Corrosion inhibition of mild steel in sodium chloride solution by Glutaric acid

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Abstract

The corrosion inhibition of mid steel in an aqueous solution containing 60 ppm of Cl^- has been evaluated by weight loss method in the absence and presence of inhibitor namely glutaric acid. The formulation consisting of 250 ppm of glutaric acid offers good inhibition efficiency of 78 %. The electrochemical studies such as polarization study and impedance spectra have been recorded. Polarization study revelas that this formulation acts as an anodic inhibitor and controls the anodic reaction more preferably. AC impedance spectra signify that a protective film is formed over the mild steel surface. The corrosion parameters obtained from weight loss method, polarization studies and impedance spectra shows this inhibitor offers good corrosion inhibition efficiency and control the control the corrosion of mild steel. Fluorescence spectral analysis was used to detect the presence of iron-inhibitor complex in solution and protective film formed over the mild steel surface. The surface morphology has been analyzed by FTIR, UV-Visible spectroscopy Scanning Electron Microscope (SEM).

Key Words

Corrosion inhibition, FTIR, Glutaric acid, EIS, Mild Steel and SEM.

Introduction

Corrosion mainly occurs due to chemical or electrochemical reaction between a material and its environment. Iron tend to corrode, it produces deterioration or destroys of the material and changes its properties. The end result of corrosion involves a metal atom being oxidized, whereby it loses one or more electrons. Corrosion research in early stage, they deal with only in electrochemical studies. The corrosion manifests itself as a break-up of bulk metal to metal powder. Corrosion degrades the useful properties of materials. Corrosion of metals and nonmetals takes place due to the gradual environmental interaction on the material surface. The structures and facilities of different materials are affected by this interaction. Even the ambient air, laden with moisture and oxygen, can start this process, known as rusting, on steel surfaces. In the case of buried structures and pipelines the soil chemistry and moisture determine the rate of damage. For example formation of oxides, diffusion of metal cations into the coating matrix, local pH changes and electrochemical potential. The study of corrosion of carbon steel and iron is a matter of tremendous theoretical and practical concern and as such has received a considerable amount of interest [1]. The consequences of corrosion are many and varied and the effects of these on the safe, reliable and efficient operation of equipment or structures are often more serious than the simple loss of a mass of metal. Failures of various kinds and the need for expensive replacements may occur even though the amount of metal destroyed is quite small [2]. Corrosion is process that damages metal due to a chemical reaction with the environment. It is a constant, continuous and costly problem often difficult to eliminate completely. The corrosion is minimized by using inhibitors. Corrosion is a natural process, which converts a refined metal to a more chemically-stable form, such as its oxide, hydroxide, or sulfide. It is the gradual destruction of materials (usually metals) by chemical and/or electrochemical reaction with their environment [3]. Inhibitors are used in the industrial process to control metal dissolution especially in acid, neutral and base environment. Most of the efficient inhibitors used in industry are the organic compounds that possess at least one functional group, which is considered as the active center for

the adsorption process. Several researchers made an attempt to study the inhibition action of various organic compounds on the corrosion of aluminium, alloys, mild steel, carbon steel and composites in acids, alkaline and neutral media. [4, 5]. The corrosion rate and inhibition efficiency was evaluated using weight loss method and electrochemical impedance spectroscopy. The protective film is formed on the mild steel surface was characterized with the help of surface analytical techniques such as fluorescence, FTIR and UV-Visible spectroscopy. The surface morphology has been analyzed by SEM. From SEM analysis we predict the smoothness of mild steel surface when compare to without inhibitor system and with inhibitor system.

Materials and Methods

Mild steel specimens; (0.026% S, 0.068% P, 0.36 % Mn, 0.13 % C and the rest iron) of dimensions 1.0 cm $\times 4.0 \times 0.2$ cm were polished to mirrors finish and degreased with acetone and used for mass loss method.

Weight loss method

Mild steel specimen triplicate were immersed in 100 ml beaker containing 100 ml of an aqueous solution$^-$ containing various concentrations of the glutaric acid inhibitor which is prepared by the standard available method [6, 7] for one day. After 24 hrs immersions the specimens were taken out, washed in running water, dried and weighed using a Shimadzu balance, model AY62.

The corrosion inhibition efficiency (IE) was calculated using the equation:

$$IE = 100[1-(w_2-w_1)] \%$$

Where w_1 is the mass loss in the absence of inhibitor and w_2 is mass loss in the presence of inhibitor.

Potentiodynamic Polarization study

Polarization studies were carried out in an H & CH electrochemical work station impedance analyzer model CHI 643B, Austin, USA three electrode cell assembly was used. The working electrode was mild steel with one face of the electrode of constant 1 cm^2 area exposed and the rest being shield with red lacquer. A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode. The area of the counter electrode was much larger when compared to the area of the working electrode. This can exert a uniform potential field on the counter electrode. The results such as the Tafel slopes, Corrosion Current (I_{corr}), Corrosion Potential(E_{corr}) and Linear Polarisation Resistance (LPR) values were calculated. The working electrode and platinum electrode were immersed in seawater in the absence and presence of inhibitor. Saturated calomel electrode was connected with the test solution through a salt bridge. Potential (E) Vs log current (I) plots were recorded.

AC impedance spectra

The instrument used for polarization study was also used for AC impedance spectra. The cell set up was the same as that was used for potentiodynamic polarization measurements. The real part (Z') and the imaginary part (Z'') of the cell impedance were measured in ohms at various frequencies. AC impedance spectra were recorded with initials $E_{(v)}$ =0V, high frequency limit was 1×10^5 Hz, low frequency limit was 1 Hz, amplitude =0.005V and quiet time t_q=2 s. The values of charge transfer resistance R_t and the double layer capacitance C_{dl} were calculated.

C_{dl}= ½ $\pi R_t f_{max}$

Where f_{max} is maximum frequency.

Surface Morphology studies

The mild steel specimens were immersed in various test solution for a period of 24 hrs. Then mild steel specimens were taken out and dried. The nature of the film formed on the surface of the mild steel specimen was analyzed by various surface analysis techniques.

