

## **Inhibition Efficiency of Tri Sodium Citrate– Al<sup>3+</sup> Mixed Inhibitors on Mild Steel in Ground Water**

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### **ABSTRACT**

*This exploration means to examine the Corrosion hindrance conduct of Tri Sodium Citrate (TSC) in blend with Aluminium particle (Al<sup>3+</sup>) on the consumption of carbon steel in ground water utilizing weight loss strategy. After effects of weight loss technique demonstrate inhibition efficiency (IE) expanded with expanding inhibitor fixation. Polarization study uncovers that TSC- Al<sup>3+</sup> framework controls the cathodic response prevalently. AC impedance spectra uncover that a defensive film is framed on the metal surface. The idea of the metal surface has been dissected by Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM).*

**Key words:** *Carbon steel, Corrosion, synergistic impact, FTIR, SEM, Tri Sodium Citrate*

### **1. INTRODUCTION**

Corrosion is a naturally occurring phenomenon which deteriorates a metallic material or its properties because of a reaction with its environment. Corrosion can cause dangerous and expensive damage to everything from pipelines, bridges and public buildings to vehicles, water and wastewater systems, and even home appliances. It is one of the most serious problems in the oil and gas industry. The uses of organic and inorganic inhibitors are the most widely practical methods for protection of metals and alloys against corrosion. The efficiency of an organic compound as a corrosion inhibitor is closely associated with the chemical adsorption [1-4]. Most well-known organic inhibitors contain nitrogen, sulfur, and oxygen atoms due to their ability to form an adsorbed protective film at the metal/ media interface. Studies report that the adsorption of organic inhibitors mainly depend on some physicochemical properties of the molecule, related to its functional groups, to the possible steric effects and electronic density of donor atoms: adsorption is supposed also to depend on the possible interaction of P-orbital of the inhibitor with d- orbital of the surface atoms, which induce greater adsorption of the inhibitor molecules onto the surface of carbon steel, leading to the formation of a corrosion protective film [5].

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A survey of the available literature reveals that the Corrosion inhibition of 2-naphthalenesulfonic acid, 2, 7-naphthalenedisulfonic acid and 2-naphthol-3, 6-disulfonic acid on Armco- iron electrode in sulfuric acid has been investigated by Vracar and Drazic. The inhibition efficiency, changes with the number of functional groups substituted on the benzene ring and increases with concentration [6]. The inhibition action of 2-mercaptobenzoxazol, 2- mercapto benzimidazole, N-cetyl pyridinium bromide and propargyl benzene sulphonate on the corrosion of carbon steel in acid media has also been studied by Prakash Rajesh Kumar Singh and Ranju Kumar. The corrosion inhibitors are used to reduce corrosion damage in sub-surface equipments in oil well fields. The corrosion inhibition activity was studied by gravimetric and Potentiostatic polarization methods in presence of 20% HCl [7]. Manickavasagam, et al., has reported the corrosion inhibition of Poly (Styrene sulphonic acid) - doped poly aniline on carbon steel in acid media. The polymer acts as an anodic inhibitor. The adsorption of the compound on the metal surface obeys Temkin's adsorption isotherm [8]. Aliev has described the influence of salts of Alkyl phenol Sulphonic acid on the corrosion of ST3 steel.

The protective effect increases with temperature. The investigated compounds inhibit corrosion of ST3 steel as a result of chemical adsorption [9]. Shakthivel and Vasudevan have studied the effect of Acrylic acid-diphenylamine sulphonic acid copolymer as threshold inhibitor for sulphate and carbonate scales in cooling water systems. The results show that the polymer acts as a very good ant scaling inhibitor both in the carbo nate and sulphate brines. Copolymer of acrylic acid-diphenyl amine sulphonic acid can be used safely in cooling water systems [10]. Perusal of several literatures reveals that there is no information regarding the use of Tri Sodium Citrate (TSC) in combination with aluminium ion ( $Al^{3+}$ ) as corrosion inhibitor. This paper focuses on the IE of TSC in controlling corrosion of carbon steel immersed in dam water in the absence and presence of  $Al^{3+}$ . The investigation is performed using weight loss method, polarization technique and AC impedance spectroscopy. The morphology of the protective film was examined by FTIR and SEM techniques. The medium which is used in the present study is ground water collected from Ramanathapuram (near sea shore area) in the state of Tamil Nadu, India.

