



**ADSORPTION, ELECTROCHEMICAL AND
SURFACE MORPHOLOGICAL STUDIES OF CARBON
STEEL CORROSION BY 2-(BENZYLIDENE-AMINO)-
BENZENETHIOL INHIBITOR IN SODIUM CHLORIDE
SOLUTION**

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Abstract

The corrosion protection effect of carbon steel is dipped in sodium chloride has been evaluated at room temperature by using conventional weight loss method. The corrosion rate and inhibition efficiency has been determined from weight loss method. The corrosion inhibition efficiency increases with increase in concentration of the 2-(benzylidene-amino)-benzenethiol inhibitor. The corrosion rate decreases when increase in concentration of the 2-(benzylidene-amino)-benzenethiol inhibitor. This is owing to at the higher concentration of inhibitor solution which prevents the active site of a carbon steel and a protective film is formed over the carbon steel surface. The rate of corrosion is decreased with increasing addition of organic compound, probably due to progressive adsorption of the 2-(benzylidene-amino)-benzenethiol on the mild steel surface. The maximum inhibition efficiency was found to be 86.79%. Electrochemical studies have been used to observe the formation of protective film over the carbon steel surface. It is also recognized by surface analysis technique like FTIR and scanning electron microscopy. Smoothness and roughness of carbon steel surface has been observed and

compared to blank and inhibitor system by SEM. The AFM microscopes confirm the roughness and smoothness of carbon steel surface.

Keywords: AFM, Corrosion, Organic Compound, FTIR, Scanning Electron Microscopy and Weight loss method.

Introduction

Corrosion is destruction of metal or alloy by chemical and electrochemical reaction with its environment. Carbon steel can often be used as a construction frame material, given that mild steel beams have incredibly high strength, just like any structural steel beam. It is cheap, suitable for different cutting and coating methods and has good weldability while providing well enough physical properties. Metal and non-metal corrosion occur which leads to environmental contact on the material surface. This relationship affects the systems and facilities of various materials. The presence of moisture and oxygen in the ambient air will initiate the rusting process on steel surfaces. Chemicals in soil and moisture decide the rate of damage to submerged structures and pipelines. Corrosion of CS and iron is a huge theoretical and practical problem, and it has sparked a lot of interest [1]. Corrosion has a wide range of implications, and the impact on the effective, stable, and productive operation of machinery or structures are often more severe than the loss of a mass of metal. Even if the amount of metal destroyed is minimal, failures of various kinds and the need for costly replacements can occur [2]. Even if the amount of metal destroyed is small, letdowns of various types will occur, necessitating costly substitutions. Corrosion is a chemical reaction with the atmosphere that causes metal to corrode. It's a never-ending, expensive dilemma that's always difficult to solve. Inhibitors are used to reduce corrosion. Corrosion is the natural conversion of a refined metal to a more chemically stable form, such as oxide, hydroxide, or sulphide. It is the gradual decomposition of materials (typically metals) as a result of chemical and/or electrochemical reactions with its environments [3]. Inhibitors are chemical substances which helpful in the engineering process to resistor metal dissolution, especially in the acid, neutral and base environment. The effective corrosion inhibitors applied in engineering industry are the hydrocarbons that possess active group, which is considered as the active center for the adsorption process. Scientist explored the effects of hydrocarbons on the metal destruction of carbon steel, aluminum, alloys, mild steel and complexes in acidic, base and neutral conditions [4-5]. The weight loss method and

electrochemical impedance spectroscopy were used to assess the corrosion rate and inhibition efficiency. To explore the surface film by FTIR spectra and Scanning Electron Microscopy. The protective layer is formed on the CS surface was characterized with the support of surface characterization systems like Fourier transform infrared spectroscopy. The surface morphology has been evaluated by scanning electron microscopy. From SEM analysis we predict the smoothness of CS surface when compare to blank system and with corrosion inhibitor system [6-8]. Moreover, the roughness and smoothness of the CS surface have been characterized by AFM technique.

Materials and Methods

Corrosion inhibition of carbon steel in sodium chloride solution by (2-(benzylidene-amino)-benzenethiol) has been investigated.

Carbon steel specimens Preparation

Carbon - 2.0 %, Sulphur - 0.026 %, Phosphorus - 0.06 %, Manganese - 0.4 % and the balance iron were polished to mirrors finish and degreased with acetone and used for weight loss method.

Preparation of stock solutions

Distilled water was used wherever necessary in the preparation of solutions. The required concentration of the organic inhibitor (2-(benzylidene-amino)-benzenethiol) stock solution was prepared by dissolving 2-(benzylidene-amino)-benzenethiol in minimum amount of ethanol and making up to the desired volume with distilled water. Then the required volume from the inhibitor stock solution was added to the distilled water solution to obtain the desired concentration [9].

Weight loss method

Weight loss measurements were done according to the described method [10]. Weight loss measurements were performed for one day by immersing the mild steel specimens in sea water without and with different concentration (50 ppm, 100 ppm, 150 ppm, 200

ppm, 250 ppm) of organic inhibitor.

After elapsed time, the specimen was taken out, washed, dried and weighed accurately

The inhibition efficiency (IE %) was determined by the following equation

$$IE (\%) = \frac{W_o - W_i}{W_o} \times 100$$

Where W_i and W_o are the weight loss values in g in presence and absence of 2-(benzylidene-amino)-benzenethiol inhibitor.

Electrochemical Techniques

In the present work inhibitive action of carbon steel immersed in various test solutions were measured by Polarization study and AC impedance spectra. Electrochemical measurements were performed in a CHI- electrochemical work station with impedance model 660A.

Polarization study

Polarization studies were carried out in a three electrode cell assembly. A SCE was used as the reference electrode. Platinum was the counter electrode. Carbon steel was the working electrode. From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a , and cathodic = b_c , and LPR (linear polarisation resistance) values were measured [11].

