zn<sup>2+</sup> System on the Corrosion of Carbon Steel in Ground Water

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## ABSTRACT

The corrosion inhibition efficiency (IE) of an aqueous extract *Allium cepa* (onion) in controlling the corrosion of carbon steel ground water in absence and presence of with Zn<sup>2+</sup> has been studied by weight loss method. The formulation consisting of 3 mL *Allium cepa* extract, 50 ppm of Zn<sup>2+</sup> and 50 ppm of sodium potassium tartarate which offers 97% inhibition efficiency. The synergistic effect exists between onion-Zn<sup>2+</sup>-tartarate system. The addition of N-cetyl-N,N,N-trimethylammonium bromide on onion-Zn<sup>2+</sup>-tartarate system does not change the excellent inhibition efficiency. Polarization study shows that the onion-Zn<sup>2+</sup>-tartarate system functions as a cathodic inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. The UV fluorescent spectra indicate the possibility of formation of Fe<sup>2+</sup>-onion complex and also Zn<sup>2+</sup>-onion complex in solution. Thus the protective film is found to be UV fluorescent.

**Keywords:** Carbon Steel; *Allium cepa*; Corrosion Inhibition Efficiency; Ground Water

## 1. Introduction

The toxic inhibitors like chromate based inhibitors were used to control corrosion process but it creates the environmental hazards. The use of chromates at high concentration has declined in recent years because of health and safety considerations. Natural products are nontoxic, biodegradable and readily available. The recent trend in research on environmental friendly corrosion inhibitors is taking us back to exploring the use of natural products as possible sources of cheap, nontoxic, and eco-friendly corrosion inhibitors. These natural products are either synthesized or extracted from aromatic herbs, spices, and medicinal plants [1-7]. Of increasing interest is the use of medicinal plant extracts as corrosion inhibitors for metals in acid solutions. This is because these plants serve as incredibly rich sources of naturally synthesized chemical compounds that are environmentally acceptable, inexpensive, readily available, and renewable sources of materials [8,9]. These chemicals include alkaloids, flavonoids, terpenoids, glycosides, tannins, saponins, fats and oils, and carbohydrates, and so forth [10-18]. Several plants extracts [19-23] and eco-friendly inhibitors attracted the researchers. Investigation of natural inhibitors

is particularly interesting because they are non-expensive, ecologically friendly/acceptable and possess no threat to the environment. Onions not only provide flavor; they also provide health-promoting phytochemicals as well as nutrients. Onion contains an acid, volatile principle that stimulates the tear glands and the mucous membranes of the respiratory tract. All the healthy compounds present in onions, two stand out: sulfur and quercetin-both being strong antioxidants. The present work is undertaken:

1) To evaluate the inhibition efficiency (IE) of an aqueous extract onion (AC) in controlling the corrosion of carbon steel in ground water, in the absence and presence of Zn<sup>2+</sup>.

2) To examine the influence of N-cetyl-N,N,N-trimethylammonium bromide (CTAB) on the IE of the AC-Zn<sup>2+</sup> system.

3) To analyze the protective film formed on the carbon steel by UV Fluorescence spectra.

4) To understand the mechanistic aspects of corrosion inhibition by polarization studies and AC impedance analysis and;

5) To propose a suitable mechanism for corrosion inhibition.

## 2. Experimental

### 2.1. Preparation of Plant Extract

An aqueous extract of *Allium cepa* (onion) was prepared by grinding 10 g of onion with double distilled water, filtering the suspending impurities, and making up to 500 mL. The extract was used as corrosion inhibitor in the present study.

### 2.2. Preparation of Specimens

The carbon steel specimens were drilled a hole at one end and numbered by punching. Carbon steel specimens (0.0267% S, 0.06% P, 0.4% Mn, 0.1% C and the rest iron) of dimensions 1.0 cm × 4.0 cm × 0.2 cm were polished with 400 grade emery paper to a mirror finish and degreased with trichloroethylene.

### 2.3. Weight-Loss Method

Relevant data on the ground water used in this study are given in **Table 1**. Carbon steel specimens in triplicate were immersed in 100 mL of the solutions containing various concentrations of the inhibitor in the presence and absence of  $Zn^{2+}$  for one day. The weight of the specimens before and after immersion was determined using Shimadzu balance model AY 62. The corrosion products were cleansed with Clarke's solution [24]. The inhibition efficiency (IE) was then calculated using the equation

$$IE = 100 \left[ 1 - \left( \frac{W_2}{W_1} \right) \right] \%$$

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

### 2.4. Polarization Study

Polarization studies were carried out in an H & CH electrochemical work station impedance analyzer model CHI 660 A. 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.

