Inhibition Efficiency of Calcium Gluconate – Al^{3+} Mixed Inhibitors on Mild Steel in Ground Water

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ABSTRACT

The inhibition efficiency (IE) of Calcium Gluconate (CG) in controlling corrosion of carbon steel immersed in ground water in the absence and presence of Al^{3+} has been evaluated by weight loss method. It is observed that the synergistic formulation consisting of 150ppm CG and 100 ppm of Al^{3+} has 96% IE. Effect of immersion period and pH of the corrosion medium has been studied. Polarization study reveals that CG- Al^{3+} system functions as a cathodic inhibitor. AC impedance study reveals that a protective film is formed on the metal surface.

The protective film has been analysed by FT-IR and optical microscope studies. It is found that the protective film consists of Fe^{2+}-CGcomplex. On the basis of all the above studies, a suitable mechanism of corrosion inhibition is proposed.

Key words: Carbon steel, corrosion inhibition, synergistic effect, cathodic inhibitor, Calcium Gluconate

1. INTRODUCTION

The usage of ground water is common in all industries. The role of dissolved oxygen in ground water is the predominant factor to cause mild steel corrosion [1,2]. Depending upon the metal /environment combinations different types of inhibitors are used in suitable concentrations. The inhibition efficiency [3-15] organic inhibitors such as carboxylates, gluconates, gluconic acids, amines [16] are explained by the adsorption of organic molecules on the metal surface incorporation into the oxide film. They can be adsorbed on the metal surface through hetero atoms such as nitrogen, oxygen, sulphur and phosphorous, multiple bonds with aromatic rings and block the active sites, thus decreasing the corrosion Rates. Several inhibitors such as phosphonic acid [17-18],

Thiourea [19], carboxy methyl cellulose [20], Sodium dodecyl sulphate [21] have been used to control corrosion of carbon steel.

Inhibitors for carbon steel in near neutral, aqueous solutions are soluble chromates, dichromates nitrates, borates, benzoates and salts of carboxylic acids. Corrosion inhibition due to the formation of oxide layer on Cu metal surface in concentrated propionic acid and dilute citric acid [22] have been reported. The corrosion inhibition of carbon steel in ground water by adipic acid and Zn^{2+} system has been reported [23]. Existence of synergism between succinic acid and Zn^{2+} in controlling corrosion of carbon steel in well water has been investigated [24]. Inhibitors such as benzoate, phthalate and other carboxylates [25-27] stabilize the oxide film on iron surface presumably. Their inhibitive action results from the bonding of the O^- ion. Carboxylates are anodic inhibitors.

The corrosion inhibition of steel by salicylic acid in acidic media has been investigated [28]. In present study synergistic effect of Calcium Gluconate CG and Al^{3+} in corrosion inhibition of carbon steel in ground water has been investigated in detail. Calcium Gluconate is an environment friendly organic compound of molecular formula is $[CH_2OH(CHOH)_4COO]_2Ca$. While the inhibition efficiencies have been evaluated by the weight loss method, the mechanistic aspects are based upon the results of potentiostatic polarization, AC impedance studies and also by the surface examination such as FT-IR and optical microscopy.

2. EXPERIMENTAL

2.1 Preparation of the specimens

Carbon steel specimens (0.025% S, 0.06% P, 0.4%Mn, 0.15%C and the rest iron) of the dimension 1.0 x 4.0 x 0.2cm were polished to a mirror finish and degreased with trichloro ethylene and used for the weight loss method and surface examination studies Ground water was collected from Vadipatty village near Kodairoad, Tamilnadu, India. Corrosion behavior of carbon steel in this water was evaluated.

