

Corrosion behavior of Mild Steel in Sodium Chloride solution by Copper Complex of (8-hydroxy quinoline) Derivative

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Abstract

The Corrosion behavior of a inhibitor namely Cu complex of 8-hydroxy quinoline derivative (Cu-HUF) in controlling corrosion of mild steel immersed in aqueous solution containing 60 ppm Cl^- has been investigated using weight loss method and electrochemical impedance spectroscopy. In presence of inhibitor the corrosion inhibition efficiency offered by 50 ppm of Cu-HUF is 80 %. The corrosion inhibition was observed due to the formation of more stable and compact protective film on the mild steel surface at minimum concentration. The surface film formed over the mild steel surface was confirmed by IR spectra. The surface of the samples exposed to the inhibited solution was analysed using SEM and EDAX techniques. The biocidal efficiency of the system has been evaluated against certain microorganisms such as E.Coli, Streptococcus, Pseudomonas, Entrobacter which shows good biocidal efficiency.

Key Words: Biocidal, Corrosion inhibition, EIS, Mild Steel, SEM, Transition metal complex.

Introduction

Corrosion caused by the weakening of material, usually metal or alloy by chemical or electrochemical reaction with their environment. Mild steel under water containing chloride is corroded slowly by means of chemical reactions [1-3]. The corrosion process will be very fast due to the presence of chloride ions and dissolved oxygen in aqueous solution. Metal and alloy is extensively used in infrastructure in marine environments [4]. Mild steel is widely used in industries. It comes in close with chloride ions very regularly under several circumstances and

situation in industries. In many industries the corrosion of metals and alloys is a severe trouble, installations, and social military such as water and dirt equipment. One of the most practical, easy and useful methods that used to manage and defend metals against decay is the use of inhibitors, particularly in neutral and acid media. The corrosion could be controlled by adding inhibitor to the corrosive media. Corrosion inhibitors are substances which when it is added in small quantity to corrosive medium decreases or avoid the reaction of the metal with the media [5]. Metal complexes are widely used as catalyst of chemical reactions, e.g. Oxidative dehydrogenation (ODH) of ethane and epoxidation of geraniol and as stabilizer or precursor in sol-gel processes [6-7]. Interaction of transition metal complexes with mild steel is greatly affected by their standard electrode potentials, their reactivity and the nature of the ligand that could stabilize the metallic complexes. Reduction of Cu (II) and Co (II) species on mild steel surface is possible due to their noble standard electrode potential compared to Fe (II). However, it should be noted that negative charged ligands like nitro, thiocyanate, Oxalato, glucinato and acetylacetonate could stabilize the higher oxidation states. Electrochemical impedance spectroscopy (EIS) as powerful non destructive test could be used to extract electrochemical parameters involved in corrosion process. Extracted parameters are useful to evaluate corrosion protection performance of inhibitors, organic and inorganic coating [8]. The aim of the present study is to evaluate corrosion resistance of copper complex of 1-(8-hydroxy quinolin-2yl-methyl) urea to mild steel immersed in aqueous solution containing 60 ppm Cl^- . The corrosion inhibition efficiency was evaluated using weight loss method and electrochemical impedance spectroscopy. The protective film formed on the metal surface characterized with the help of IR spectra. The surface of the samples exposed to the inhibited solution was analysed using SEM techniques. The EDAX technique useful to measure the elements presents on the mild steel surface. The biocidal efficiency of the system has been evaluated against certain microorganisms such as E.Coli, Streptococcus, Pseudomonas, Entrobacter which shows good biocidal efficiency.

Experimental Details

Mild steel specimens Preparation

The experiments were performed with mild steel specimens. Mild steel strips with the composition of 0.028% S, 0.066% P, 0.37 % Mn, 0.12 % C and iron remainder and size of 1.0 cm ×4.0×0.2 cm were used for mass loss measurements.

Weight loss Measurements

Mass loss measurements were performed at room temperature for 24 hours immersing the mild steel strips into an aqueous solution containing 60 ppm of Cl⁻ without and with various amount of inhibitor. After the elapsed time, the specimens were taken out washed, dried and weighed accurately.

AC impedance spectra

Impedance spectra were carried out in a CHI electrochemical workstation with impedance model 643, Austin, USA. A three electrode cell assembly was used. The working electrode was mild steel. The exposed surface area was 1 cm². A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode. 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.

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

Where f_{max} is maximum frequency.

Surface Characterization studies

The mild steel specimens were immersed in various test solution for a period of one day. After one day 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.

Surface analysis by FTIR spectroscopy

The spectra were recorded in a Bruker (Model Tensor 27) spectrophotometer. The film was carefully removed, mixed thoroughly with KBr and made into pellets and the FTIR spectra were recorded.