## **2. EXPERIMENTAL METHOD**

The chemicals used in this study, Tri Sodium Citrate (TSC) and  $Al^{3+}$  ions (Aluminium sulphate  $Al_2(SO_4)_3 \cdot 16H_2O$ ) co inhibitor were AR grade.

### **2.1. Preparation of the specimen**

Carbon steel specimens of size 1.0 cm × 4.0 cm × 0.2 cm, (area 10 cm<sup>2</sup>) and chemical composition 0.026 % Sulphur, 0.06 % Phosphorous, 0.4 % Manganese, 0.1 % Carbon and the rest iron (density 7.87 gm/cm<sup>3</sup>), were polished to a mirror finish and degreased with trichloroethylene and used for the weight loss method and surface examination studies.

## **2.2. Weight–loss method**

Carbon steel specimens were immersed in 100 ml of the medium containing various concentrations of the inhibitor (TSC) in the absence and presence of Al<sup>3+</sup> for 3 days. The weights of the specimens before and after immersion were determined using a Digital Balance (Model AUY 220 SHIMADZU). The corrosion products were cleaned with Clarke’s solution prepared by dissolving 20 gms of Sb<sub>2</sub>O<sub>3</sub> and 50 gms of SnCl<sub>2</sub> in one litre of Conc.HCl of specific gravity (1.9) [11]. The corrosion IE was then calculated using the equation

$$\text{IE} = 100 [1 - (W_2/W_1)] \% \text{ ----- (1)}$$

**Where,**

W<sub>1</sub> is the weight loss value in the absence of inhibitor,

W<sub>2</sub> is the weight loss value in the presence of inhibitor.

Corrosion rates were calculated using the following relationship.[12]

$$\text{Corrosion rate} = \frac{\text{Loss in weight (mg)}}{\text{Surface area of the specimen (dm}^2\text{) x Period of immersion (days)}} \text{ ----- (2)}$$

The corrosion rate is expressed in mdd units [mdd = mgm/(dm<sup>2</sup>) (day)].

## **2.3 Potentiodynamic Polarization**

Polarization studies were carried out in a CHI– electrochemical work station with impedance model 660A. It was provided with iR compensation facility. A three electrode cell assembly was used. The working electrode was carbon steel. Saturated calomel electrode was the reference electrode. Platinum was the counter electrode. From polarisation study, corrosion parameters such as corrosion potential (E<sub>corr</sub>), corrosion current (I<sub>corr</sub>), Tafel slopes anodic = ba and cathodic = bc were calculated and linear polarization resistance (LPR) was also calculated. The scan rate (V/S) was 0.01. Hold time at E<sub>f</sub> (s) was zero and quiet time was two second s.

## **2.4 AC impedance spectra**

The instrument and cell set up used for polarization study was used to record AC impedance spectra also. The real part (Z') and imaginary part (Z'') of the cell impedance

were measured in ohms at various frequencies. Values of charge transfer resistance ( $R_t$ ) and the double layer capacitance ( $C_{dl}$ ) were calculated.

$$R_{ct} = (R_s + R_{ct}) - R_s \text{-----} \quad (3)$$

where  $R_s$  = solution resistance

$$C_{dl} = 1/2\pi R_t f_{max} \text{-----} \quad (4)$$

Where,

$f_{max}$  = frequency at maximum imaginary impedance.

AC impedance spectra were recorded with initial  $E(v) = 0$ , high frequency (Hz) =  $1 \times 10^5$ , low frequency (Hz) = 1, amplitude (V) = 0.005 and quiet time (s) = 2.