2.2 Weight loss method

The parameters of ground water used in the present study are given in Table-1. Carbon steel specimens in triplicate were immersed in 100ml of ground water containing various concentrations of the inhibitor in the absence and presence of Al^{3+} for three days. The weights of the specimens before and after immersion were

determined using a balance, Shimadzu AY62 model. The corrosion products were cleaned with Clarke's solution [29]. The inhibition efficiency (IE) was then calculated using the equation

$$\textbf{IE = 100 [1-(w_2/w_1)] \%} \qquad\qquad \textbf{------ (1)}$$

Where,

w_1 = Corrosion rate (mdd) in absence of inhibitor

w_2 = Corrosion rate (mdd) in presence of inhibitor

2.3 Surface examination study

The carbon steel specimens were immersed in various test solutions for a period of three days. After three days, the specimens were taken out and dried. The nature of the film formed on the surface of metal specimens was analyzed by various surface analysis techniques, namely FT-IR spectra and, Optical Microscopy Studies.

2.3.1 FT-IR spectra

These spectra were recorded in a Perkin – Elmer 1600 spectrophotometer. The film was carefully removed, mixed thoroughly with KBr and made into pellets and the FT-IR spectra were recorded.

2.3.2 Potentiostatic polarization study

This study was carried out using CHI 66A Electrochemical Workstation model. A three-electrode cell assembly was used. The working electrode was used as a rectangular specimen of carbon steel with one face of the electrode of constant 1 cm^2 area exposed. A saturated calomel electrode (SCE) was used as reference electrode. A rectangular platinum foil was used as the counter electrode. The results such as Tafel slopes, I_{corr} (corrosion current) and E_{corr} (corrosion potential) were calculated.

Tangents were drawn on the cathodic and anodic polarization curves. From the point of intersection of the two tangents I_{corr} and E_{corr} were calculated.

2.3.3 AC impedance measurements

The CHI 66A Electrochemical Workstation model was used for AC impedance measurements. The cell set up was the same as that used for polarization measurements. The real part (Z') and imaginary part (Z") of the cell impedance were measured in ohms for various frequencies. The R_{ct} (charge transfer resistance) and C_{dl} (double layer capacitance) values were calculated.

2.3.4 Surface analysis by optical microscopy

Samples for SEM analysis were mounted directly on scotsch double adhesive tape and analyzed in a Hitachi scanning electron microscope model s-450 operated at 15 Kv and photographed.

2.5 Influence of duration of immersion period and pH on the IE of CG- Al^{3+} System

Influence of duration of immersion period (1 to 7 days) and pH on the IE of CG- Al^{3+} System has been analysed various from 6 to 12.

2.6. Synergism Parameters (S₁)[30]

The synergism parameter (S_1) is calculated using the relation as stated below

$$S_I = \frac{1 - I_{1+2}}{1 - I'_{1+2}} \qquad\qquad \text{---------------- (2)}$$

Where,

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

I'_{1+2} = combined inhibition efficiency of substance 1 and substance 2.

3. Results and Discussion

3.1 Parameters of ground water

Corrosion behaviour of carbon steel in ground water was evaluated and the various parameters are given in Table 1.

3.2. Analysis of the results of weight loss method

The various parameters of ground water are given in Table 1.

The inhibition efficiencies (IE) of CG in controlling corrosion of carbon steel in ground water, for a period of three days in the absence and presence of Al^{3+} by weight loss method are given in Tables 2 to 6. 150ppm of CG alone has some 61% IE, whereas 100 ppm of Al^{3+} alone has 3%. In the absence of CG the rate of transport of Al^{3+} from the bulk of the solution towards the metal surface is slower . Similar observations have already been reported [31-36]. When CG is combined with Al^{3+} ions it is found that the IE also increases. Interestingly their combination of 150 ppm CG and 100ppm of Al^{3+} shows 96% IE. This suggests a synergistic effect between CG and Al^{3+} ions; CG is able to transport Al^{3+} towards the metal surface.

3.3. Analysis of the results of potentiostatic polarization study of CG- Al^{3+} system.

The corrosion parameters of carbon steel immersed in various test solutions obtained by polarization study are given in Table 7. The polarization curves are shown in Fig. 1.