Determination of inhibition efficiency.

By Tafel method

$$IE = I_{corr} - I_{corr(1)} / I_{corr} \times 100$$

where,

$I_{corr}$  is corrosion current without inhibitor.

$I_{corr(1)}$  is corrosion current with inhibitor.

The results, such as Tafel slopes, and  $I_{corr}$  and  $E_{corr}$  values, were calculated. During the polarization study, the scan rate (v/s) was 0.01; hold time at  $E_f$ (s) was zero and quiet time(s) was 2.

### 2.5. 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 the 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

$$C_{dl} = 1/2 \pi R_t f_{max}$$

where  $f_{max}$  = maximum frequency

AC impedance spectra were recorded with initial  $E(v) = 0$ ; high frequency (Hz) =  $1 \times 10^5$ ; low frequency (Hz) = 1; amplitude (v) = 0.05; and quiet time (s) = 2.

### 2.6. Surface Examination

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

### Fluorescence Spectra

These spectra were recorded in a Hitachi F-4500 fluorescence spectrophotometer.

## 3. Results and Discussion

### 3.1. Analysis of Results of Mass Loss Method

The corrosion inhibition efficiency of carbon steel immersed in ground water in the absence and presence of inhibitor systems are given in **Tables 3**. The inhibition efficiencies (IE) are also given in these Tables. It is seen from **Table 2** that the aqueous extract of *Allium cepa* (AC) alone is poor inhibitor. As concentration AC in-

**Table 1. Parameters of ground water.**

Parameter	Value
pH	7.38
Conductivity	512 micromhos/cm
TDS	373 ppm
Chloride	72 ppm
Sulphate	12 ppm
Total hardness	112 ppm

**Table 2. Corrosion inhibition efficiency (IE) of Carbon steel in aqueous solution in the presence of inhibitor obtained by weight loss method.**

AC Extract mL	Zn <sup>2+</sup>	SPT	Zn <sup>2+</sup>
	0 (ppm)	50 (ppm)	50 (ppm)
	IE%	IE%	IE%
0	-	8	22
1	-42	68	70
2	-37	70	82
3	-50	74	97
4	-64	69	96

Inhibitor system: AC + SPT + Zn<sup>2+</sup> system.

**Table 3. Corrosion inhibition efficiency (IE) of Carbon steel in aqueous solution in the presence of inhibitor obtained by weight loss method.**

AC Extract mL	Zn <sup>2+</sup> ppm	SPT ppm	CTAB ppm	IE %
3	50	50	0	97
3	50	50	50	97
3	50	50	100	97
3	50	50	150	97
3	50	50	200	97

Inhibitor system: AC + SPT + CTAB + Zn<sup>2+</sup> system.

creases, IE slowly decreases. That is, at higher concentrations, AC accelerates corrosion. It favours dissolution of carbon steel in ground water. For example, 3 mL of AC shows -50% IE; But addition of 50 ppm of Sodium Potassium Tartarate (SPT) with *allium cepa* shows 74% IE. The formulation consisting 3 mL of AC, 50 ppm Zn<sup>2+</sup> and 50 ppm SPT shows 97% IE. This suggests that a synergistic effect exists between AC-SPT-Zn<sup>2+</sup> system [25].

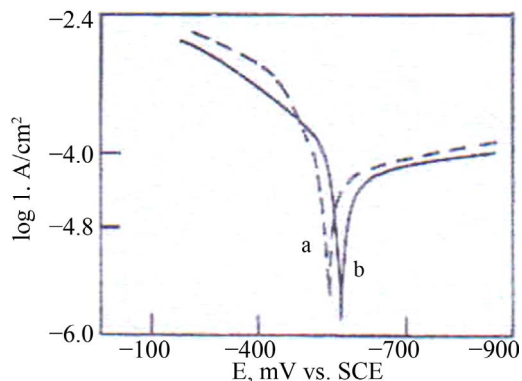
### 3.2. Influence of CTAB on AC-SPT-Zn<sup>2+</sup> System

The Influence of CTAB on AC-SPT-Zn<sup>2+</sup> system is given in **Table 3**. The N-cetyl-N,N,N-trimethylammonium bromide (CTAB) is a biocide. It can control the corrosion caused by microorganism [26]. When various concentration of CTAB added to the AC-SPT-Zn<sup>2+</sup> system, the inhibition efficiency does not altered. The AC-SPT-Zn<sup>2+</sup> system are much transported towards the metal surface, hence protective film is stable.