### **2.5 Surface examination study**

The carbon steel specimens were immersed in various test solutions for a period of 1 day. After immersion, 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.

#### **2.5.1 Fourier Transform Infrared spectra (FTIR)**

These spectra were recorded in a Perkin–Elmer–1600 spectrophotometer using KBr pellet. The FT-IR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr and making the pellet.

#### **1.5.2. Synergism Parameters ( $S_1$ )**

The synergism parameter is calculated using the following equation:

$$S_1 = \frac{1 - I_{1+2}}{1 - I_1 - I_2}$$

**Where;**

$S_1$ = synergism parameter

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

$I_1$ = Inhibition efficiency of TSC

$I_2$ = Inhibition efficiency of  $Al^{3+}$

$I_{1+2}$  = combined inhibition efficiency of substance TSC and substance  $Al^{3+}$

### **3.0. Results and Discussion**

### **3.1 Analysis of Weight-loss study**

The different boundaries of ground water are given in Table 1. The hindrance efficiencies (IE) of TSC in controlling consumption of carbon steel in ground water, for a time of three days in the nonattendance and nearness of  $Al^{3+}$  by weight reduction strategy are given in Tables 2 to 6. TSC alone has nearly 53 % IE, while  $Al^{3+}$  has 15% IE. Without TSC, the pace of transport of  $Al^{3+}$  from the bulk part of the solution towards the metal surface is moderate [13]

At the point when TSC is joined with  $Al^{3+}$  particles it is discovered that their blend shows 97% IE. This proposes a synergistic impact among TSC and  $Al^{3+}$  particles; TSC can move  $Al^{3+}$  towards the metal surface.

### **3.2. Synergism Parameters ( $S_I$ )**

Synergism parameters are indications of synergistic effect existing between inhibitors [14, 15]. From Table 7 it is clear that % IE for TSC- $Al^{3+}$  system inhibitor is higher than the % IE for single TSC and for single  $Al^{3+}$  inhibitor, which is synergistic in nature.  $S_I$  approaches unity when there are no interactions between the inhibitor compounds, while  $S_I > 1$  points to a synergistic effect; in the case of  $S_I < 1$ , the antagonistic interaction prevails.

### **3.3. ANALYSIS OF THE RESULTS OF POTENTIOSTATIC POLARIZATION STUDY OF TSC- $Al^{3+}$ SYSTEM.**

Fig.1. represents the Potentiodynamic polarization curves of carbon steel in ground water in the absence and presence of the inhibitor system. The cathodic branch represents the oxygen reduction reaction, while the anodic branch represents the iron dissolution reaction. The electrochemical parameters such as corrosion potential ( $E_{corr}$ ), corrosion current ( $I_{corr}$ ) and Tafel slopes (ba and bc,) are given in Table 8.

When carbon steel is immersed in ground water, the corrosion potential is  $-455$  mV vs SCE. The formulation consisting of 150ppm TSC+50ppm  $Al^{3+}$  shifts the corrosion potential to  $-483$  mV vs SCE. It is observed that the corrosion potential is shifted to the cathodic side. It is also observed that the shift in the cathodic slope (from 196 mV/dec to 155 mV/dec) is higher than the shift in the anodic slope (from 143mV/dec to 82 mV/dec). Hence, it can be said that the inhibitor system predominantly controls the cathodic reaction [16].

For the formulation of TSC- $Al^{3+}$  the corrosion current value has decreased  $5.29 \times 10^{-3}$  to  $0.122 \times 10^{-3}$  A/cm<sup>2</sup>. The decrease in corrosion current indicates adsorption of the inhibitor on

the metal surface to block the active sites and inhibit corrosion and reduce the corrosion rate [17, 18].

### **3.3 ANALYSIS OF AC IMPEDANCE SPECTRA**

AC impedance spectra have been used to detect the formation of film on the metal surface. If a protective film is formed, the charge transfer resistance ( $R_{ct}$ ) increases and double layer capacitance ( $C_{dl}$ ) value decreases [19, 20]. Nyquist representations of carbon steel in ground water in the absence and presence of the inhibitor system are shown in Fig. 2.