SEM Analysis

SEM (Tescan, Vega 3, USA) provides a pictorial representation in the surface to understand the nature of the surface film in the absence and presence of inhibitors and extent of corrosion of mild steel. The SEM micrographs of the surface are examined.

Energy Dispersive Analysis of X-rays (EDAXs)

The mild steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, dried and observed in an Energy Dispersive Analysis of X-rays (EDAXs) to examine the elements present on the metal surface. The elements present on the metal surface were examined using Bruker computer controlled Energy Dispersive Analysis of X-rays (Bruker, Nano, GMBH, Germany).

Bacterial Enumeration

Bacterial cell are count based on their colony forming unit (CFU) by standard plate count. The protocols are as follows. Label the plate 10^{-2} , 10^{-4} , 10^{-6} and 10^{-8} at the bottom of plate and one plate as a blank or control. Using aseptic technique transfer 1ml of four different cultures to 99ml of sterile saline blank. A test tube labelled 10^{-2} and the dilution is proceeded by taking 1ml from 10^{-2} test tube to the next 99ml sterile saline blank labeled as to the next 99ml sterile saline blank

labeled as 10^{-4} and proceed until 10^{-8} . Shake all the test tubes for equal distribution of bacteria. Transfer 1ml of the sample into each of the labelled plates 10^{-2} to 10^{-8} correspondingly. Containing agar medium all the plates are immersed and incubate at 37°C for 24 hours. After the incubation, select the plate contains 30 to 300 colonies and counts the colonies. The number of CFU are calculated as $\text{CFUs/dilution} \times \text{amount plated} = \text{No.of bacterial cell / ml}$.

Results and Discussion

The corrosion rates (CR) of mild steel immersed in aqueous solution containing 60 ppm Cl^- and also inhibition efficiencies(IE) in the absence and presence of inhibitor Copper complex of 8-hydroxy quinoline obtained by weight loss method are given in Table 1. It is observed from Table-1 that Cu-HUF shows 80 % inhibition efficiency [9].

Cl^- (ppm)	Cu-HUF (ppm)	CR (mdd)	IE (%)
60	0	34.55	-
60	50	6.91	80
60	100	8.98	75
60	150	10.36	71
60	200	12.78	64
60	250	14.51	59

Table1. Corrosion rates (CR) of mild steel immersed in an aqueous solution containing 60 ppm Cl^- in the presence and absence of Cu-HUF inhibitor systems at various concentrations and the inhibition efficiency (IE %) obtained by weight loss method.

AC impedance spectra

AC impedance spectra [electrochemical impedance spectra] have been used to confirm the formation of protective film on the metal surface [8, 10-13]. The AC impedance spectra of mild steel immersed in aqueous solution containing 60ppm of Cl^- in the absence and presence of inhibitors are shown in Fig.1(Nyquist plots) and Fig.2 (Bode plots). The impedance parameters namely charge transfer resistance (R_t) double layer capacitance (C_{dl}) and impedance $\lg(z/\text{ohm})$ are given in Table-2. If a protective film is formed on the metal surface, R_t value increases and the C_{dl} value decreases.

Systems	R_t $\Omega \text{ cm}^2$	C_{dl} F cm^{-2}	Impedance, Log ($Z \text{ ohm}^{-1}$)
60 ppm Cl^-	20.19	5.235×10^{-5}	0.973
60 ppm $\text{Cl}^- + 50$ ppm Cu- HUF	182.11	5.804×10^{-6}	1.944

Table2. The AC impedance spectra of mild steel immersed in aqueous solution containing 60ppm of Cl^- in the absence and presence of Cu-HUF inhibitor system.

When mild steel is immersed in aqueous solution containing 60 ppm of Cl^- , R_t value is $20.19 \Omega \text{ cm}^2$ and C_{dl} value is $5.235 \times 10^{-5} \text{ F cm}^{-2}$. When Cu-HUF are added to the aqueous solution containing 60ppm of Cl^- R_t value increases from $20.19 \Omega \text{ cm}^2$ to $182.11 \Omega \text{ cm}^2$ and the C_{dl} value decreases from $5.235 \times 10^{-5} \text{ F cm}^{-2}$ to $5.804 \times 10^{-6} \text{ F cm}^{-2}$. The impedance value increases from 0.973 to 1.944. This account for the high inhibition efficiency of Cu-HUF system and a

protective film is formed on the metal surface. This is also supported by the fact that for the inhibitor system the phase angle increases from 47.88 to 62.10 ° (Fig.2).