### 3.3. Analysis of Polarization Curves

The potentiodynamic polarization curves of carbon steel immersed in ground water in the absence and presence of inhibitors are shown in **Figure 1**.

The corrosion parameters are given in **Table 4**. When carbon steel is immersed in ground water the corrosion potential is -538 mV vs. SCE (Saturated Calomel Electrode). The formulation consisting of 3 mL of AC, 50 ppm of SPT and 50 ppm of Zn<sup>2+</sup> shifts the corrosion potential to -563 mV vs SCE.



a) Ground water; b) 3 mL AC + 50 ppm of Zn<sup>2+</sup> + 50 ppm SPT.

**Figure 1. Polarization curves of carbon steel immersed in various solutions.**

**Table 4. Corrosion parameters of carbon steel immersed in ground water in the absence and presence of inhibitors.**

SPT ppm	AC mL	Zn <sup>2+</sup> ppm	E <sub>corr</sub> mV vs. SCE	b <sub>a</sub> mV/decade	b <sub>c</sub> mV/decade	I <sub>corr</sub> A/cm <sup>2</sup>
0	0	0	-538	73	260	3.981 × 10 <sup>-5</sup>
50	3	50	-563	117	70	3.162 × 10 <sup>-5</sup>

Inhibitor system: AC + SPT + Zn<sup>2+</sup> system.

This suggests that the cathodic reaction is controlled predominantly. But Tafel slopes (b<sub>a</sub> & b<sub>c</sub>) are not shifted equally [27,28]. The corrosion current is 3.981 × 10<sup>-5</sup> A/cm<sup>2</sup> to 3.162 × 10<sup>-5</sup> A/cm<sup>2</sup>. This suggests the inhibitive nature of this inhibitor system.

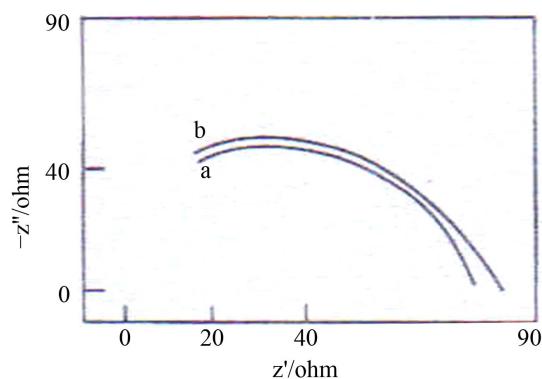
### 3.4. Analysis of AC Impedance Spectra

The AC impedance spectra of carbon steel immersed in ground water, in the absence and presence of inhibitors are shown in **Figure 2**.

The AC impedance parameters such as charge transfer resistance (R<sub>t</sub>) and double layer capacitance (C<sub>dl</sub>) are given in **Table 5**. When carbon steel is immersed in ground water, the charge transfer resistance (R<sub>t</sub>) is 60.06 ohm·cm<sup>2</sup>; the double layer capacitance C<sub>dl</sub> is 3.27 × 10<sup>-8</sup> F/cm<sup>2</sup>. When carbon steel is immersed in the formulation consisting of AC-SPT-Zn<sup>2+</sup>, the R<sub>t</sub> value increases and C<sub>dl</sub> value decreases. This confirms that a protective film is formed on the metal surface. This accounts for very high inhibition efficiency [29-32].

### 3.5. Analysis of Fluorescence Spectra

A few drops of the AC extract were dried on a glass palte. A red solid mass was obtained. Its emission spectrum (λ<sub>ex</sub> = 300 nm) is shown in **Figure 3(a)**. two prominent peaks appeared at 361 nm and 505 nm. A few drops of the AC extract were mixed with a few drops of freshly prepared Fe<sup>2+</sup> ions (ferrous sulphate). Fe<sup>2+</sup>-AC complex



a) Ground water; b) 3 mL AC + 50 ppm of  $Zn^{2+}$  + 50 ppm + SPT.