UV-Visible absorption spectra of solutions

The possibility of the formation of iron inhibitor complex in solution was examined by mixing the respective solutions and recording their UV-Visible absorption spectra using carry Eclipse Varian (Model U.3400) UV-Visible spectrophotometer.

Surface analysis by fluorescence spectra

Fluorescence spectra of solutions and also the film formed on the mild steel surface were recorded in a Jasco-F-6300 Fluorescence Spectrophotometer.

Surface analysis by FTIR spectra

FTIR spectra were recorded in a Perkin Elmer Spectrum Version spectrophotometer. The film was carefully removed, mixed thoroughly with KBr made into pellets and the FTIR spectra were recorded. After immersion period of one day in various environments, the specimens were taken out of the test solutions and dried. The film formed on the surface was scratched carefully and it was thoroughly mixed so as to make it uniform throughout. FTIR spectrum of the powder (KBr pellet) was recorded using a Perkin Elmer Spectrum Version spectrophotometer.

Surface analyzing by Scanning Electron Microscopy (SEM)

The mild steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of the mild steel were examined using Tescan, Vega 3, USA computer controlled scanning electron microscope.

Results and Discussion

Analysis of the weight loss method

The corrosion rates (CR) of mild steel immersed in an aqueous solution containing 60 ppm of Cl^- and also inhibition efficiencies (IE) in the absence and presence of glutaric acid inhibitor obtained by weight loss method are given in Table.1. It is observed that 250 ppm of glutaric acid offers 78 % of inhibition efficiency in an aqueous solution containing 60 ppm of Cl^-. It is observed from Table 1 that glutaric acid shows good inhibition efficiency. As the concentration of glutaric acid increases, the IE increases. This is due to an increase of surface coverage at higher concentration of the glutaric acid which retards dissolution of mild steel. The electron donating properties of oxygen atoms can be attributed for higher inhibition efficiencies. This surveillance is in good agreement with the results reported by many researchers [8].

Table V.1 Corrosion rates (CR) and the inhibition efficiency (IE %) of mild steel immersed in an aqueous solution containing 60 ppm of Cl^- in the presence and absence of Glutaric acid systems at various concentration obtained by weight loss method.

➤ Inhibitor System: Glutaric acid (250 ppm)

➤ Medium : An aqueous solution containing 60 ppm of Cl^-

➢ Immersion period: 1 day

system	Glutaric Acid (ppm)	CR (mdd)	IE (%)
An aqueous solution containing 60 ppm of Cl$^-$	0	9.54	----
	50	9.09	50
	100	8.59	55
	150	6.68	65
	200	5.73	70
	250	4.21	78

Analysis of results of potentiodynamic polarization study

Polarization study has been used to confirm the formation of protective film on the mild steel surface during corrosion inhibition process [9-10]. If a protective film is formed on the mild steel surface, the linear polarization resistance values (LPR) increases and the corrosion current value (I_{corr}) decreases and corrosion potential increases (E_{corr}) [11 - 15].

The potentiodynamic polarization curves of mild steel immersed in an aqueous solution containing 60 ppm of Cl$^-$ and in the absence and presence of glutaric acid are shown in Fig 1. The corrosion parameters are given in Table 2. When mild steel was immersed in an aqueous solution containing 60 ppm of Cl$^-$ the corrosion potential was -0.288 mV Vs SCE. When glutaric

acid (250 ppm) was added to the above system, the corrosion potential shifted to the positive side -0.407mV Vs SCE. This indicates that the protective film is formed on the anodic sites of the mild steel surface. This film controls the anodic reaction of carbon steel dissolution by forming complex on the anodic sites of the mild steel surface [11-15]. Further, the LPR value in an aqueous solution containing 60 ppm of Cl^- increases from 45424.1 ohm cm^2 to 100738.5 ohm cm^2, the corrosion current decreases from $9.186 \times 10^{-7} A/cm^2$ to 3.979×10^{-7} A/cm^2. Thus polarization study confirms the formation of a protective film on the mild steel surface [16-20].

Table 2. Corrosion parameters of mild steel in an aqueous solution containing 60 ppm of Cl^- and in the absence and presence of Glutaric acid system obtained by potentiodynamic polarization method.

Systems	E_{corr} vs SCE (mV)	I_{corr} (A/cm^2)	ba (mV/dec)	bc (mV/ec)	LPR (ohmcm^2)
Blank An aqueous solution containing 60 ppm of Cl^-	- 0.288	9.186×10^{-7}	0.2197	0.1704	45424.1
An aqueous solution containing 60 ppm of Cl^- + 250 ppm of Glutaric Acid	-0.407	3.979×10^{-7}	0.1867	0.1820	100738.5
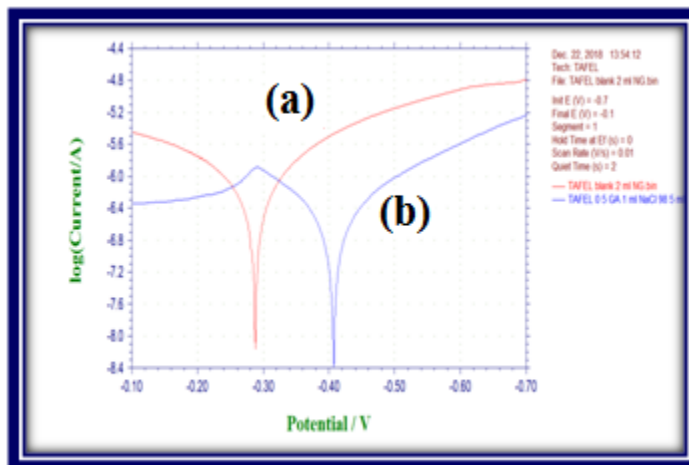
Fig.V.1: Polarization curves of mild steel immersed in test solutions

(a) Blank solution (an aqueous solution containing 60 ppm of Cl⁻)

(b) An aqueous solution containing 60 ppm of Cl⁻ + Glutaric Acid (250 ppm)

Analysis of results of AC impedance spectra

AC impedance spectra (electrochemical impedance spectra) have been used to confirm the formation of protective film on the mild steel surface [21]. If a protective film is formed on the mildn steel surface, charge transfer resistance (R_t) increases; double layer capacitance value (C_{dl}) decreases and the impedance log (z/ohm) value increases [10-13].