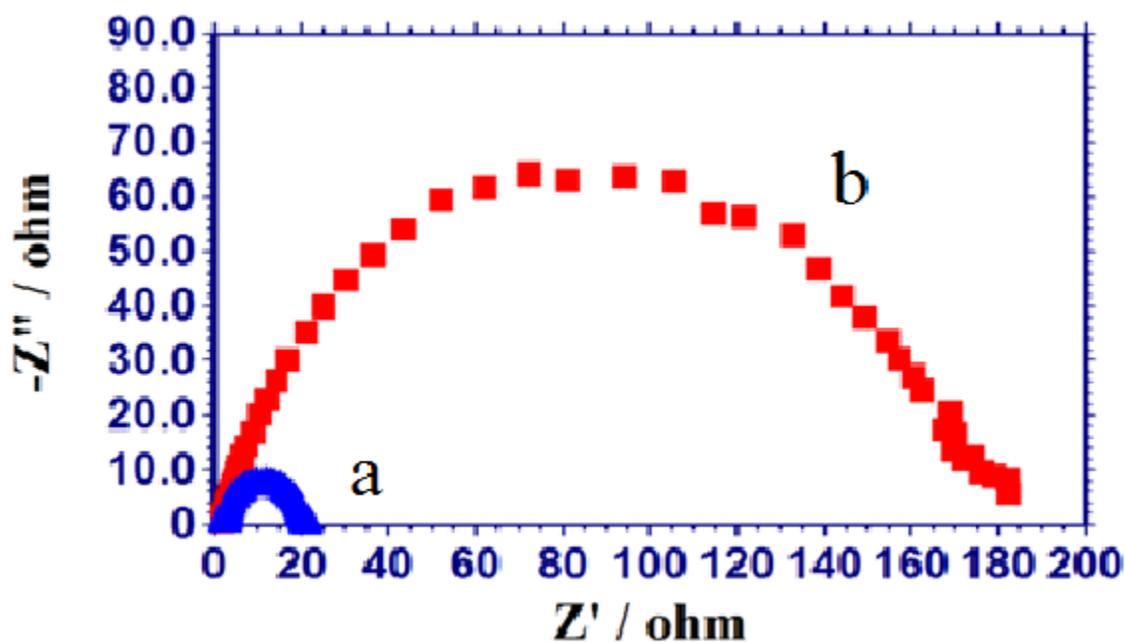


Figure 1.AC impedance spectra (Nyquist plots) of mild steel immersed in various test solutions
a) aqueous solution containing 60ppm of Cl^- . b) aqueous solution containing 60ppm Cl^- + 50 ppm Cu-HUF

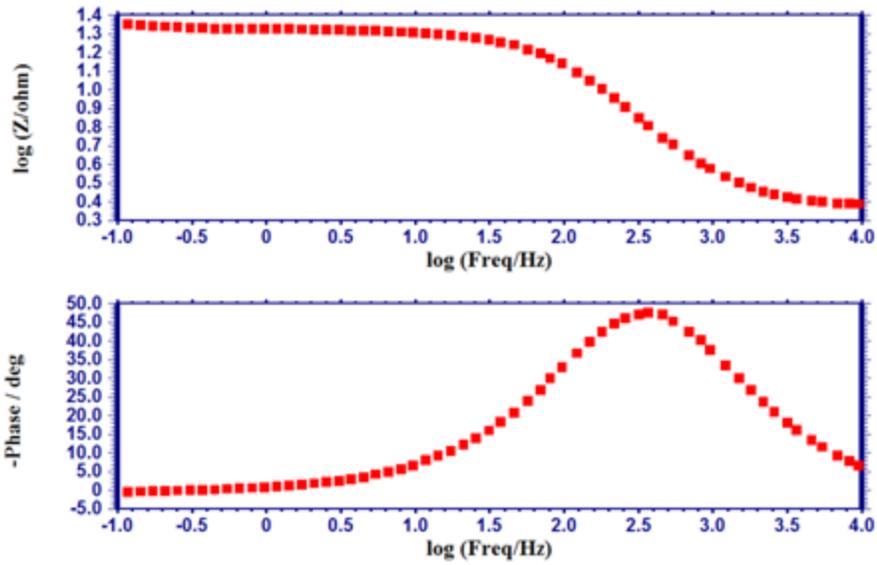


Figure 2a. AC impedance spectra (Bode Plot) of mild steel immersed in aqueous solution containing 60ppm of Cl^-