It is clear from the plots that the impedance response of carbon steel was significantly changed after addition of the inhibitors. The impedance diagrams obtained have an almost semicircular appearance. This indicates that the corrosion of carbon steel in aqueous solution is mainly controlled by a charge transfer process [21]. The deviation from the perfect semicircle shape (depression) is due to the frequency dispersion of interfacial impedance. This anomalous behavior is generally attributed to the inhomogeneity of the metal surface arising from surface roughness or interfacial phenomena [22]. The AC impedance parameters, namely solution resistance ( $R_s$ ), charge transfer resistance ( $R_{ct}$ ) and double layer capacitance ( $C_{dl}$ ) are given in Table 9.

When carbon steel is immersed in groundwater, ( $R_{ct}$ ) value is  $5.06 \Omega \text{ cm}^2$  and ( $C_{dl}$ ) value is  $1.6 \times 10^{-2} \text{ F/cm}^2$ . When TSC– $\text{Al}^{3+}$  are added to ground water, ( $R_{ct}$ ) value increases from  $5.06 \Omega \text{ cm}^2$  to  $189 \Omega \text{ cm}^2$  and the ( $C_{dl}$ ) decreases from  $1.6 \times 10^{-2} \text{ F/cm}^2$  to  $2.1 \times 10^{-5} \text{ F/cm}^2$ . This suggests that a protective film is formed on the surface of the metal. Results obtained from impedance measurements can be interpreted in terms of the equivalent circuit of the electrical double-layer presented in Fig. 3, which was used previously to model other interaction involving metal/solutions interface [23, 24]. This type of circuit is known as Randle circuit. Many authors also suggested Randle circuit for similar graphs [25,26]

### **3.4 ANALYSIS OF FTIR SPECTRA**

Earlier researchers have confirmed that FT-IR spectrometer is a powerful instrument that can be used to determine the type of bonding of inhibitors adsorbed on the metal surface [27]. FT-IR spectra have been used to analyze the protective film formed on metal surface. FT-IR spectrum of pure 150ppm TSC is given in Fig.4. The FT-IR spectrum of the film formed on the metal surface after immersion in the ground water for one day containing 150ppm of TSC and 50 ppm of  $\text{Al}^{3+}$  is also shown in Fig.4. FT-IR spectrum is shown in Fig.4 the C=O stretching frequency appears at  $1627 \text{ cm}^{-1}$ . The O-H stretching frequency appears at  $3390 \text{ cm}^{-1}$ . The O-C stretching frequency appears at  $2881 \text{ cm}^{-1}$ . The C=O stretching

frequency has shifted from 1627 to 1606  $\text{cm}^{-1}$ . The O-C stretching frequency has shifted from 2925 to 2924  $\text{cm}^{-1}$ . The O-H stretching frequency has shifted from 3390 to 3790  $\text{cm}^{-1}$ . The peak at 860  $\text{cm}^{-1}$  is due to  $\text{Al}_2\text{O}_3$  and the peak at 1388  $\text{cm}^{-1}$  corresponds to  $\text{Al}(\text{OH})_3$ . So, it is concluded that  $\text{Al}(\text{OH})_3$  is formed on cathodic sites of the metal surface [28].

### 3.5. ANALYSIS OF OPTICAL MICROSCOPES

The photo micro graphs of different magnification (120, 600 times) of carbon steel specimen immersed in the groundwater for 3 days in the absence and presence of inhibitor system are shown in Fig 5, shows the rough film that indicates the corrosion products spread on the mild steel surface immersed in groundwater and the smooth film that indicates the effect of inhibitor system on the mild steel surface [29].