AC impedance measurements

An electrochemical workstation impedance analyzer CHI- electrochemical work station with impedance model 660A [12]. The cell setup was identical to that used to test polarization. The device was given a time interval of 5 to 10 minutes to reach a steady-state open circuit potential. An AC potential of 10 mV was then superimposed over this steady-state potential. For different frequencies, the AC frequency was varied from 100 kHz to 100 MHz, and the actual (Z) and imaginary (z'') sections of the cell impedance were calculated in ohms. The C_{dl} (double layer capacitance) and R_t (charge transfer resistance) values were determined. The following relationship was used to measure C_{dl} values.

$$C_{dl} = \frac{1}{2 \times 3.14 \times R_t \times f_{max}}$$

Surface Examination Techniques

The mild steel specimens were immersed in blank, as well as aqueous extract of *Datura metel* plant leaves inhibitor solutions, for a period of 24 hours. After 24 hours, the specimens were taken out and dried. The nature of the film formed on the surface of the mild steel specimens was analyzed by various analysis techniques.

Surface analysis by FTIR spectra

FTIR spectra were recorded in a Perkin–Elmer 1600 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 [13-14]. FTIR spectrum of the powder (KBr pellet) was recorded using Perkin–Elmer 1600 FTIR spectrophotometer with a resolving power of 4 cm^{-1} .

Scanning Electron Microscopic studies (SEM)

The carbon steel specimen immersed in blank and in the inhibitor solution for a period of 24 hours 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 carbon steel were examined using CAREL ZEISS EVO 18, Hitachi computer controlled scanning electron microscope [15].

Atomic Force Microscopy characterization (AFM)

The carbon steel specimen immersed in blank and in the inhibitor solution for a period of 24 hours was removed, rinsed with double distilled water, dried and subjected to the surface examination [16]. The surface morphology measurements of the carbon steel surface were carried out by atomic force microscopy (AFM) using Agilent technologies 5500 series mode.

Results and Discussion

The effect of concentration of corrosion inhibitor on the carbon steel in sodium chloride solution was shown in table:1. From the values, the concentration of the

inhibitor is increases whereas inhibition efficiency increases. The increase in inhibition efficiently is due to the blocking effect on the surface of the metal by adsorption. It is due to presence of nitrogen atom and double bond in corrosion inhibitor. In addition with the delocalized π -electrons also present in the compound which enhances the inhibition efficiencies. This makes it formation of surface film on the carbon steel and also many reviews are reported good agreement with these results. The corrosion rates (CR) and inhibition efficiencies (IE) of carbon steels are immersed in sea water in the absence and presence of 2-(benzylidene-amino)-benzenethiol inhibitor obtained by weight loss method. It is observed that 250 ppm of 2-(benzylidene-amino)-benzenethiol offers 86.79% of inhibition efficiency. As the concentration of 2-(benzylidene-amino)-benzenethiol increases, the inhibition efficiency increases and corrosion rate decreases. This is due to an increase of surface coverage at higher concentration of the inhibitor which retards dissolution of zinc metal. The electron donating properties of nitrogen and sulphur atoms can be attributed for higher inhibition efficiencies. This surveillance is in good agreement with the results reported by many researchers [17-22].

Table V.1. Corrosion rates (CR) and the inhibition efficiency (IE %) carbon steel is immersed in sodium chloride in absence and presence of inhibitor systems at various concentration obtained by weight loss method.

- Inhibitor System: 2-(benzylidene-amino)-benzenethiol (ppm)
- Immersion period: 24 hours

Blank	2-(Benzylidene -Amino)- Benzenethiol (ppm)	CR (mdd)	IE (%)
Sodium chloride	-	38.58	----
	50	28.00	23.25

	100	22.42	33.25
	150	17.58	48.10
	200	13.52	67.45
	250	8.90	86.79

Analysis of results of potentiodynamic polarization study

The potentiodynamic polarization studies confirm that corrosion inhibitor formed protective film on the carbon steel surface. Its values of Corrosion potential (E_{corr}), Corrosion current density (I_{corr}) and anodic and cathodic Tafel plots (b_a , b_c) undergo analysis from Tafel plots. The linear Tafel segments of anodic and cathodic curves were extrapolate to corrosion potential to obtain corrosion current densities (I_{corr}). Polarization study has been used to confirm the formation of protective film on the mild steel surface during corrosion inhibition process. If a protective film is formed on the carbon steel surface, the linear polarization resistance values (LPR) increases and the corrosion current value (I_{corr}) decreases [23-25].

The potentiodynamic polarization curves of carbon steel immersed in sea water and also inhibition efficiencies (IE) in the presence and absence of inhibitor are shown in Figure V.1 (a, b). The corrosion parameters are given in Table V.2. When carbon steel was immersed in sea water the corrosion potential was -0.699 mV vs SCE. When 250 ppm of 2-(benzylidene-amino)-benzenethiol was added to the above system, the corrosion potential shifted to the noble side -0.696 mV vs SCE. This indicates that the protective film is formed on the anodic and cathodic sites of the mild steel surface. This film controls the anodic and cathodic reactions of carbon steel dissolution by forming Fe^{2+} - inhibitor complex on the carbon steel surface [26].

Further, the LPR value increases from 1139 ohm cm² to 2010 ohm cm², the corrosion current decreases from 2.905×10^{-5} A/cm² to 1.8667×10^{-5} A/cm². Thus polarization study confirms the formation of a protective film on the carbon steel surface.

Table - 2 Corrosion parameters of carbon steel immersed in sodium chloride solution and inhibition efficiencies (IE) in the absence and presence of inhibitor system obtained by potentiodynamic polarization method.