The AC impedance spectra of carbon steel immersed in an aqueous solution containing 60 ppm of Cl⁻in the absence and presence of glutaric acid are shown in Fig 2 (a & b) (Nyquist plots) and Figs 3 (a & b) (Bode plot). The AC impedance parameters namely charge transfer resistance (R_t) and double layer capacitance (C_{dl}) derived from Nyquist plots are given in Table 3. The impedance log (z/ohm) values derived from Bode plots are also given in Table 3. It is observed that when the inhibitor (250 ppm

of Glutaric Acid) is added, in the medium of an aqueous solution containing 60 ppm of Cl$^-$, the charge transfer resistance (R$_t$) increases from -3.358$\times$ 10^4 Ω cm^2 to 4.964 $\times$ 10^3 Ω cm^2. The C$_{dl}$ value decreases from 5.5307$\times$ 10^{-1} F cm^{-2} to 8.1758 $\times$ 10^{-2} F cm^{-2}. The impedance value [log (z/ohm)] increases from 1.291$\times$ 10^4 to 5.420$\times$ 10^4.These results lead to the conclusion that a protective film is formed on the mild steel surface.

Table 3: Corrosion parameters of mild steel immersed in an aqueous solution containing 60 ppm of Cl$^-$ in the absence and presence of Glutaric acid system obtained from AC impedance spectra.

Systems	Nyquist plot		Bode plot
	Rt Ω cm^2	Cdl F cm^{-2}	Impedance Lg (Z ohm^{-1})
Blank An aqueous solution containing 60 ppm of Cl$^-$	-3.358$\times$10^5	5.5307$\times$ 10^{-1}	1.291$\times$ 10^4

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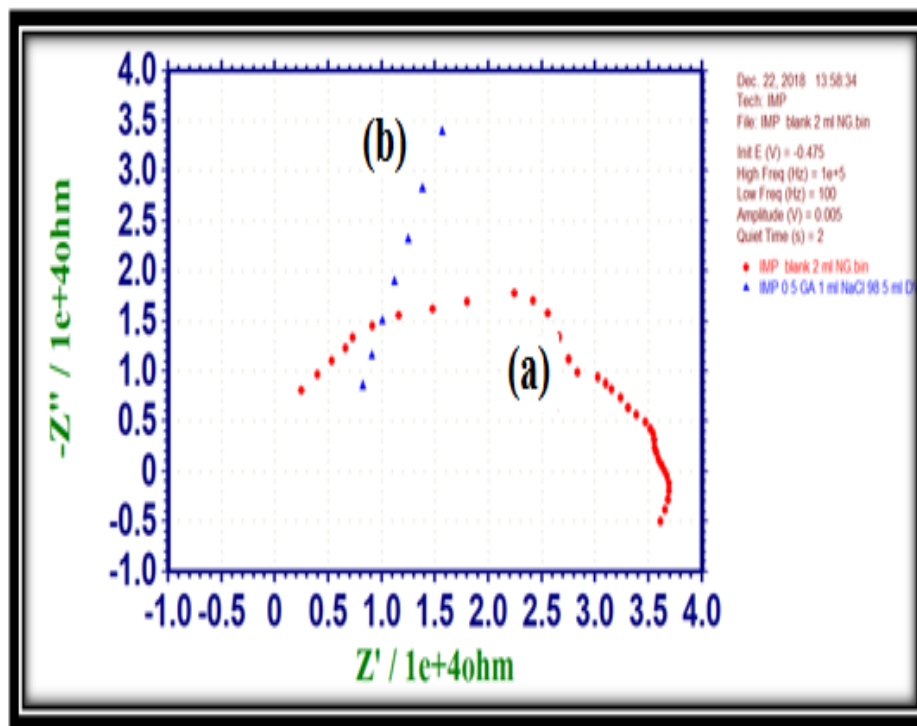|			
An aqueous solution containing 60 ppm of Cl⁻ + 250 ppm of Glutaric Acid	4.964×10^3	8.1758×10^{-2}	5.420×10^4

Fig.2: AC impedance spectra (Nyquist plots) of mild steel immersed in test solution

a)   Blank (an aqueous solution containing 60 ppm of Cl⁻)