### 3.6. MECHANISM OF CORROSION INHIBITION

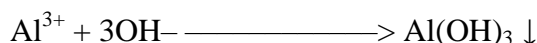
With these discussions, a mechanism may be proposed for the corrosion inhibition of carbon steel immersed in dam water containing 150ppm of TSC+50ppm of  $\text{Al}^{3+}$ .

When the formulation consists of 150ppm of TSC+50ppm of  $\text{Al}^{3+}$  in ground water, there is formation of TSC-  $\text{Al}^{3+}$  complex in solution

1. When carbon steel is immersed in this solution TSC-  $\text{Al}^{3+}$  complex diffuses from the bulk of the solution towards the metal surface.
2. TSC-  $\text{Al}^{3+}$  complex is converted into TSC- $\text{Fe}^{2+}$  complex on the anodic sites of the metal surface with the release of  $\text{Al}^{3+}$  ion.



The released  $\text{Al}^{3+}$  combines with  $\text{OH}^-$  to form  $\text{Zn}(\text{OH})_2$  on the cathodic sites of the metal surface



3. Thus the protective film consists of TSC- $\text{Fe}^{2+}$  complex and  $\text{Al}(\text{OH})_3$
4. This account for the synergistic effect of TSC-  $\text{Al}^{3+}$  system.

### 4. CONCLUSIONS

The present study leads to the following conclusions.

The inhibition efficiency (IE) of TSC controlling corrosion of carbon steel immersed in ground water in the absence and presence of  $\text{Al}^{3+}$  has been evaluated by weight loss method. The formulation consisting of 150ppm of TSC+50ppm of  $\text{Al}^{3+}$  has 97% IE.



Polarization study reveals that TSC– Al<sup>3+</sup> system controls the cathodic reaction predominantly. AC impedance spectra and SEM study reveal that a compact protective film is formed on the metal surface.

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**Table 1 The parameters of ground water which is used for investigation**

<b>Parameter</b>	<b>Value</b>
pH	8.0
TDS	766 mg/l
Alkalinty	380mg/l
Chloride	12 mg/l
Sulphate	18 mg/l
Calcium	70 mg/l
Magnesium	81 mg/l
Barium	11mg/l

**Table.2. Corrosion rate (CR) of carbon steel in ground water in the presence of inhibitor system and the inhibition efficiency (IE) obtained by weight loss method**

**Inhibitor system: TSC + Al<sup>3+</sup> (0 ppm)**

**Immersion period –3 days**

<b>TSC ppm</b>	<b>Al<sup>3+</sup> ppm</b>	<b>CR mdd</b>	<b>IE %</b>
0	0	19.52	-
50	0	2.70	85.0
100	0	10.40	46.3
150	0	9.17	53.0
200	0	8.70	55.0

**Table.3. Corrosion rate (CR) of carbon steel in ground water in the presence of inhibitor system and the inhibition efficiency (IE) obtained by weight loss method**

**Inhibitor system: TSC + Al<sup>3+</sup> (50 ppm)**

**Immersion period – 3 days**

<b>TSC</b>	<b>Al<sup>3+</sup> ppm</b>	<b>CR mdd</b>	<b>IE %</b>
0	50	16.5	15.2
50	50	1.85	90.5
100	50	11.55	40.8
<b>150</b>	<b>50</b>	<b>0.58</b>	<b>97.0</b>
200	50	7.51	61.5

**Table.4 Corrosion rate (CR) of carbon steel in ground water in the presence of inhibitor system and the inhibition efficiency (IE) obtained by weight loss method**

**Inhibitor system: TSC + Al<sup>3+</sup> (100 ppm)**

**Immersion period – 3 days**

<b>TSC ppm</b>	<b>Al<sup>3+</sup> ppm</b>	<b>CR Mdd</b>	<b>IE %</b>
0	100	8.1	58.5
50	100	6.79	65.2
100	100	2.98	84.7
<b>150</b>	<b>100</b>	<b>0.70</b>	<b>96</b>
200	100	12.5	37.2

**Table.5. Corrosion rate (CR) of carbon steel in ground water in the presence of inhibitor system and the inhibition efficiency (IE) obtained by weight loss method**