Systems	E _{corr} vs SCE (mV)	I _{corr} (A/cm ²)	b _a (mV/dec)	b _c (mV/dec)	LPR (ohm cm ²)
Blank (Sodium chloride)	-0.699	2.905×10^{-5}	0.102	0.252	1139
Sodium chloride + 250 ppm of inhibitor	-0.696	1.8667×10^{-5}	0.128	0.207	2010

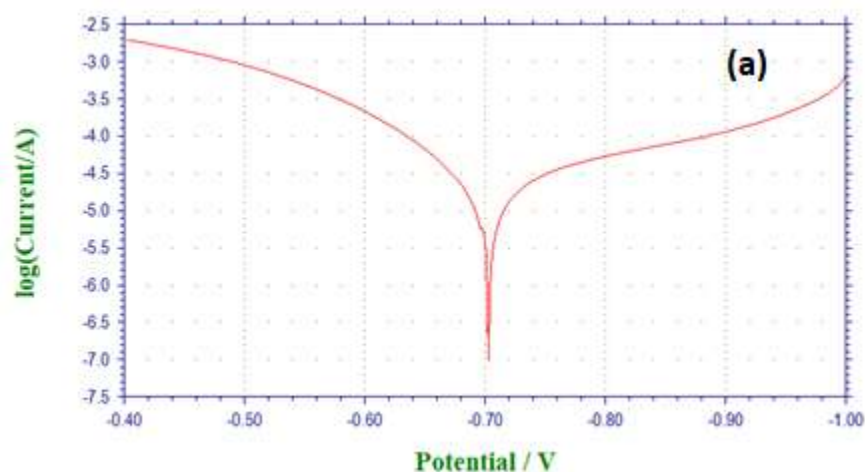
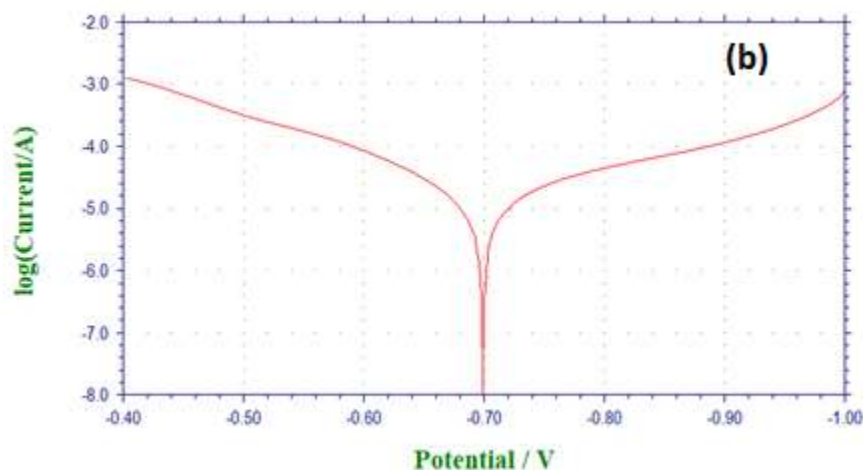


Figure.1: Polarization curves of carbon steel immersed in test solutions
(a) Sodium chloride (blank)



(b) Sodium chloride + 250 ppm of 2-(benzylidene-amino)-benzenethiol

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 carbon steel surface [27-32]. If a protective film is formed on the carbon steel surface, charge transfer resistance (R_t) increases; double layer capacitance value (C_{dl}) decreases and the impedance $\log(z/\text{ohm})$ value increases. The AC impedance spectra of carbon steel immersed in sodium chloride solution in the absence and presence of inhibitor (2-(benzylidene-amino)-benzenethiol) are shown in Figures 2 (a, b) (Nyquist plots), Figures 3 (a, b) (Bode plots) and Figures 4 (a, b) (Phase angle). The values are given in table.3.

It is observed that when the inhibitor (250 ppm of 2-(benzylidene-amino)-benzenethiol) is added to the system, the charge transfer resistance (R_t) increases from $31.98 \Omega \text{ cm}^2$ to $40.99 \Omega \text{ cm}^2$ and the C_{dl} value decreases from $67.099 \times 10^{-6} \text{ F cm}^{-2}$ to $52.5869 \times 10^{-6} \text{ F cm}^{-2}$. The impedance value [$\log(z/\text{ohm})$] increases from 0.570 to 0.648. Moreover, the phase angle value is also increases from 29.5° to 32.5° . These results lead to the conclusion that a protective film is formed on the carbon steel surface [33].

Table 3: Corrosion parameters of carbon steel immersed in sodium chloride solution in the absence and presence of inhibitor system obtained from AC impedance spectra.

Systems	Nyquist plot		Bode plot
	R_t $\Omega \text{ cm}^2$	C_{dl} F cm^{-2}	Impedance $\text{Lg} (Z \text{ ohm}^{-1})$
Blank (sodium chloride solution)	31.98	67.099×10^{-6}	0.570
Sodium chloride + 250 ppm of 2-(benzylidene-amino)-benzenethiol	40.99	52.5869×10^{-6}	0.648

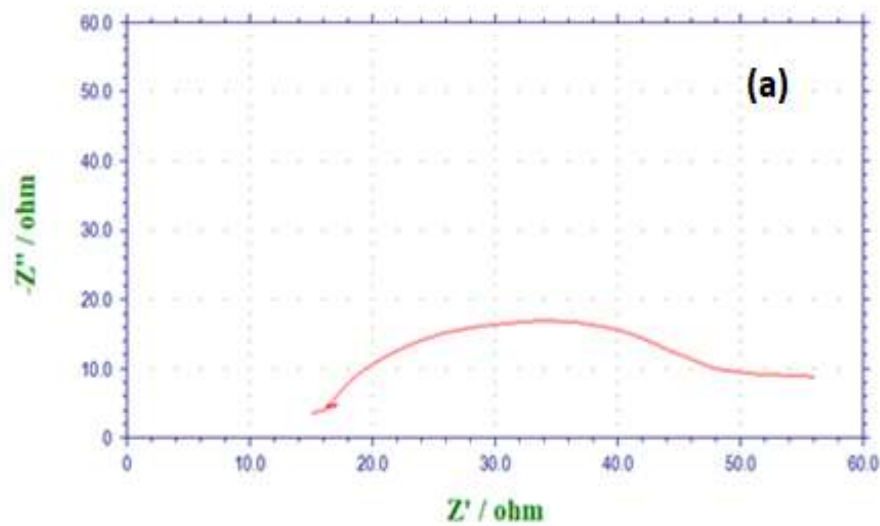


Figure. 2a: AC impedance spectra of carbon steel immersed in sodium chloride solution (blank)