When carbon steel is immersed in ground water, the corrosion potential – 455mV Vs saturated calomel electrode (SCE). The formulation consisting of 150ppm of CG and 100 ppm of Al^{3+} shifts the corrosion potential to -488 mV Vs SCE. This suggests that the cathodic reaction is controlled predominantly, since more CG is transported to the cathodic sites in the presence of Al^{3+}, which is further confirmed by more shifts in the cathodic Tafel Slope as shown on Table 7. This result suggests that the CG– Al^{3+} formulation functions as cathodic inhibitor. The corrosion current for ground water is 5.29 x 10^{-3} A/cm^2. It is decreased to 0.095 x 10^{-3} A/cm^2 by 150ppm of CG and 100 ppm of Al^{3+}. This indicates that a protective film is formed on the metal surface.

3.4. Analysis of the results of AC impedance spectra

AC impedance spectra of carbon steel in various solutions were regarded (Fig. 2). The AC impedance parameters, namely, charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) are given in Table 8. When carbon steel is immersed in ground water R_{ct} value is 5.06 Ω cm^2 and C_{dl} value is 1.6 x 10^{-2} F cm^{-2}. The formulation consisting of 100ppm of Al^{3+} and 150 ppm of SG shifts the corrosion potential to -488mV Vs SCE which are added to ground water and therefore R_{ct} value increases to 228 Ω cm^2. The C_{dl} value has change from 1.6 x 10^{-2} Fcm^{-2} to 1.1 x 10^{-5} Fcm^{-2}. This suggests that a protective film is formed on the surface of the metal. This accounts for the high IE of CG– Al^{3+}system.

3.5. Analysis of FT-IR spectra

The FT-IR spectrum of pure CG the C=O stretching frequency occurs at 1560cm^{-1}. The FT-IR spectrum of the film scratched from the surface of the metal immersed in ground water containing 150 ppm of CG and 100ppm of Al^{3+} is given in Fig.3. It is seen from the spectrum that C=O stretching frequency of CG in the free state has shifted from 1560 cm^{-1} to 1540cm^{-1}. This shift indicates that the carbonyl oxygen atom is coordinated to from Fe^{2+}-CG complex on the anodic sites of the metal surface [37-39]. The band at 1426 cm^{-1} is due to Al (OH)$_3$ [40-41].

3.6. Analysis of optical microscopes

Giacomelli [42] and Jagdheesh [43] studied the inhibitor effect of succinic acid on the corrosion resistance of mild steel by optical microscopic studies and established the protective layer formation from photographic images. The photo micro graphs of different magnification (120, 600 times) of mild steel specimen immersed in the groundwater for 3 days in the absence and presence of inhibitor system are shown in Fig 4, 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.

3.7. Influence of duration of immersion period and pH on the IE of the CG – Al^{3+} System

The influence of duration of immersion on the IE of the CG- Al^{3+} system is given in Table 9. It is found that as the duration of immersion period increases with the decrease of inhibition efficiency. When the immersion period is1 day IE= 85%, when the immersion period is 3 days IE = 96%. When immersion period is 5 days IE=86%, when the immersion period is 7 days IE = 55%.

This may be due to the fact that as the period of immersion increases the protective film formed on the metal surface namely Fe^{2+} - CG Complex is broken by the aggressive chloride ion present in ground water and hence IE decreases[44].

Further there is a competition between the formation of $FeCl_2$ (and also $FeCl_3$ and formation (Fe^{2+} - CG) complex. It seems that as the immersion period increases the formation of $FeCl_2$ is more favored than the formation of Fe^{2+}-CG complex at the anodic states of the metal. Hence a decrease in inhibition efficiency is noticed as the period of immersion increases.

The influence of pH on the IE of CG- Al^{3+} system is given in Table 10. It is found from that when P^H is decreased from 8 to 6 (by addition of dil. H_2SO_4) the IE decreases from 95% to 76%. This is due the attack of H^+ ion of the H_2SO_4 on the metal surface and hence increases in corrosion rate and decrease in IE.

When P^H is increased from 8 to 10 (by addition of dil NaOH) the IE decrease from 95% to 76%. This is due to the fact when NaOH is added Al^{3+} is precipitated as $Al(OH)_3$ in the bulk of the solution so the transport of inhibitor towards the metal surface decreases[45] and hence the IE decrease when P^H is increased from 10 to 12 (by addition of dil. NaOH) the IE increases from 76% to 81% . This is due to the fact that further addition of NaOH converts $Al(OH)_3$ precipitate into soluble sodium aluminate ($NaAlO_2$). Now the transport of inhibitors towards the metal surface is favoured. Hence the IE increases.