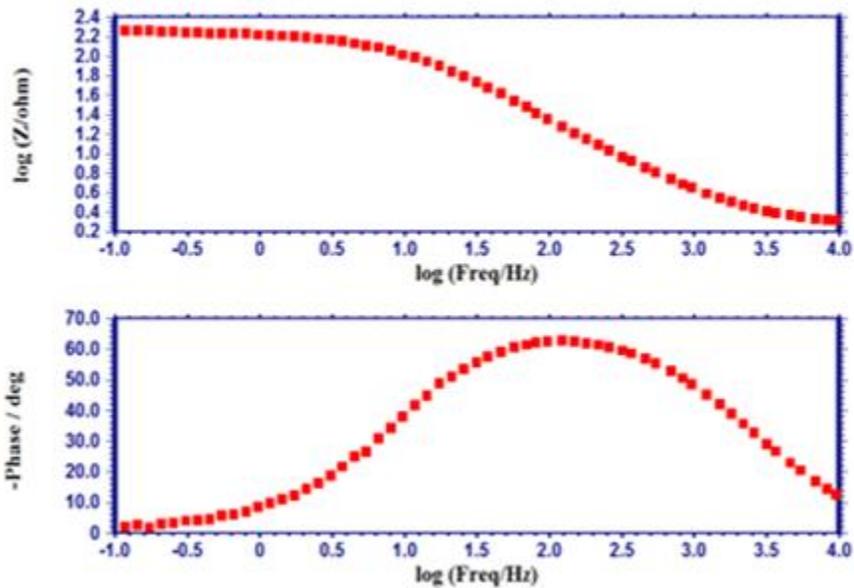
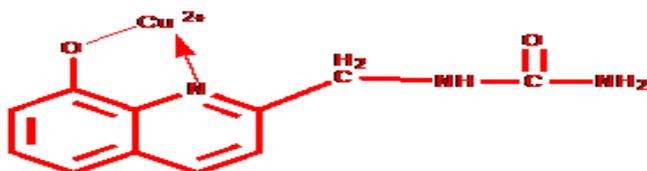


Figure 2b. AC impedance spectra (Bode Plot) of mild steel immersed in solution containing 60 ppm of Cl^- + 50ppm of Cu- HUF

Analysis of FTIR spectra

FTIR spectra have been used to analyze the protective film formed on the mild steel surface [14-20]. The structure of Cu-HUF is shown in scheme 1. The FTIR spectrum (KBr) of pure Copper complex of 1-(8-hydroxy quinolin-2-yl-methyl) urea (Cu-HUF) is shown in Fig.3a. The C=O stretching frequency appears at 1609.50 cm^{-1} . The aliphatic CH stretching frequency appears at 2887.03 cm^{-1} . The peak due to secondary nitrogen (NH) appears at 2978.00 cm^{-1} . The peak due to pyridine nitrogen (C=N) appears at 1398.33 cm^{-1} . The peak due to aromatic C=C appears at 1520.98 cm^{-1} . The peak due to primary nitrogen appears in the region of 3500.01 cm^{-1} .

The FTIR spectrum (KBr) of the film formed on the mild steel surface after immersion in an aqueous solution containing 60 ppm of Cl^- + 50 ppm of Cu-HUF is shown in Fig.3b. The pyridine nitrogen (C=N) stretching has shifted from 1398.33 cm^{-1} to 1410.10 cm^{-1} . The C=O stretching frequency has appeared very slightly in the region 1610.50 cm^{-1} . The aliphatic CH stretching frequency appears at 2900.03 cm^{-1} . This observation suggests that HUF has coordinated with Fe^{2+} through the oxygen atom of phenolic group and nitrogen atom of pyridine ring resulting in the formation of Fe^{2+} -HUF complex on the cathodic sites of the mild steel surface. The peak at 498.00 cm^{-1} corresponds to Cu-O stretching. The peak at 3460.50 cm^{-1} is due to OH^- stretching. This confirms that $\text{Cu}(\text{OH})_2$ is formed on the anodic sites of the mild steel surface [19-20]. Thus the FTIR spectral study leads to the conclusion that the protective film consists of Fe^{2+} - HUF complex and $\text{Cu}(\text{OH})_2$.