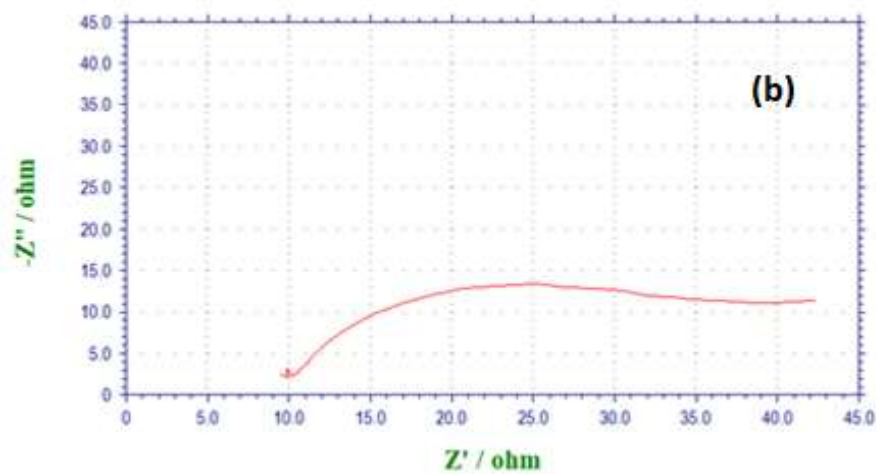


Figure 2b: AC impedance spectra of carbon steel immersed in sodium chloride (blank) + 250 ppm of 2-(benzylidene-amino)- benzenethiol

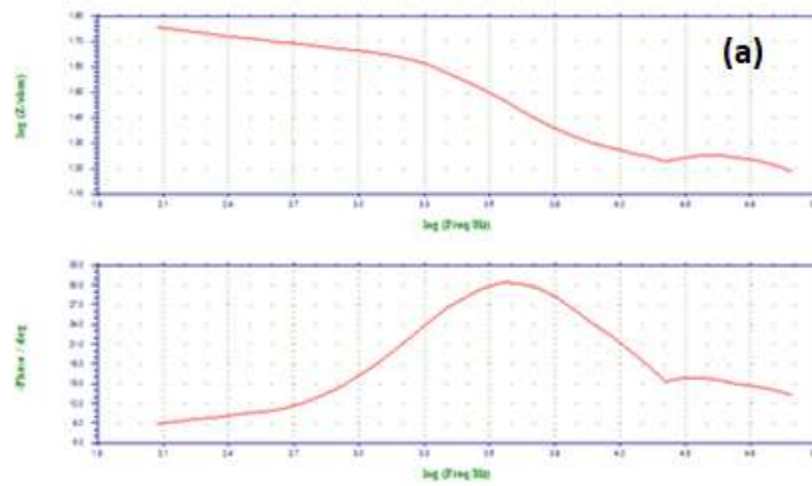


Figure 3a: Bode plot of AC impedance spectra of carbon steel immersed in sodium chloride solution (blank)

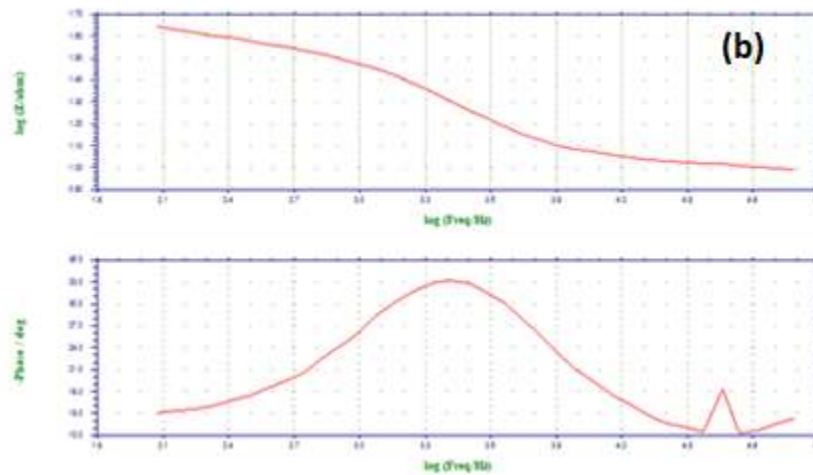


Figure. 3b: Bode plot of AC impedance spectra of carbon steel immersed in sodium chloride solution and 250 ppm of 2-(benzylidene-amino)- benzenethiol.

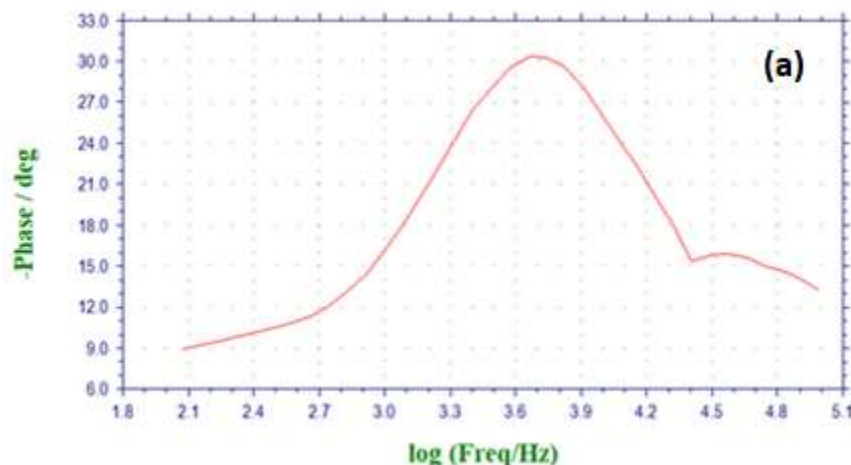


Figure. 4a: The phase angle of AC impedance spectra of carbon steel immersed in sodium chloride solution (blank)