b)   An aqueous solution containing 60 ppm of Cl⁻ + 250 ppm Glutaric Acid
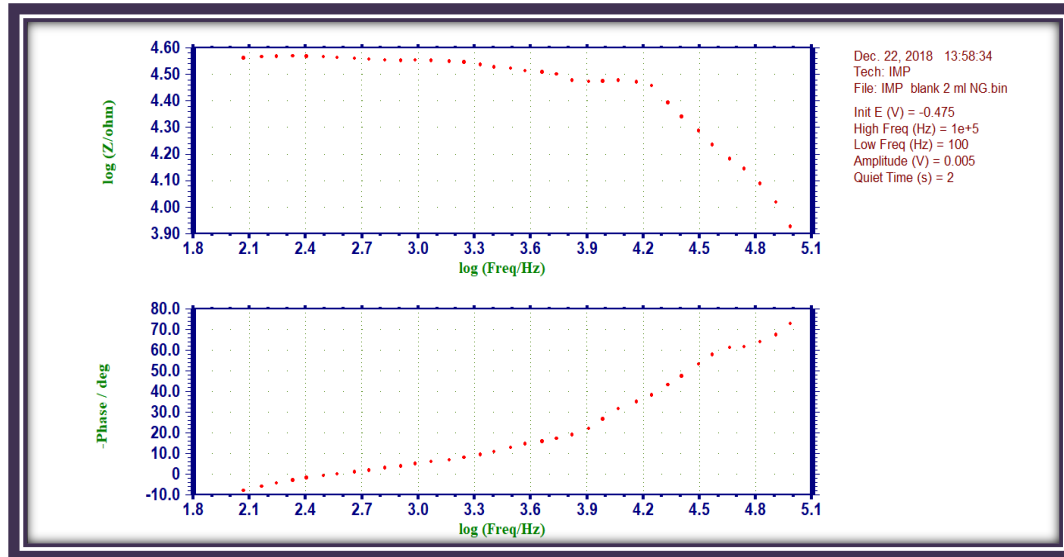
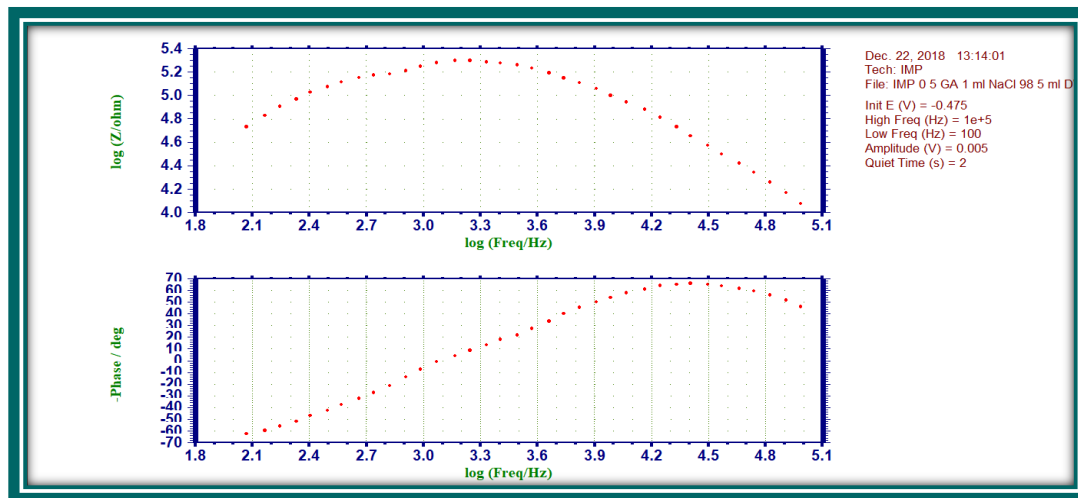Figure 3(a). AC impedance spectra (Bode Plot) of mild steel immersed in a test solution

Figure 3 (b).AC impedance spectra (Bode Plot) of mild steel immersed in a test solution + 250

ppm of Glutaric Acid

Analysis of UV-Visible absorption spectra

The UV-Visible absorption spectra of a test solution containing glutaric acid are shown in Figure 4 (a, b). Peaks appear at 242 nm and 278 nm. When Fe^{2+} solution is added to the solution new peaks appear at 309 nm and 401 nm. This indicates that a complex is formed between Fe^{2+} - Glutaric Acid in solution [26, 27].
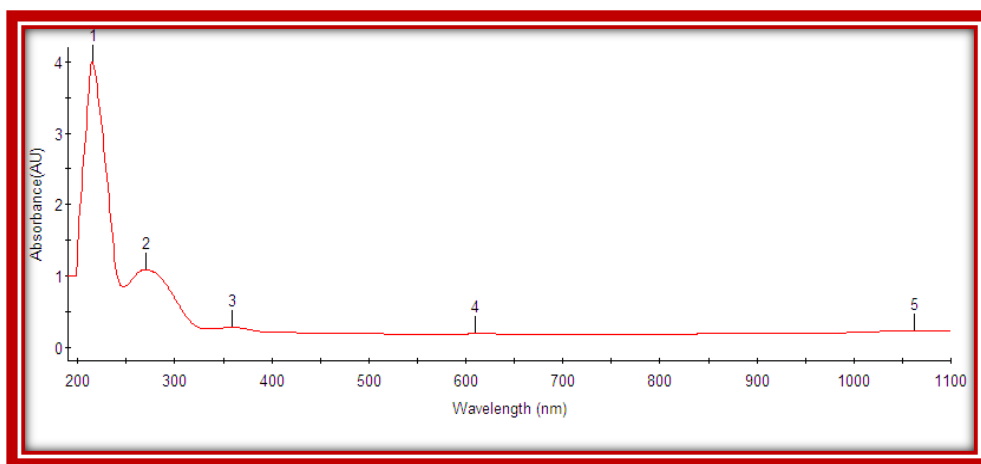

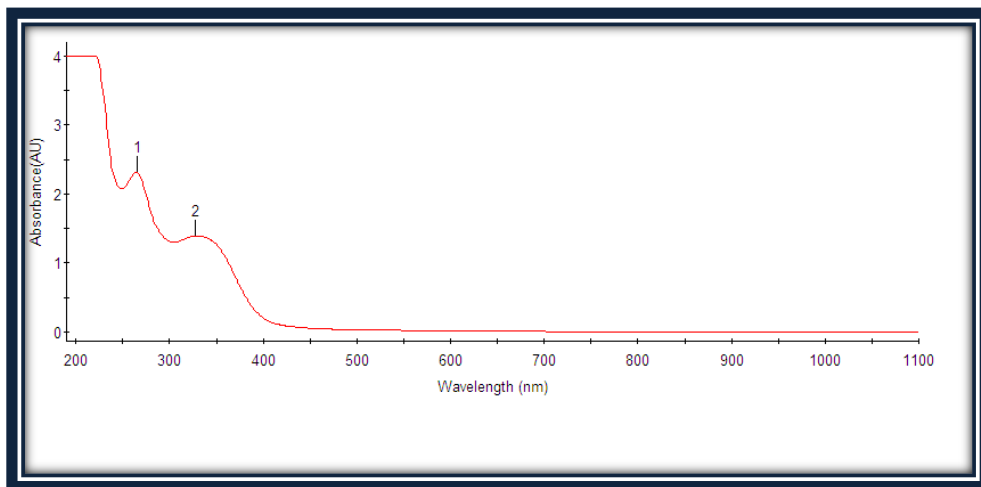Fig 4 (a). UV adsorption spectrum of solution containing glutaric Acid

Fig 4(b). UV adsorption spectrum of solution containing (Fe^{2+}) + glutaric Acid

Analysis of the Fluorescence spectra

Fluorescence spectra have been used to detect the presence of Fe^{2+}-glutaric acid complex formed on the mild steel surface [28, 29].The emission spectrum of the Fe^{2+} - glutaric acid solution, prepared by mixing an aqueous solution of Fe^{2+} (freshly prepared from FAS) and glutaric acid is shown in Fig.5a. A peak appears at 647 nm.