Scheme-I

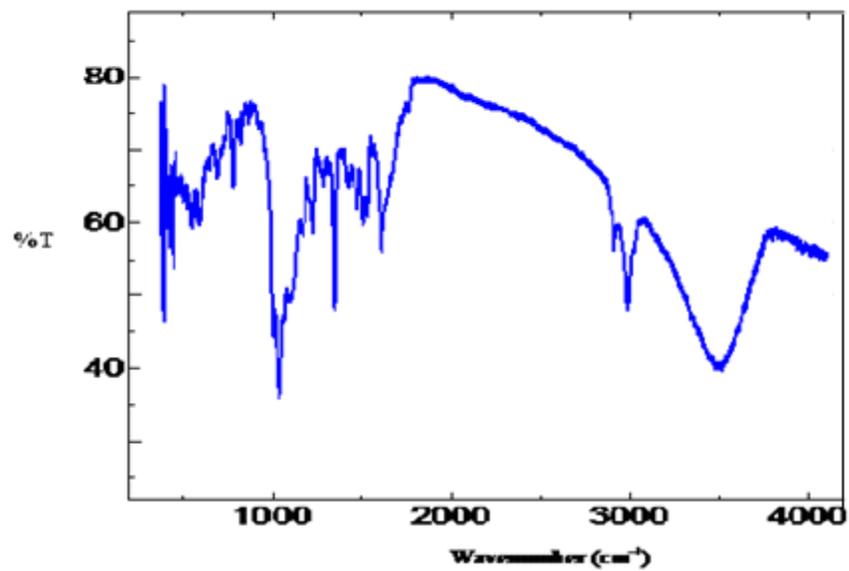


Fig..3a. FTIR spectrum of pure Cu-HUF

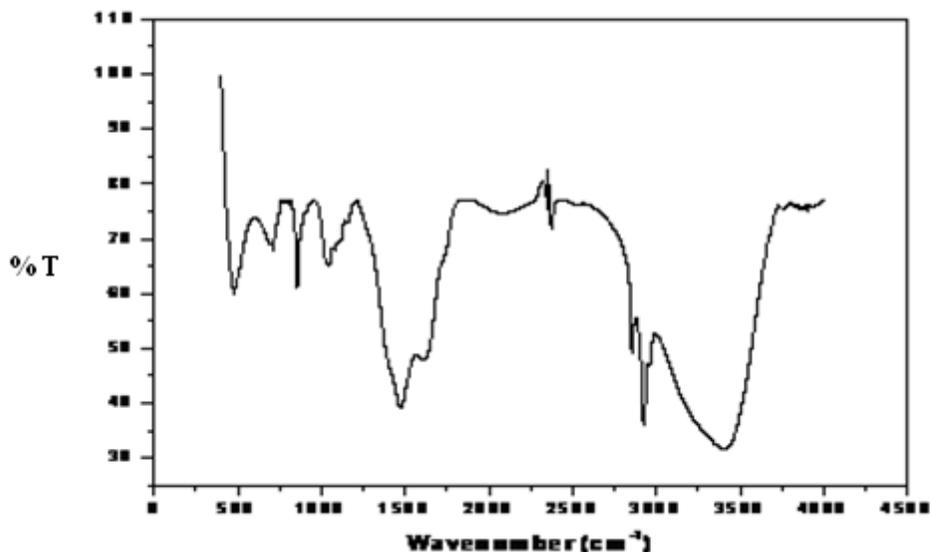


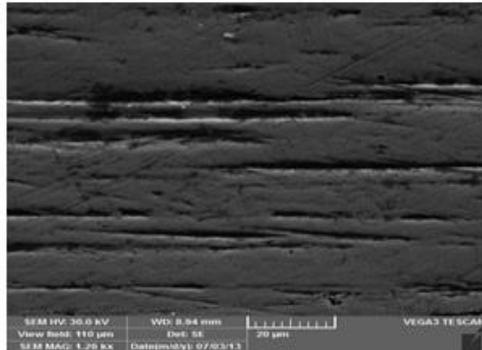
Fig.3b. FTIR spectrum of film formed on the mild steel surface after immersion in anaqueous solution containing 60 ppm of Cl^- + 50 ppm of Cu-HUF

SEM Analysis of Mild steel Surface

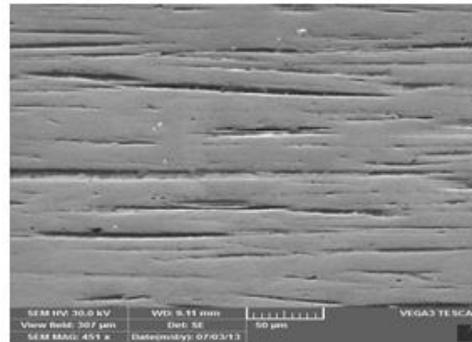
SEM provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and presence of inhibitors and the extent of corrosion of mild steel, the SEM micrographs of the surface are examined [21-23]. The SEM images of different magnification (X 20, X 50) of 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.4 (a,b) and Fig.4 (c,d,e,f) respectively. The SEM micrographs of polished mild steel surface (control) in Fig.4 (a,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.

The SEM micrograph of mild steel surface immersed in an aqueous solution containing 60 ppm of Cl^- (Fig.4 (c,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.V.4 (e,f) indicate that in the presence of inhibitor (50 ppm of Cu-HUF)

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 [21]. In the presence of Cu-HUF, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of mild steel.