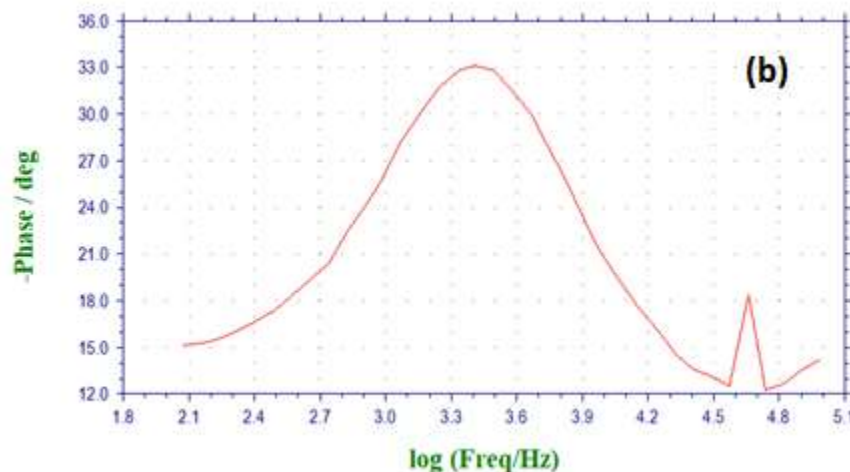


Figure. 4b: The phase angle of AC impedance spectra of carbon steel immersed in sodium chloride solution and 250 ppm of 2-(benzylidene-amino)-benzenethiol

Analysis of FTIR spectra

FTIR is used to analyze the protective film formed over on the carbon steel surface [34-36]. The structure of 2-(benzylidene-amino)- benzenethiol is figure 5 and the values are in the table 4. The FTIR spectrum (KBr) of pure 2-(benzylidene-amino) - benzenethiol is shown in Figure 6a. The C-N stretching frequency appears at 1688.92 cm^{-1} . The peak

due to C=N appears at 1593.76 cm^{-1} . The peak appears at 3365.68 cm^{-1} due to CH stretching frequency. The SH stretching frequency appears at 1013.46 cm^{-1} . The C=S stretching frequency appears at 1157.91 cm^{-1} . The peak due to aromatic C=C appears at 1285.57 cm^{-1} .

The FTIR spectrum (KBr) of the film formed over the carbon steel surface after immersion in sodium chloride solution and 250 ppm of 2-(benzylidene-amino)-benzenethiol is shown in Figure 6b. The C-N stretching has shifted from 1688.92 cm^{-1} to 1569.88 cm^{-1} . The C=N stretching frequency has shifted from 1593.76 cm^{-1} to 1596.88 cm^{-1} . The CH stretching frequency has shifted from 3365.68 cm^{-1} to 2977.34 cm^{-1} . The SH stretching frequency has shifted from 1013.46 cm^{-1} to 1019.74 cm^{-1} . The C=S stretching frequency has shifted from 1157.91 cm^{-1} to 1158.95 cm^{-1} . The peak due to aromatic C=C has shifted from 1285.57 cm^{-1} to 1298.03 cm^{-1} . A new peak appears in the region of 619.82 cm^{-1} due to a zinc complex is formed on the carbon steel surface. The sulphur and nitrogen atoms of 2-(benzylidene-amino)-benzenethiol have coordinated with Fe^{2+} and form Fe^{2+} – inhibitor complex is formed on the surface of carbon steel. Thus the FTIR spectral study leads to the conclusion that the protective film consists of Fe^{2+} – inhibitor complex [37-39].

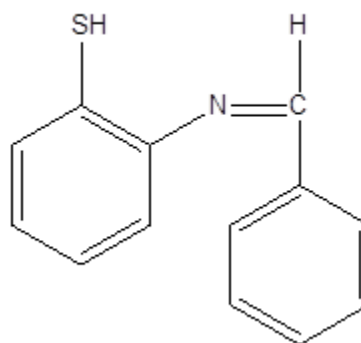


Figure 5. Structure of 2-(benzylidene-amino)- benzenethiol

Table 4. IR values of Pure 2-(benzylidene-amino)- benzenethiol and film formed over the carbon steel surface is obtained from IR Spectra.

Functional Group	Pure 2-(benzylidene-amino)- benzenethiol compound (Peak Appears At) (cm⁻¹)	The Protective film consists of Fe²⁺ -2-(benzylidene-amino)- benzenethiol (cm⁻¹)
C-N	1688.92	1569.88
CH	3365.68	2977.34