The emission spectrum of the film formed on the mild steel surface after immersion in an aqueous solution containing 60 ppm of Cl^- and 250 ppm glutaric acid is shown in figure 5b. A peak appears at 641 nm. This indicates that the film present on the mild steel surface consist of Fe^{2+}-glutaric acid complex. Further, this value is very close to that of Fe^{2+}-glutaric acid complex in solution. The number of peak obtained is only one in that position. Hence it is inferred that the complex is of somewhat highly symmetric.
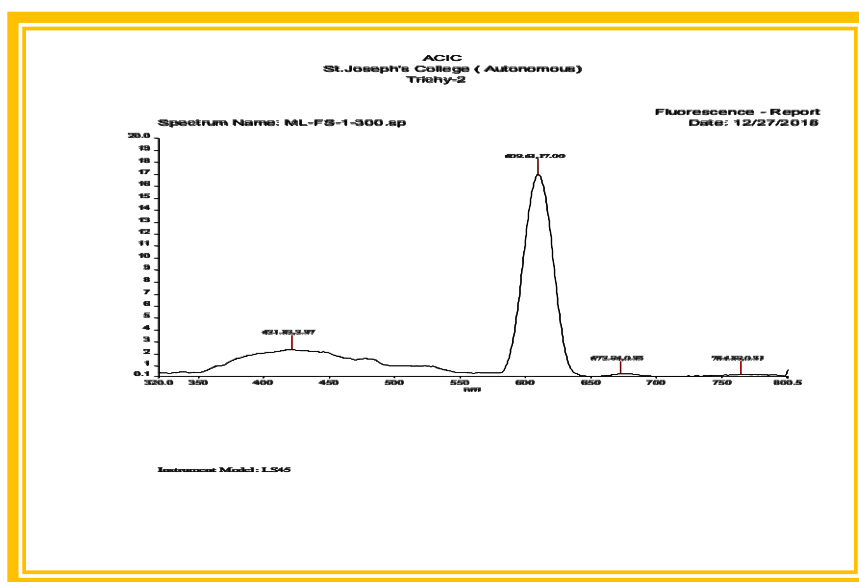
Fig 5(a) .Fluorescence spectrum of solution containing Fe^{2+} -Glutaricacid complex
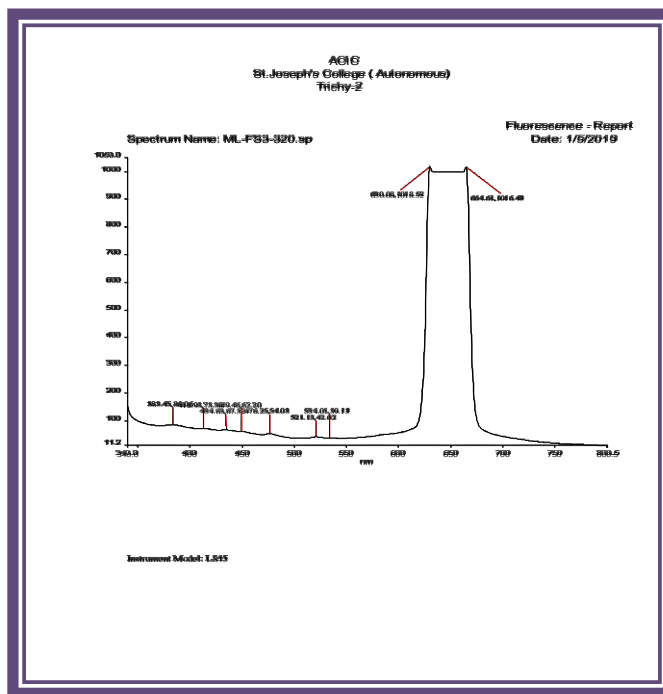

Fig 5 (b) Protective film formed on the surface of mild steel after immersion in an aqueous

solution containing 60 ppm of Cl$^-$ solution+ 250 ppm of Glutaric acid

Analysis of FTIR spectra

FTIR spectra have been used to analyze the protective film formed on the mild steel surface [30].

The structure of glutaric acid is shown in figure 6. The FTIR spectrum (KBr) of pure glutaric

acid is shown in Fig.7(a) and the values are in the table 4. The peak due to C=O appears at

1696.43 cm^{-1}. The peak appears at 3045.05.53 cm^{-1} due to OH stretching frequency. The C-H

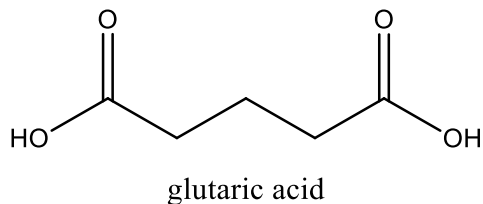stretching frequency appears at 2904.52 cm^{-1}.

glutaric acid

Figure 6

The FTIR spectrum (KBr) of the film formed on the mild steel surface after immersion in in an aqueous solution containing 60 ppm of Cl^- + 250 ppm of glutaric acidic shown in Fig. 7(b) and the values are in table 4. The C=O stretching has shifted from 1696.43 cm^{-1} to 1712.26 cm^{-1}. The CH stretching frequency has shifted from 2904.52 cm^{-1} to 2926.59 cm^{-1}. The O-H stretching frequency has shifted from 3045.05 cm^{-1} to 3418.26 cm^{-1}. A new peak appears at 698.90 cm^{-1}due to M-O which confirms the formation of Fe – inhibitor complex [19-22]. This observation suggests that Glutaric Acid has coordinated with Fe^{2+} through the oxygen atom resulting in the formation of Fe^{2+}-glutaric acid complex on the anodic sites of the mild steel surface. Thus the FTIR spectral study leads to the conclusion that the protective film consists of Fe^{2+}-glutaric acid.