(a)

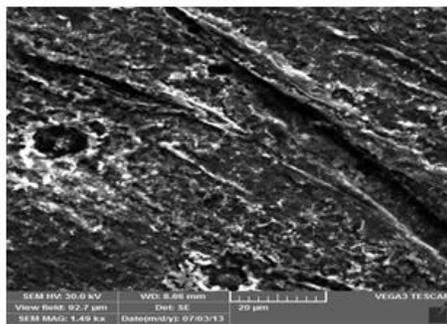


(b)

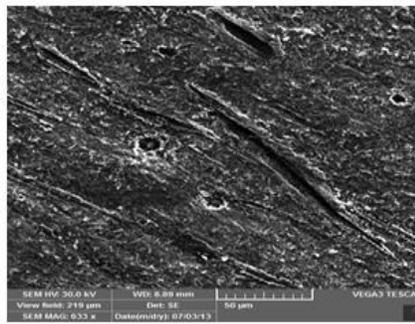
Fig.4: SEM analysis of

(a) Mild steel; Magnification X 20 (control)

(b) Mild steel; Magnification X 50 (control)



(c)



(d)

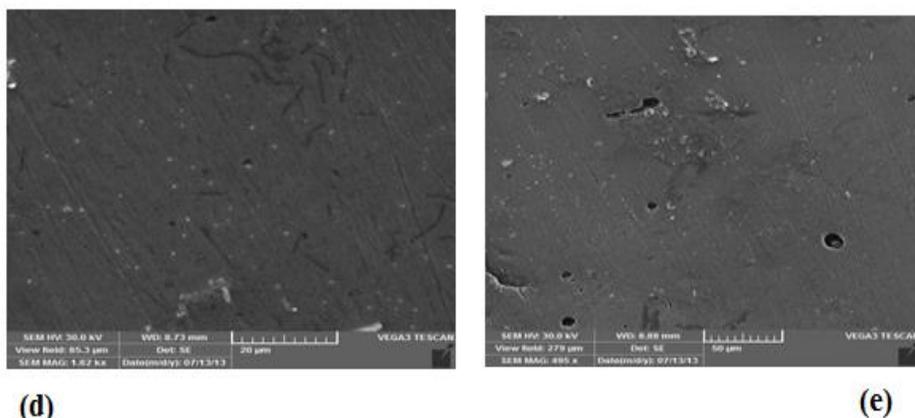


Fig.4: SEM analysis of

(c) Mild steel in an aqueous solution containing 60 ppm of Cl^- ; Magnification X 20

(d) Mild steel in an aqueous solution containing 60 ppm of Cl^- ; Magnification X 50

(e) Mild steel in an aqueous solution containing 60 ppm of Cl^- + 50 ppm of Cu-HUF;
Magnification X 20

(f) Mild steel in an aqueous solution containing 60 ppm of Cl^- + 50 ppm of Cu-HUF;
Magnification X 50

Energy dispersive analysis of X-rays (EDAX)

The EDAX survey spectra were used to determine the elements present on the mild steel surface before and after exposure to the inhibitor solution [24-26]. The objective of this section was to confirm the results obtained from chemical and electrochemical measurements that a protective surface film of inhibitor is formed on the mild steel surface. To achieve this, EDAX examinations of the mild steel surface were performed in the absence and presence of inhibitors system [24-26].

The EDAX spectrum of mild steel is shown in Fig 5 (a) and EDAX spectrum of mild steel immersed in an aqueous solution containing 60 ppm of Cl^- is shown in Fig.5(b). They show the characteristic peaks of some of the elements constituting the mild steel sample. The EDAX spectrum of mild steel immersed in an aqueous solution containing 60 ppm of Cl^- and 50 ppm of Cu-HUF is shown in Fig.5(c). It shows the additional line characteristic for the existence of Cu, the intensity of O signals is reduced and the intensity of Fe signal is increased. The appearance of Fe signal and this enhancement of

O signal are due to the presence of inhibitor. These data show that mild steel surface is covered with the N,O, C, Fe and Cu atoms. This layer is undoubtedly due to the inhibitor system. The Fe, Zn signal and this high contribution of O and C is not present on the mild steel surface exposed in an aqueous solution containing 60 ppm of Cl^- .

Fig.5(c) shows that the Fe peaks observed in the presence of inhibitor are considerably suppressed relative to these observed in an aqueous solution containing 60 ppm of Cl^- (blank solution). The suppression of the Fe peaks occurs because of the overlying inhibitor film. This observation indicates the existence of an adsorbed layer of inhibitor that protects steel against corrosion. These results suggest that O, N and C atoms of Cu-HUF has coordinated with Fe^{2+} , resulting in the formation of Fe^{2+} -HUF complex on the cathodic sites of mild steel surface and presence of Cu atoms are precipitated as $\text{Cu}(\text{OH})_2$ on the anodic sites of mild steel surface.