**Figure 2. AC impedance spectra of carbon steel immersed in various solutions.**

**Table 5. AC impedance parameters of carbon steel immersed in ground water in the absence and presence of inhibitors.**

SPT ppm	AC mL	$Zn^{2+}$ ppm	$R_t$ ohm·cm <sup>2</sup>	$C_{dl}$ F/cm <sup>2</sup>
0	0	0	60.06	$3.27 \times 10^{-8}$
50	4	50	68.22	$2.88 \times 10^{-8}$

was stormed. It was dried. Its emission spectrum ( $\lambda_{ex} = 300$  nm) is shown in **Figure 3(b)**. The intensity of the peak at 361 nm decreased. The peak at 505 nm disappeared.

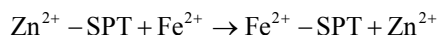
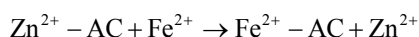
The emission spectrum ( $\lambda_{ex} = 300$  nm) of the ground film formed on surface of the metal after immersion in the solution containing ground water, 3 mL of AC and 50 ppm of  $Zn^{2+}$ , is shown in **Figure 3(c)**. The nature of the spectrum matched well with that of the  $Fe^{2+}$ -AC complex prepared. The excitation spectra ( $\lambda_{ex} = 361$  nm) corresponding to **Figures 3(a)**-(**c**), are shown in **Figures 3(d)**-(**f**), respectively. A peak appeared at 265 nm, in all the cases. This confirmed the presence of  $Fe^{2+}$ -AC complex formed on the anodic sites of the metal surface [33-35].

#### 4. Mechanism

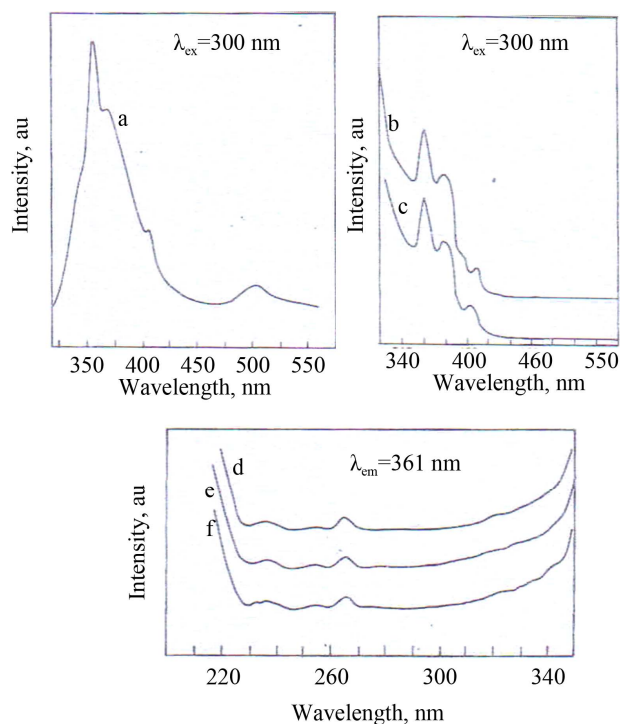
1) When the environment consisting of 50 ppm of  $Zn^{2+}$  and 50 ppm of SPT + 3 mL of AC is prepared, there is a formation of  $Zn^{2+}$ -AC complex and  $Zn^{2+}$ -SPT complex in solution.

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.

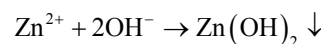


4) The released  $Zn^{2+}$  combined with  $OH^-$  to form  $Zn(OH)_2$  on the cathodic sites.



a) and d) Emission and excitation spectra of solid mass obtained by evaporating onion extract; b) and e) Emission and excitation spectra of solid  $Fe^{2+}$ -AC complex prepared; c) and f) Emission and excitation spectra of film formed on surface of carbon steel specimen after immersion in ground water containing 3 mL onion extract and 50 ppm of  $Zn^{2+}$  + 50 ppm SPT.

**Figure 3. Fluorescence spectra.**



5) Thus protective film consists of  $Fe^{2+}$ -SPT complex,  $Fe^{2+}$ -AC complex and  $Zn(OH)_2$  [36].

#### 5. Conclusions

The present study leads to the following conclusions:

- The formulation consisting of 3 mL *allium cepa* (onion) extract, 50 ppm of  $Zn^{2+}$  and 50 ppm of SPT offers 97% inhibition efficiency;
- The synergistic effect exists between onion- $Zn^{2+}$ -tartrate system;
- The addition of CTAB (a biocide) on onion- $Zn^{2+}$ -tartrate system does not change the excellent inhibition efficiency;
- 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 film is found to be UV-fluorescent.

#### 6. Acknowledgements

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