3.8. Synergism Parameters (S_1)

The synergism parameters are given in Table.11. The combined effect is called synergistic effect. The synergism parameters (S_1) calculated from surface coverage was found to be more than unity. This result clearly shows the synergistic effect between CG and Al^{3+}

3.9. Mechanism of corrosion inhibition

The results of the weight loss study show that the formulation consisting of 150ppm of CG and 100ppm of Al^{3+} has 96% IE in controlling corrosion of carbon steel, in ground water. A synergistic effect exists between Al^{3+} and CG. Polarization study reveals that the formulations functions as mixed inhibitor. AC impedance spectra reveal that the protective film is formed on the metal surface. FT-IR spectra study reveals that the protective film consists of Fe^2 – CG complex and $Al(OH)_3$. In order to explain these facts the following mechanism of corrosion inhibition is proposed.

♦ When the solution containing ground water, 100ppm of Al^{3+} and 150ppm of CG is prepared, there is formation of Al^{3+}-CG complex in solution.

♦ When carbon steel is immersed in the solution, the Al^{3+}-CG complex diffuses from the bulk of the solution towards metal surface.

♦ On the metal surface, Al^{3+} – CG complex is converted in to Fe^{2+} – CG complex on the anodic sites. Al^{3+} is released.

♦ Al^{3+}-CG+ Fe^{2+} -----------> Fe^{2+}–CG + Al^{3+}

♦ The released Al^{3+} combines with OH^- form $Al(OH)_3$ on the cathodic sites.

♦ Al^{3+} + $3OH^-$ ------------> $Al(OH)_3$ ↓

♦ Thus the protective film consists of Fe^{2+}–CG complex and $Al(OH)_3$.

4.Conclusions

The present study leads to the following conclusions

- A synergistic effect exists between Calcium Gluconate CG and Al^{3+} in controlling corrosion of carbon steel immersed in ground water.

- The formulation consisting of 150ppm of CG and 100 ppm of Al^{3+} has 96% IE.

- Polarization study reveals that CG– Al^{3+} system functions as a cathodic inhibitor.

- AC impedance spectra reveal that a protective film is formed on the metal surface.

- FT-IR spectra reveal that the protective film consists of Fe^{2+}-CG complex and $Al(OH)_3$

- This formulation may find application in cooling water system.

- Optical micro graphic images the formation of protective layer on the metal surface with its surface morphology.

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ANNEXURES:

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

Parameter	Value
pH	8.0
TDS	790 mg/l
Alkalinty	365mg/l
Chloride	13 mg/l
Sulphate	16 mg/l
Calcium	74 mg/l
Magnesium	86 mg/l
Barium	10mg/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: Calcium gluconate + Al^{3+} (0 ppm) Immersion period – 3 days

Calcium Gluconate ppm	Zn^{2+} Ppm	CR mdd	IE %
0	0	13.64	--
50	0	7.5	45
100	0	5.73	58
150	0	4.77	65
200	0	4.09	70
250	0	3.82	72

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: Calcium gluconate + Al^{3+} (5 ppm) Immersion period – 3 days

Calcium Gluconate ppm	Al^{3+}ppm	CR mdd	IE %
0	5	13.64	--
0	5	15.00	-10
50	5	10.64	22
100	5	11.80	18
150	5	11.59	15
200	5	12.00	12
250	5	13.23	3

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: Calcium gluconate + Al^{3+} (10 ppm) Immersion period – 3 days

Calcium Gluconate ppm	Al^{3+}ppm	CR mdd	IE %

0	0	13.64	--
0	10	12.96	5
50	10	4.09	70
100	10	7.36	46
150	10	8.86	35
200	10	12.96	5
250	10	13.37	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: Calcium gluconate + Al^{3++} (25 ppm) Immersion period – 3 days

Calcium Gluconate ppm	Al^{3+}ppm	CR mdd	IE %
0	25	13.64	--
0	25	12.28	+10
50	25	5.18	62
100	25	5.45	60
150	25	10.91	20
200	25	11.05	19
250	25	12.96	5

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: Calcium gluconate + Al^{3++} (50 ppm) Immersion period – 3 days

Calcium Gluconate Ppm	Al^{3+}ppm	CR mdd	IE %
0	0	13.64	--
0	50	12.00	12
50	50	4.77	65
100	50	2.05	85
150	50	8.18	40

200	50	9.27	32
250	50	11.59	15

Table 7: Corrosion Parameters obtained by Potentiostatic Polarization method.