**Inhibitor system: TSC + Al<sup>3+</sup> (150 ppm)**

**Immersion period – 3 days**

<b>TSC ppm</b>	<b>Al<sup>3+</sup> ppm</b>	<b>CR mdd</b>	<b>IE %</b>
0	150	7.52	61.5
50	150	11.42	41.5
100	150	11.9	39.0
150	150	11.1	43.3
200	150	9.37	52.0

**Table.6. Corrosion rate (CR) of carbon steel in ground water in the presence of inhibitor system and the inhibition efficiency (IE) obtained by weight loss method**

**Inhibitor system: TSC + Al<sup>3+</sup> (200 ppm)**

**Immersion period –3 days**

<b>TSC ppm</b>	<b>Al<sup>3+</sup> ppm</b>	<b>CR mdd</b>	<b>IE %</b>
0	200	4.06	79.2
50	200	8.58	56.0
100	200	0.97	95.0
150	200	2.93	85.0
200	200	4.64	76.2

Table.8. Synergistic Parameters ( $S_1$ ) of TSC- $Al^{3+}$  system.

Concn. of TSC ppm	I.E. % $I_1$	Concn. of $Al^{3+}$ ppm	I.E. % $I_1$	Combined I'E. $I_{1+2}$	Synergism $S_1$
50	85.9	100	15.2	90.5	13.45
100	46.3	100	15.2	40.8	16.11
150	53	100	15.2	97.0	7.67
200	55.0	100	15.2	61.5	12.64

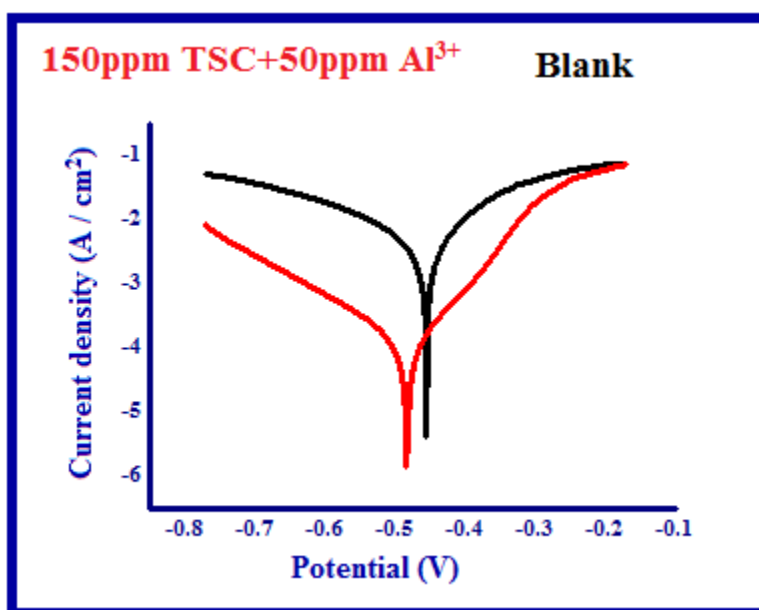


Fig.1. Polarization curves of carbon steel immersed in ground water in presence and absence of inhibitors