C=N	1593.76	1569.88
SH	1013.46	1019.74
C=S	1157.91	1158.95
C=C	1285.57	1298.03
M-O	-	619.82

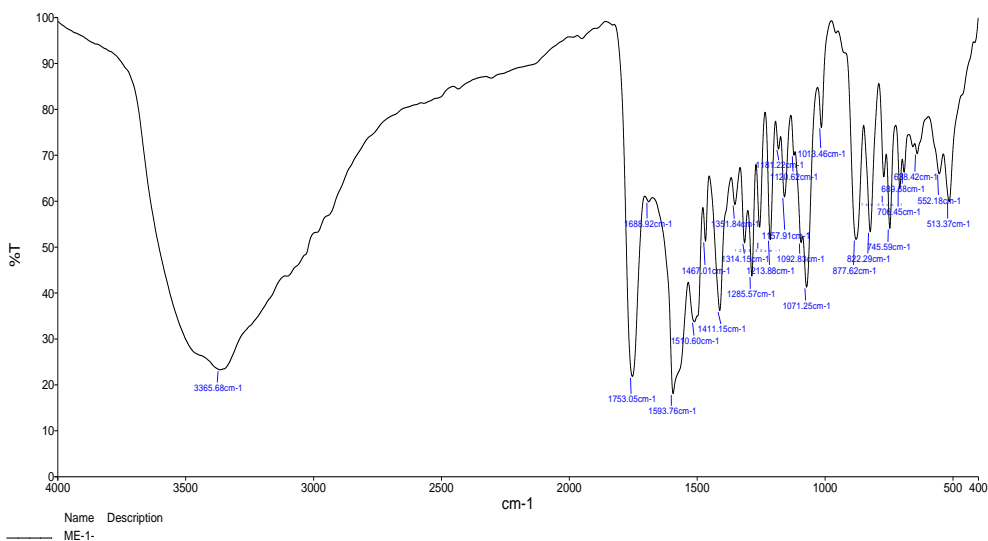


Figure. 6a. FTIR spectrum of pure 2-(benzylidene-amino)- benzenethiol

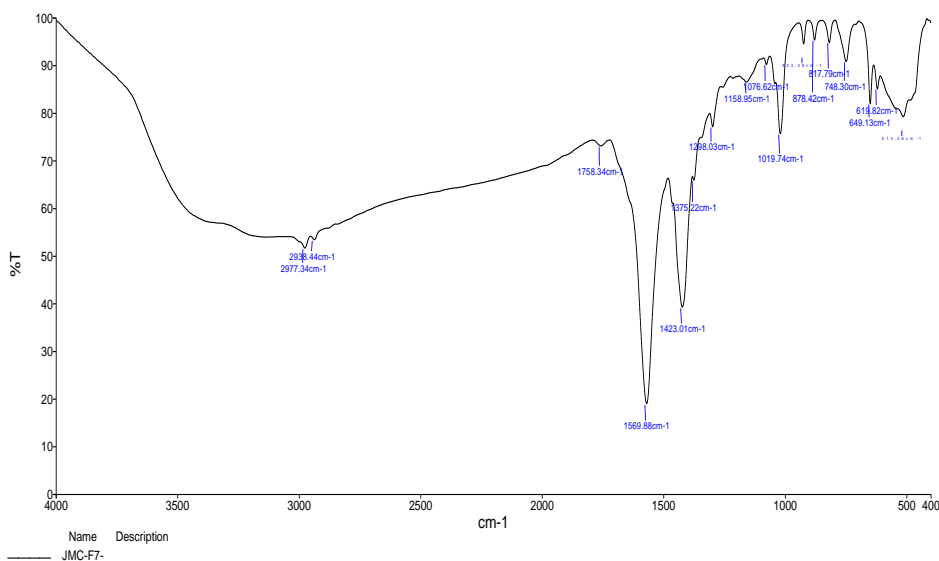


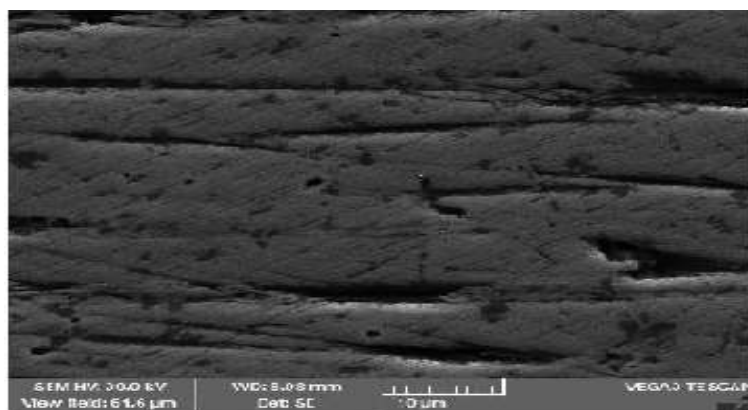
Figure. 6b. FTIR spectrum of film formed on the carbon steel surface after immersion in sodium chloride solution containing 250 ppm of 2-(benzylidene-amino)- benzenethiol

SEM Analysis of carbon steel Surface

SEM study gives a surface morphology result to understand the nature of the surface film in presence and absence of corrosion inhibitors and extent of corrosion of carbon steel. The SEM images of the carbon steel surface are examined. To understand the nature of the surface film in the absence and presence of inhibitors and extent of corrosion of carbon steel, the SEM micrographs of the surface are examined [40-46].

The SEM images of carbon steel specimen immersed in sea water for one day in the presence and absence of inhibitor system are shown in Figures. 7 (a, b and c) respectively. The SEM micrographs of polished carbon steel surface (control) in Figure 7 (a) shows the smooth surface of the carbon steel. This shows the absence of any corrosion products or inhibitor complex formed on the carbon steel surface.

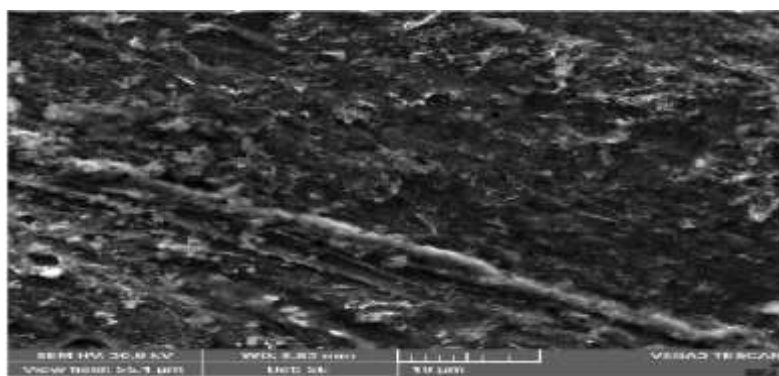
The SEM micrograph of carbon steel surface immersed in sodium chloride (Figure. 7b) show the roughness of the carbon steel surface which indicates the highly corroded area in sodium chloride solution. However in Figure. 7c indicate that in the presence of inhibitor (250 ppm of 2-(benzylidene-amino)- benzenethiol) the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The carbon steel surface almost free from corrosion due to the formation of insoluble complex on the surface of the carbon steel. In the presence of 2-(benzylidene-amino) - benzenethiol, the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of carbon steel [47].