System	E_{Corr}	I_{Corr}	b_c	b_a	R_p	% I.E
Blank	-0.455	5.29×10^{-3}	196.85	143.55	7	------
CG 150 ppm + Al^{3+} 100ppm	-0.480	0.94×10^{-3}	164.23	75.25	237	98.21

Table 8: Corrosion Parameters obtained by AC Impedance Studies.

System	R_s (ohm cm^{-2})	R_{ct} (ohm cm^{-2})	C_{dl} (F/cm^2)	% I.E
Blank	2.568	5.063	1.6×10^{-2}	-----
CG 150 ppm + Al^{3+} 100ppm	-7.309	228.444	1.1×10^{-5}	97.78

Table 9. Influence of duration of immersion on the IE of the CG(150ppm) – Al^{3+} (100ppm) system.

Immersion Period Day	1	3	5	7
Blank Corrosion rate Mdd	15.63	19.52	22.63	18.5
CG150ppm + Al^{3+} 100ppm Corrosion rate mdd	1.52	0.74	1.98	2.56
IE %	85.36	96.50	86.36	55.36

Table.10. Influence of P^H on the IE of the CG(150ppm) – Al^{3+} (100ppm) system.

pH	6	8	10	12
Blank Corrosion rate mdd	12.56	11.56	9.58	9.36
CG150ppm + Al^{3+} 100ppm Corrosion rate mdd	1.52	0.56	1.55	1.33
IE %	76.3	95.6	78.6	81.2

Table.11. Synergistic Parameters (S$_1$) of CG-- Al^{3+}system.

Concn. of CG ppm	I.E. % I_1	Concn. of Al^{3+} ppm	I.E. % I_1	Combined I'E. I_{1+2}	Synergism S_1
50	6.09	100	58.5	90.8	3.23
100	7.90	100	58.5	85.9	4.65
150	61.58	100	58.5	96.2	36.50
200	16.46	100	58.5	60.9	14.18