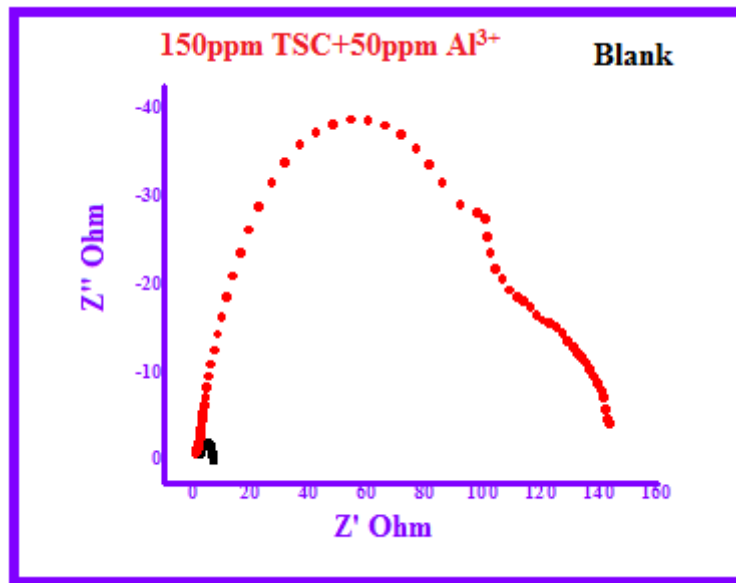


Fig.2. AC impedance study of carbon steel immersed in ground water in presence and absence of inhibitors