(a)

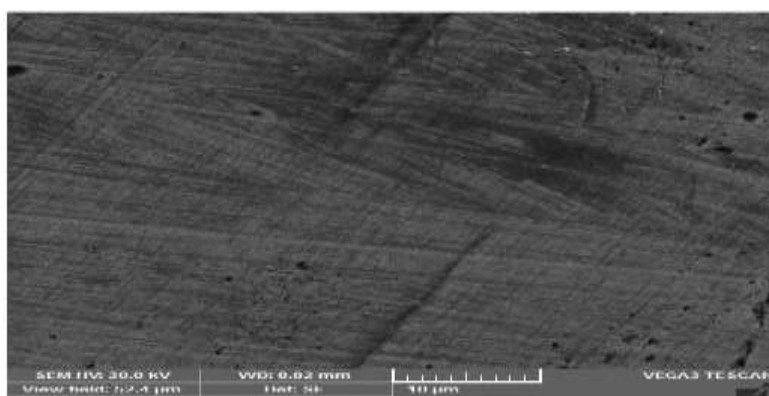
Figure. 7: SEM analysis of

(a) Carbon steel; Magnification X 10 μm (control)



(b)

(b) Carbon steel immersed in sodium chloride⁻; Magnification X 10 μm (blank)



(c)

© Carbon steel immersed in sodium chloride⁻ and 250 ppm of 2-(benzylidene-amino)-benzenethiol; Magnification X 10 μ m

Analysis of the results of Atomic force microscopy

Atomic force microscopy is a powerful technique for the gathering of roughness statistics from a variety of surfaces. Atomic force microscopy was studied to find out the corrosion inhibition is due to the formation of a film on the carbon steel surface through adsorption. Surface roughness analysis for carbon steel immersed in the sodium chloride solution, and presence the inhibitor compounds, and surface roughness analysis for polished carbon steel surface (reference) was studied by using atomic force microscopy [48-52].

Table V.5 shows the values obtained, when using the AFM technique, such as the values of S_p , S_q , S_a , and S_y for the polished mild steel surface (reference) are (86.75, 21.36, 18.20, 161.21) nm, which show more homogeneous surface compared with blank (1325, 278.53, 209.24 and 2289) nm respectively, this will shows that blank has a larger surface roughness than the polished carbon steel surface. Furthermore, this shows that the unprotected carbon steel surface is rougher due to the corrosion of metal in aqueous solution of sodium chloride (Figure: 8a).

From the table .5. It is clear that, when the presence corrosion inhibitor contain sodium chloride solution leads to decrease the average roughness (S_a) value from 18.20 nm in inhibitor to 209.24 nm with blank. The value shows that large difference in these parameters confirms that the surface appears smoother (Figure: 8c), due to the formation of a compact protective film. The corrosion product was not allowed to deposit on the carbon steel surface. Further, these results are confirmed by the clearly visible differences among the optical cross section analysis. The carbon steel surface was covered with a protective film, thereby, forming a barrier against attack by aggressive ions from the corrosive environment. With the addition of inhibitor, the average roughness was reduced to 588 nm, which suggested the film formation of the inhibitor over the carbon steel surface [53-56].

Table 5 AFM data for carbon steel immersed in the presence and absence of inhibitor systems

Samples	Value in nm			
	S_p	S_q	S_a	S_y
Polished carbon steel	86.75	21.36	18.20	161.21
Polished carbon steel + sodium chloride	1325	278.53	209.24	2289
Polished carbon steel + sodium chloride solution + 250 ppm of 2-(benzylidene-amino)- benzenethiol	241.30	61.74	47.21	544.70

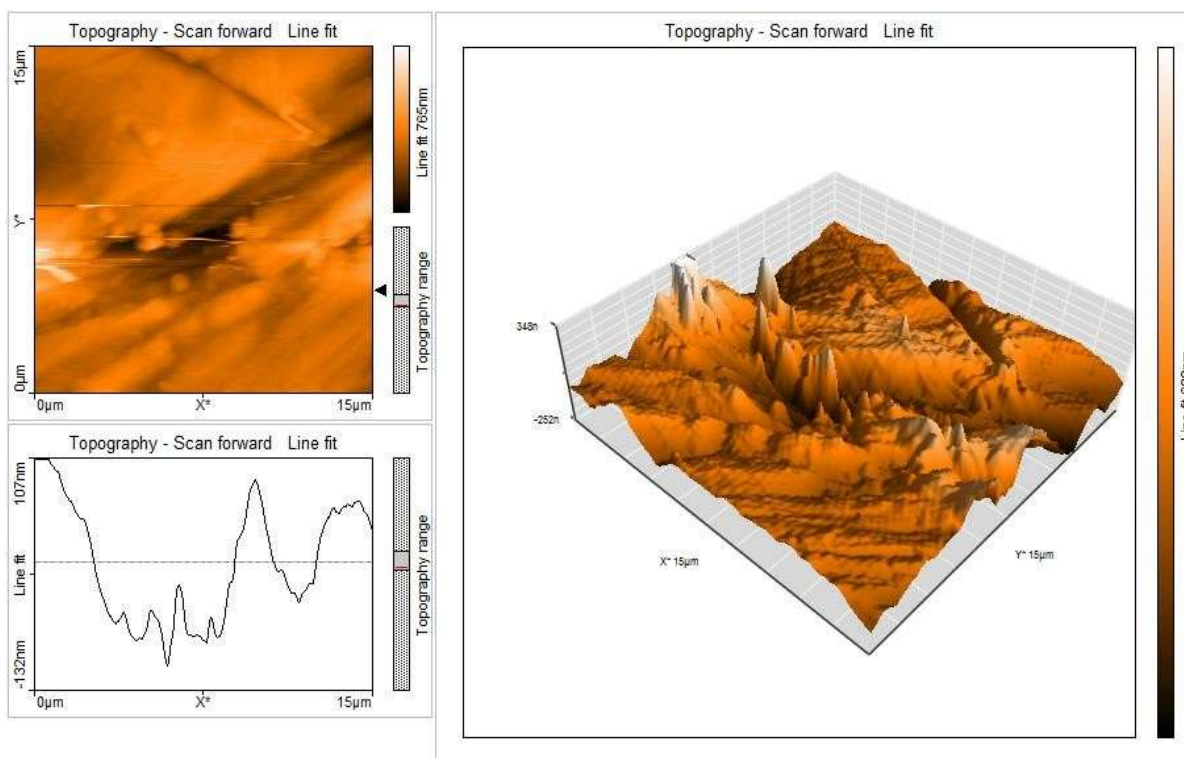


Figure 8a. AFM image of polished carbon steel specimen (control)