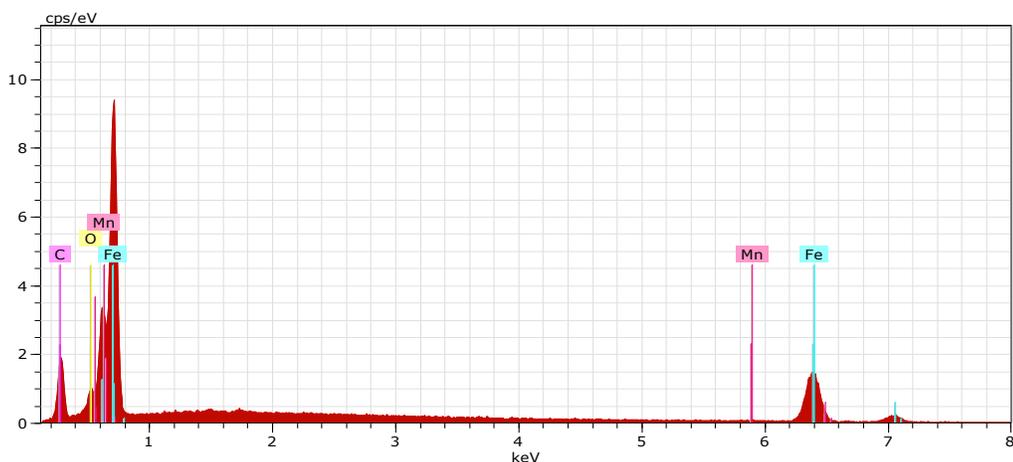


Fig.5 : EDAX spectrum of

(a) Mild steel specimen (control)

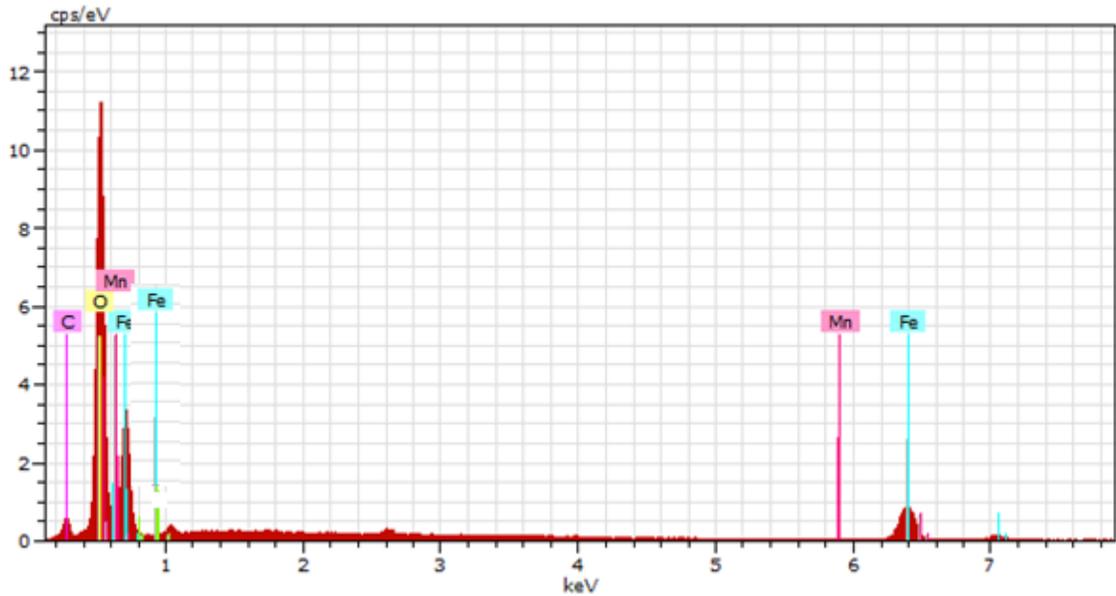


Fig.5 : EDAX spectrum of

(b) mild steel sample after immersion in an aqueous solution containing 60 ppm of Cl^-

(blank)