Table 4. IR values of pure Glutaric acid and film formed over the mild steel surface is obtained from IR Spectra.

	Pure Glutaric Acid	The Protective film

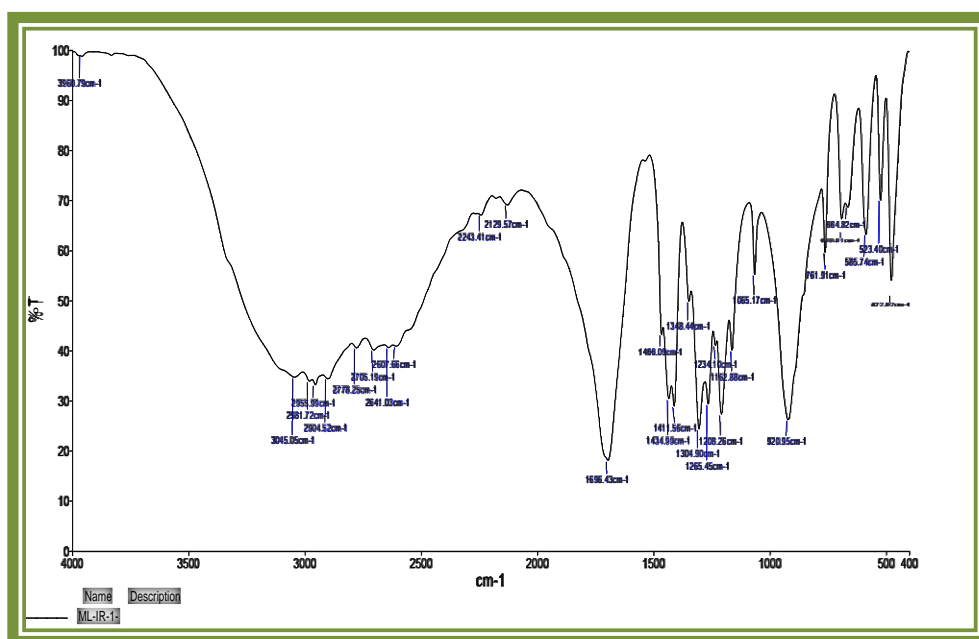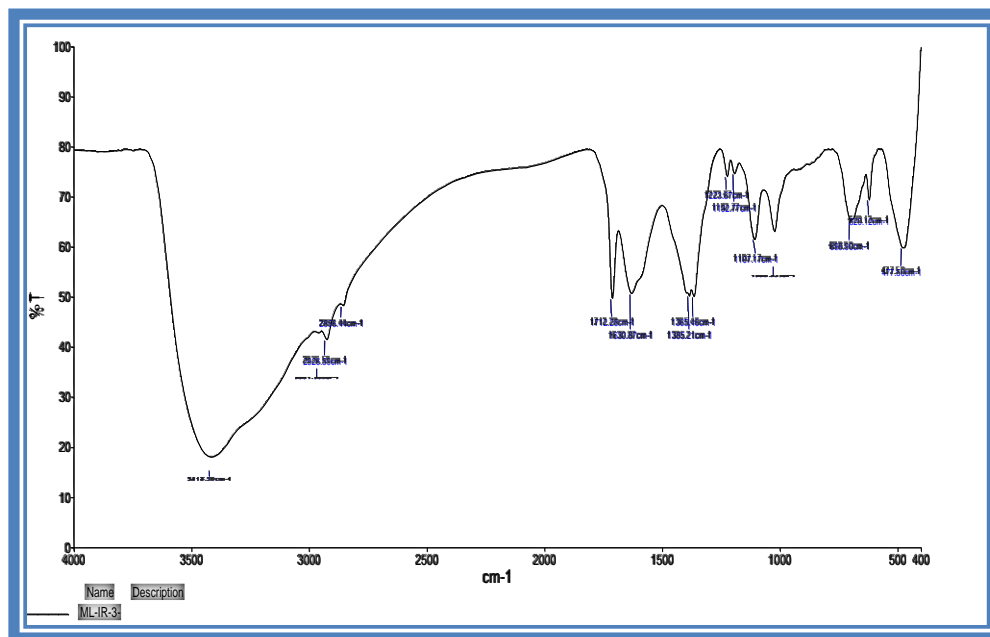| Functional Group	Compound (Peak Appears at cm^{-1})	consists of Fe^{2+} - Glutaric Acid (Peak Appears at cm^{-1})
C=O	1696.43	1712.26
CH	2904.52	2926.59
O-H	3045.05	3418.26
M-O	-	698.90

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Fig.7 (a). FTIR Spectrum of pure glutaric acid

Fig.7 (b). FTIR spectrum of film formed on mild steel surface after immersion in an aqueous

solution containing 60 ppm of Cl$^-$ + 250 ppm of Glutaric acid

SEM Analysis of mild steel Surface

SEM provides a pictorial representation of the surface of the mild steel. To understand the nature of the surface film in the absence and presence of inhibitor and the extent of corrosion of mild steel, the SEM micrographs of the surface are examined [35-36].