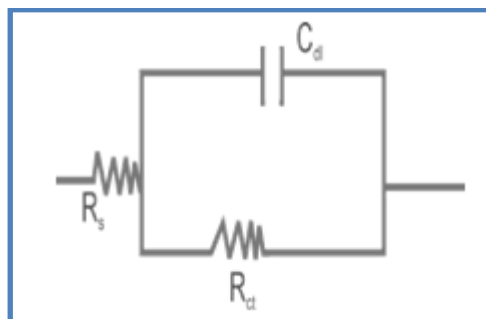


Fig. 3. Equivalent electrical circuit diagram



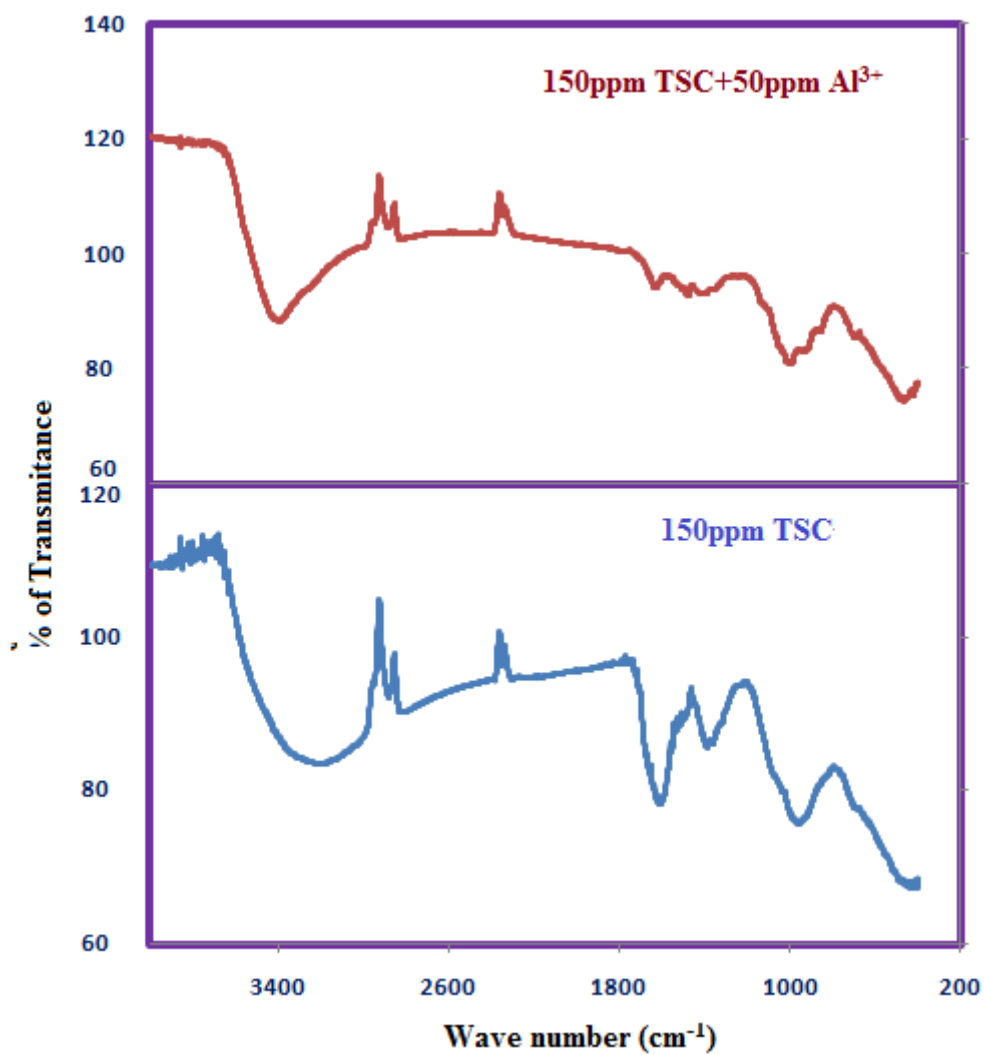
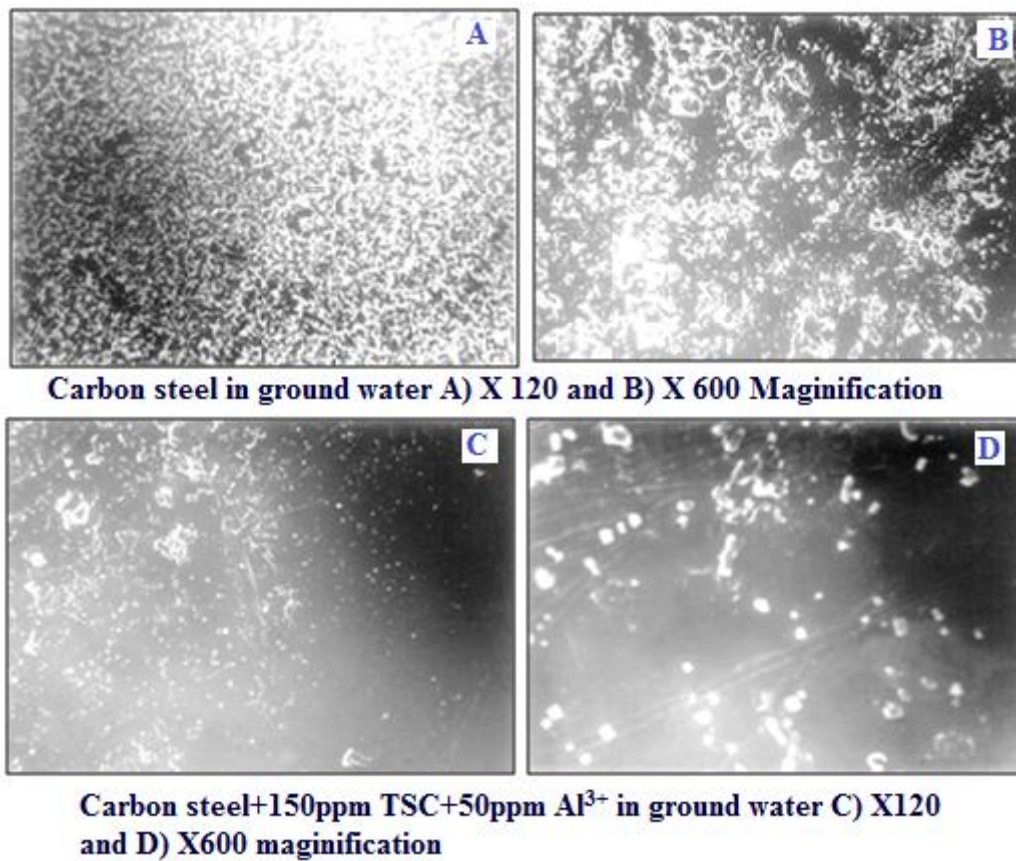


Fig.3. FT-IR spectra of carbon steel in ground water in presence and absence of inhibitors



**Fig.4. SEM Images of carbon steel in ground water in presence and absence of inhibitors**