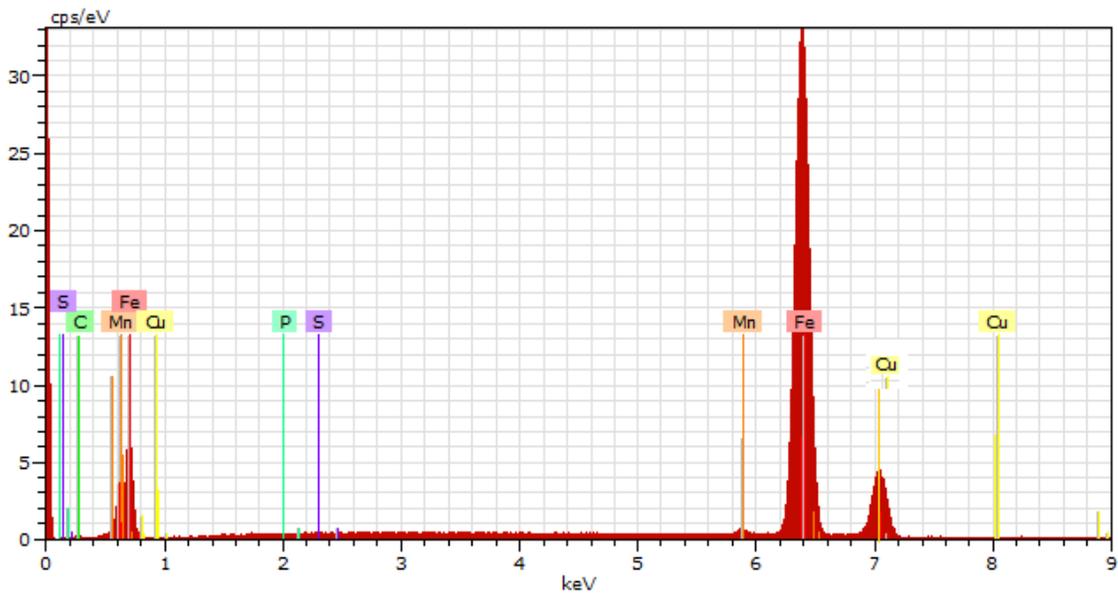


Fig.5 : EDAX spectrum of

(c) Mild steel sample after immersion in an aqueous solution containing 60 ppm of Cl^- + 50 ppm of Cu-HUF

Analysis of biocidal efficiency of the system

The results of bacterial enumeration count [25-28] of the aqueous solution containing 60 ppm of Cl^- and with Cu-HUF inhibitor are presented in Table 3. The mild steel specimens were immersed in various test solutions for a period of one day. After one day, the test solutions were tested for bacterial enumeration count. The aqueous solution containing 60ppm of Cl^- without inhibitor shows more bacterial count against the growth of pathogenic bacteria strains such as *E.Coli*, *Streptococcus*, *Pseudomonas* and *Entrobacter*. The aqueous solution containing 60 ppm of Cl^- with Cu-HUF inhibitor shows less bacterial count against the growth of pathogenic bacteria strains such as *E.Coli*, *Streptococcus*, *Pseudomonas* and *Entrobacter*. A good result was obtained when addition of Cu-HUF inhibitor to the corrosive media.

Systems	Colony Forming Unit (per ml)			
	<i>E.Coli</i>	<i>Streptococcus</i>	<i>Pseudomonas</i>	<i>Entrobacter</i>
60 ppm Cl^-	140×10^6	146×10^6	130×10^6	127×10^6
60 ppm Cl^- + 50 ppm Cu-HUF	50×10^6	59×10^6	62×10^6	60×10^6
Biocidal activity (%)	64.28	59.58	52.30	52.75

Table 3. Colonies forming unit (CFU) of mild steel in an aqueous solution containing 60 ppm of Cl^- in the absence and presence of Cu-HUF inhibitor obtained by bacterial enumeration count method.

Mechanism of corrosion inhibition

The results of the weight loss study show that the formulation consisting of 50 ppm of Cu-HUF has 80% IE in controlling corrosion of mild steel in an aqueous solution containing 60 ppm of Cl^- .

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+} -HUF complex and $\text{Cu}(\text{OH})_2$. In order to explain these facts that the following mechanism of corrosion inhibition is proposed [29-35].

- When the solution containing 60 ppm of Cl^- and 50 ppm of Cu-HUF is prepared. There is formulation of Cu^{2+} -HUF complex in solution.
- When mild steel is immersed in this solution, Cu^{2+} -HUF complex diffuses from the bulk solution towards mild steel surface.
- Cu^{2+} -HUF complex diffuses from the bulk solution to the surface of the mild steel and is converted into Fe^{2+} -HUF, which is more stable than Cu^{2+} -HUF [43].
- On the mild steel surface Cu^{2+} -HUF complex is converted in to Fe^{2+} -HUF on the cathodic sites. Cu^{2+} is released.



- The released Cu^{2+} combines with OH^- to form $\text{Cu}(\text{OH})_2$ on the anodic sites [29, 30].
$$\text{Cu}^{2+} + 2\text{OH}^- \longrightarrow \text{Cu}(\text{OH})_2 \downarrow$$
- Thus the protective film consists of Fe^{2+} - HUF complex and $\text{Cu}(\text{OH})_2$. This is confirmed by FTIR spectra.
- The EDAX analysis and SEM micrographs confirm the formation of protective layer on the mild steel surface.
- The formulation with 50 ppm of Cu-HUF has 80% corrosion inhibition efficiency and shows biocidal efficiency.

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