The SEM images of different magnification (X 10, X 100) of mild steel specimen immersed in an aqueous solution containing 60 ppm of Cl$^-$ for one day in the absence and presence of inhibitor system are shown in Fig.8 (a,b,c,d,g and h) respectively.
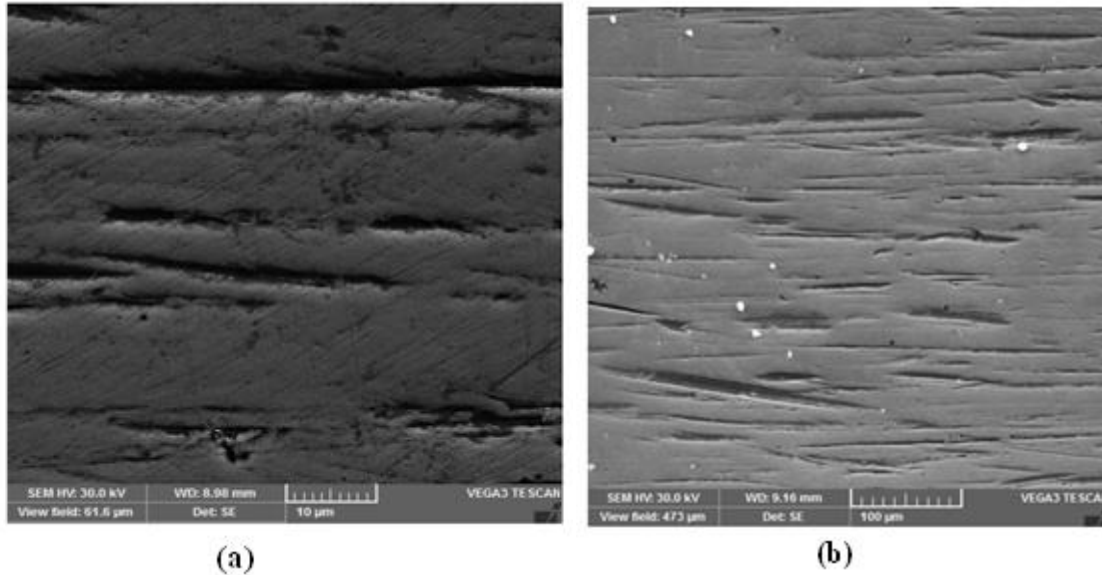
(a)   (b)

Fig.8: SEM analysis of

(a) Mild steel; Magnification X 10 (control)

(b) Mild steel; Magnification X 100 (control)

The SEM micrographs of polished mild steel surface (control) in Fig.V.8 (a and b) shows the smooth surface of the mild steel. This shows the absence of any corrosion products or inhibitor complex formed on the mild steel surface.
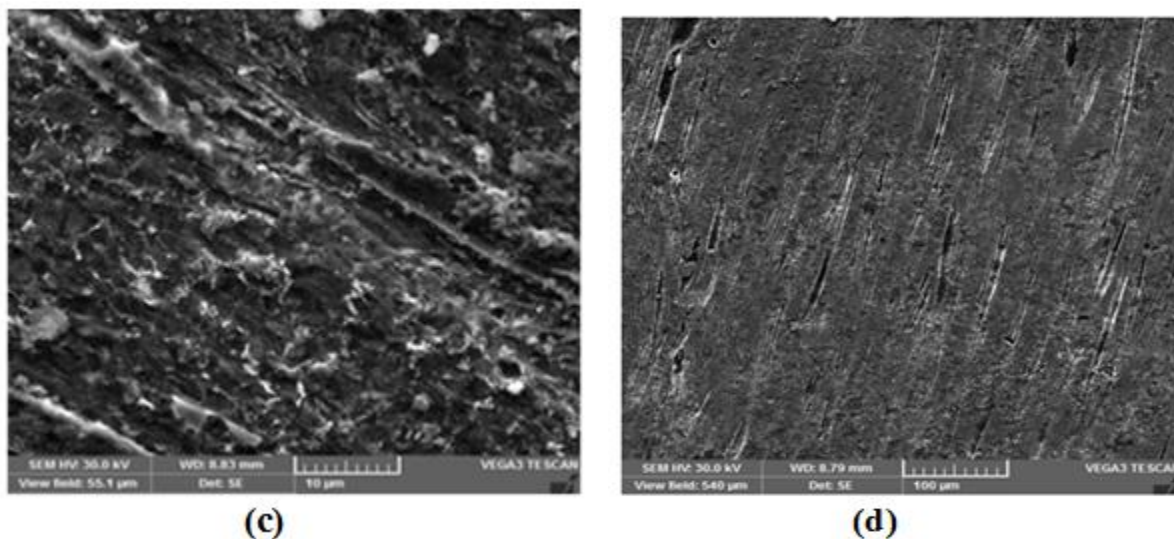

(c)   (d)

Fig.8: SEM analysis of

(c) Mild steel in an aqueous solution containing 60 ppm of Cl^- Magnification X 100

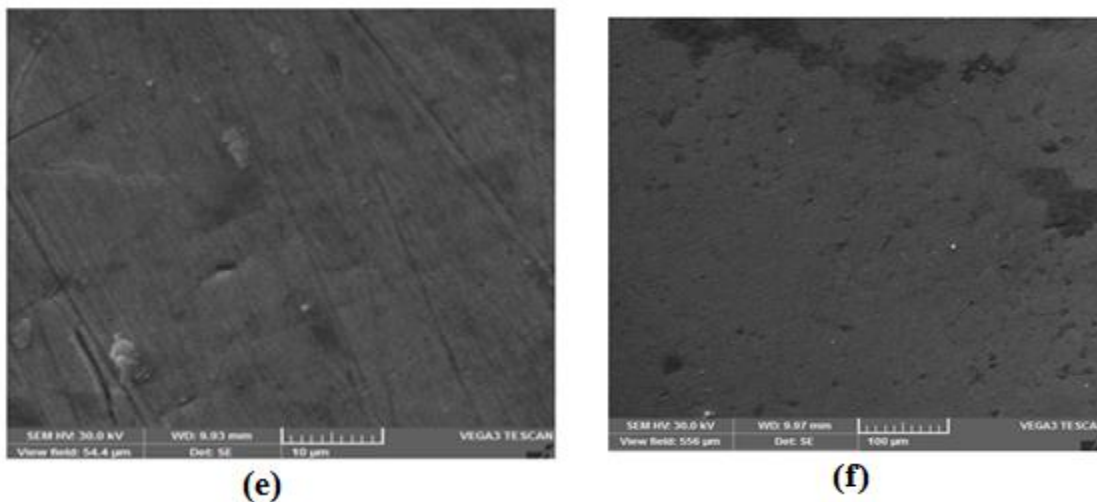(d) Mild steel in an aqueous solution containing 60 ppm of Cl^- Magnification X 100