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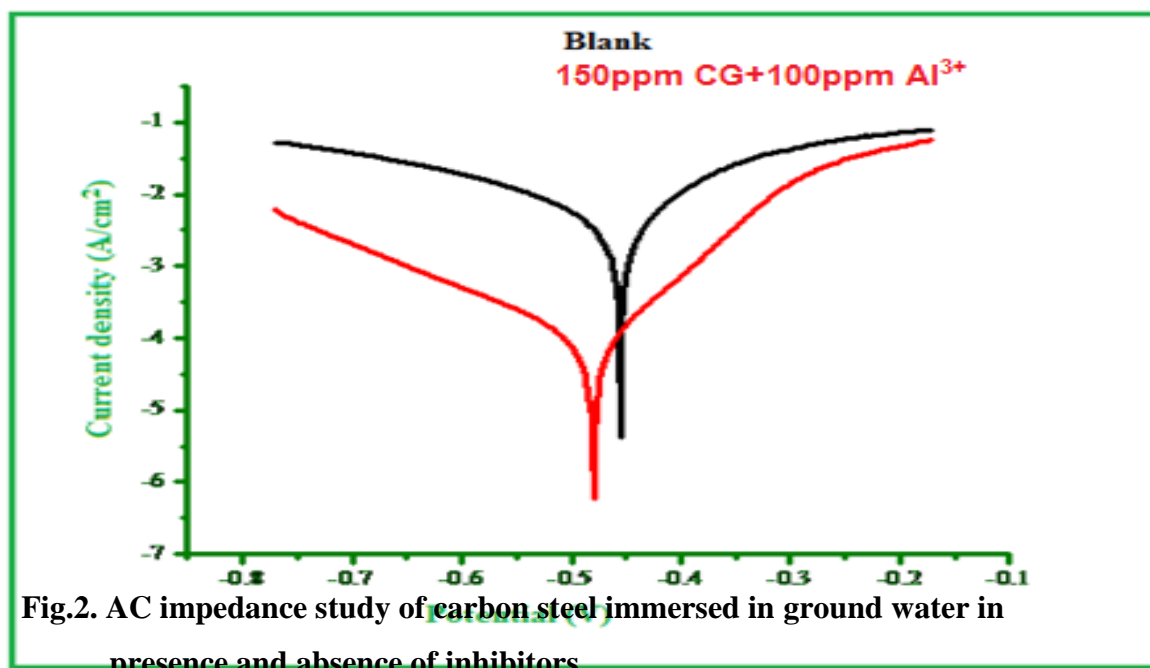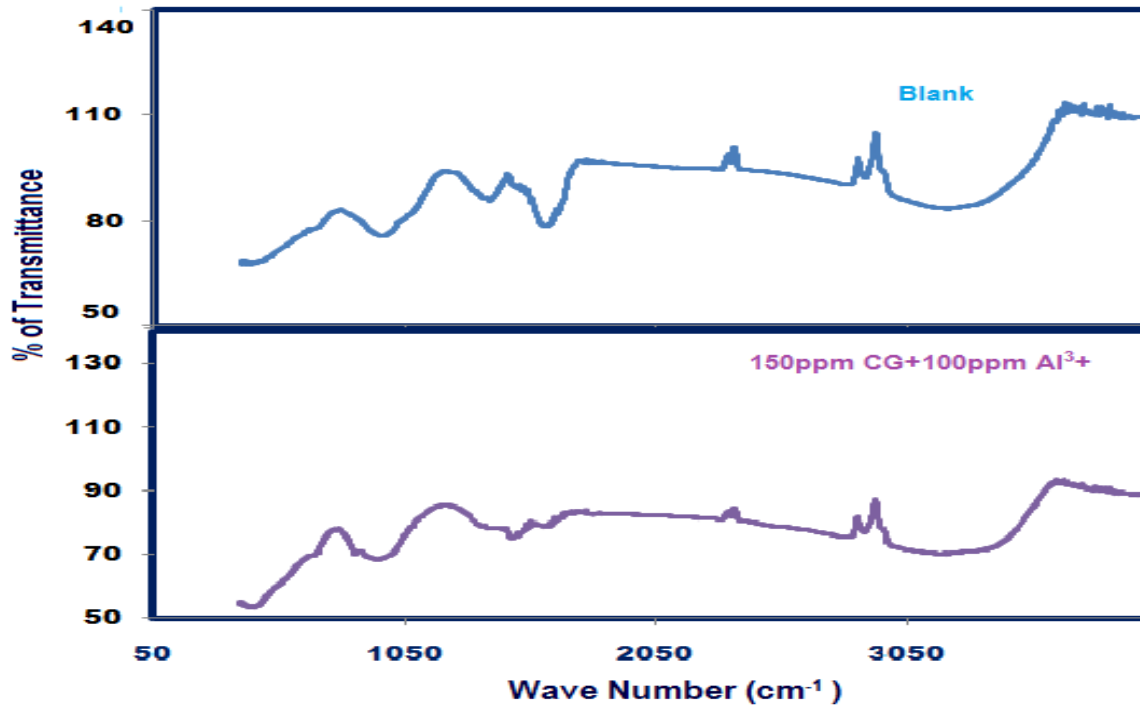