(e)

(f)

Fig.8: SEM analysis of

(e) Mild steel in an aqueous solution containing 60 ppm of Cl^- + 250 ppm of Glutaric acid Magnification X 10

(f) Mild steel in an aqueous solution containing 60 ppm of Cl^- + 250 ppm of Glutaric acid Magnification X 100

The SEM micrograph of mild steel surface immersed in an aqueous solution containing 60 ppm of Cl^- (Fig.8 [c and d]) show the roughness of the mild steel surface which indicates the highly corroded area of mild steel in an aqueous solution containing 60 ppm of Cl^-. However in Fig.8 (e and f) (Mild steel in an aqueous solution containing 60 ppm of Cl^- + 250 ppm of Glutaric acid) indicate that in the presence of inhibitor, the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The mild steel surface almost free from corrosion due to the formation of insoluble complex on the surface of the mild steel [36]. In the presence of glutaric acid, the surface is covered by a thin layer of inhibitor which effectively controls the dissolution

of mild steel.

Mechanism of corrosion inhibition

It is clearly that organic molecule which is glutaric acid carry their inhibition action via the adsorption on the mild steel surface. The adsorption process depends on the chemical structure of the inhibitor, the nature and charged surface of the metal and the distribution of charge over the whole inhibitor molecule. In general, owing to the complex nature of adsorption and inhibition action of glutaric acid, it is impossible for single adsorption mode between glutaric acid and mild steel surface.

Organic inhibitor molecules may be adsorbed on the mild steel surface in one or more of the following ways:

(a) Electrostatic interaction between the charged molecules and the charged mild steel,

(b) Interaction of unshared electron pairs in the molecule with the mild steel,

(c) Interaction of π-electrons with the mild steel [23-24]

Generally, Heteroatom's of inhibitor may be adsorbed on the surface of mild steel through the chemisorption mechanism, involving the displacement of water molecules from the mild steel surface and the sharing electrons between the inhibitor atoms and iron.

The glutaric acid molecules can also adsorb on the mild steel surface on the basis of donor-acceptor interactions oxygen atom of inhibitor and vacant d-orbital of surface iron.

The results of the weight loss study show that the formulation consisting of 250 ppm of glutaric acid has 78% IE in controlling corrosion of mild steel in an aqueous solution containing 60 ppm

of Cl^-. Polarization study reveals that, formulation function as anodic inhibitor.AC impedance spectra reveal that a protective film is formed on the mild steel surface. FTIR spectra reveal that the protective film consists of Fe^{2+}-glutaric acid complex.

In order to explain these facts, the following mechanism of corrosion inhibition is proposed.

➢ An aqueous solution containing 60 ppm of Cl^- and 250 ppm of glutaric acid is prepared. Glutaric acid is ionized in this solution.

➢ When mild steel is immersed in this solution, the ionized glutaric acid goes to the surface of the mild steel and is converted in to a Fe^{2+} -glutaric acid complex which is more stable.

Glutaric acid + Fe^{2+} $\longrightarrow$ Fe^{2+}-Glutaric acid

➢ Thus the protective film consists of Fe^{2+} - Glutaric acid complex. This is confirmed by FTIR spectra.

➢ The SEM micrographs confirm the formation of protective film over the mild steel Surface.

Conclusion

The glutaric acid inhibitor has potent to inhibit the corrosion of mild steel is immersed in an aqueous solution containing 60 ppm of Cl^-. The inhibition efficiency increased with the increasing concentration of inhibitor. The maximum inhibitor efficiency was observed at an optimum concentration of 250 ppm is 78%. Data obtained from the conventional weight loss method and electrochemical measurements have shown that the

compound has excellent inhibiting properties for mild steel in an aqueous solution containing 60 ppm of Cl^-.

➢ The weight loss technique shows maximum the inhibition efficiency is 78%.

➢ Corrosion parameters like I_{corr}, E_{corr}, b_a, b_c, R_{ct} and C_{dl} values were evaluated using electrochemical measurements.

➢ The results of potentiodynamic polarization measurements revealed that the glutaric acid act as adsorbed type of inhibitor in an aqueous solution containing 60 ppm of Cl^-r that could effectively suppress anodic and processes through the chemical adsorption on mild steel surface.

➢ Decrease in I_{corr} and C_{dl} values and increase in R_t values confirmed the inhibition action of the inhibitor, owing to increased thickness of the adsorbed layer.

➢ Electrochemical impedance measurements indicate that an increase the charge transfer resistance (R_t), decrease the double layer capacitance (C_{dl}) and corrosion current ($_{Icorr}$) values owing to the increased thickness of adsorbed layer.

➢ FTIR spectra of the film products of mild steel immersed in glutaric acid in an aqueous solution containing 60 ppm of Cl^- showed either a decrease in the transmittance or disappearance of some of the bands, giving a strong evidence for the interaction between the metal and the functional groups such as OH, CH and C=O leading to the formation of film of large surface coverage which served as a barrier between the corrosive acid medium and the metal thereby inhibiting corrosion. FTIR spectra reveal that the protective film consists of Fe^{2+}- glutaric acid complex.

➢ Surface studies involving SEM confirmed the efficiency of the glutaric acid as corrosion inhibitor for mild steel. Surface smoothness is in relation to the concentration of the glutaric acid.

➢ The comparison of the images from scanning electron microscopy revealed that the molecules of the glutaric acid are adsorbed on the carbon steel surface, thereby decreasing the corrosion attack on the mild steel surface.

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