Fig.2. AC impedance study of carbon steel immersed in ground water in presence and absence of inhibitors
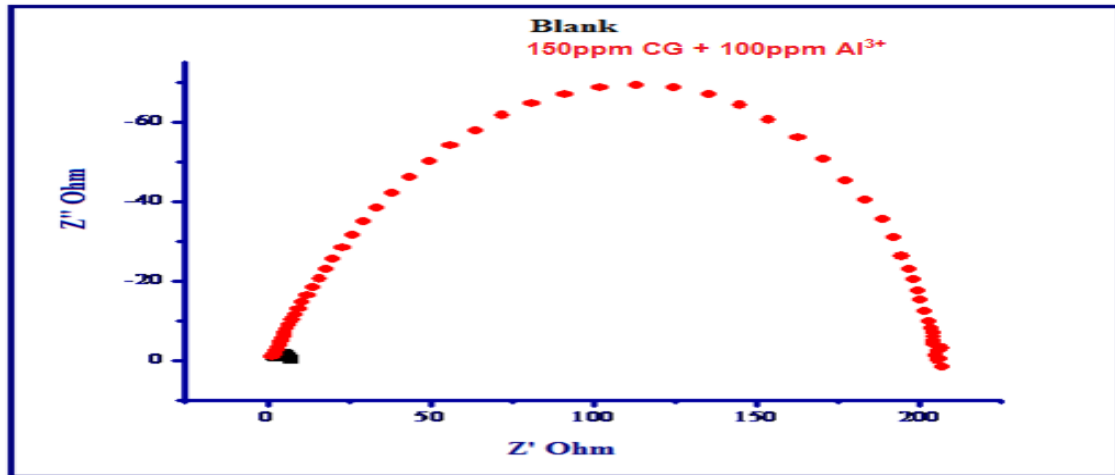
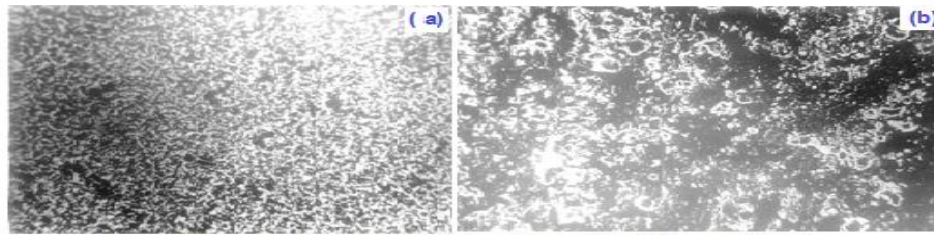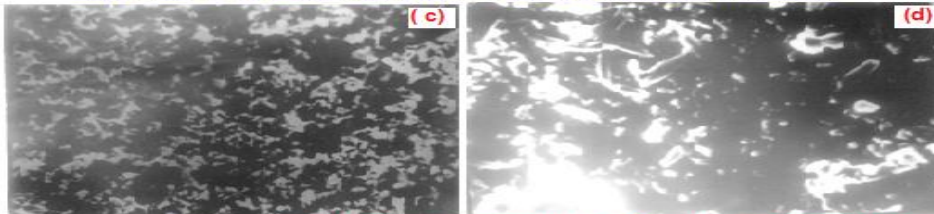**Fig.3. FT-IR spectra of carbon steel in ground water in presence and absence of inhibitors**

Fig.4. SEM images of carbon steel in ground water in presence and absence of inhibitors
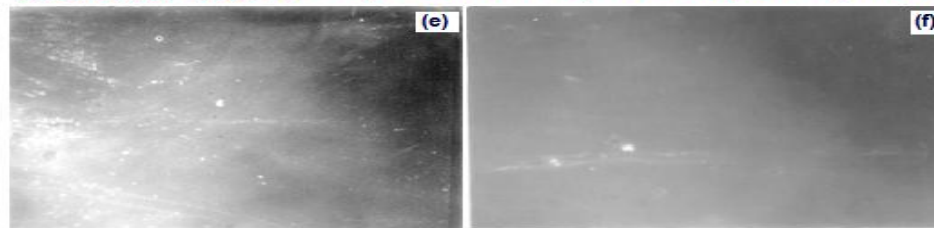
carbon steel in ground water with a) x120 and b) x600 magnification

carbon steel + 100ppm Al³⁺ in ground water with c) x120 and d) x600 magnification

carbon steel+150ppm CG+100ppm Al³⁺ with e)x120 and f)x600 magnification