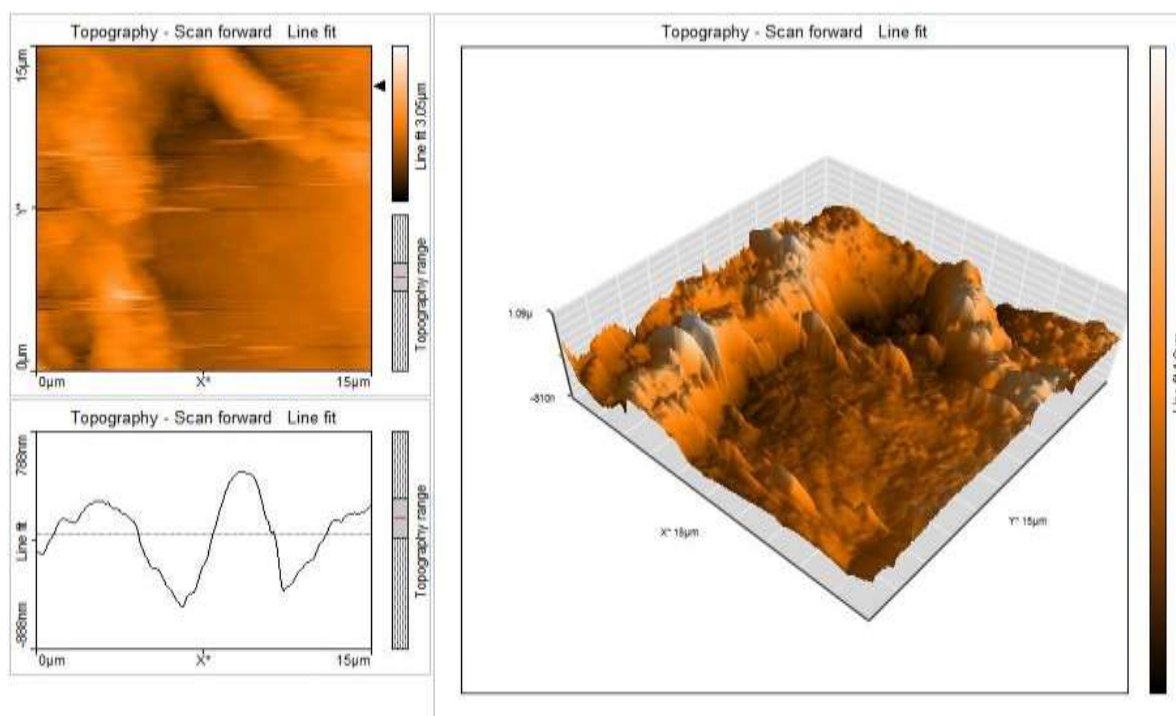


Figure 8b AFM image of carbon steel specimen immersed in sodium chloride solution

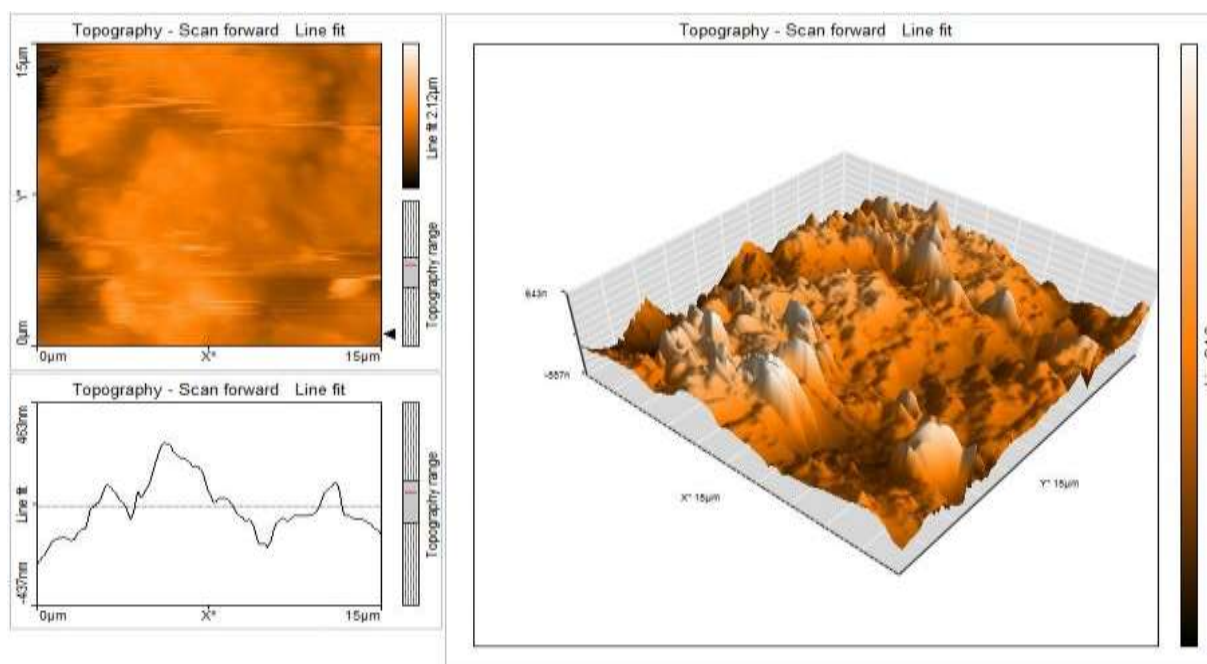


Figure.8c. AFM image of carbon steel specimen immersed in sodium chloride solution with inhibitor (2-(benzylidene amino)- benzenethiol)

Conclusion

In this present research work, the compound 2-(benzylidene-amino)-benzenethiol has been used as a corrosion inhibitor to prevent the corrosion of carbon steel immersed in sodium chloride solution. The mechanistic aspects of corrosion inhibition are proposed based on the results obtained from the weight – loss method, the polarization study, AC impedance measurements and surface examination techniques like FTIR spectroscopy and Scanning Electron Microscopy. The rate of corrosion is decreased with increasing addition of organic compound, probably due to progressive adsorption of the 2-(benzylidene-amino)-benzenethiol on the carbon steel surface. The maximum inhibition efficiency was found to be 86.79%. The inhibitor act as mixed type. FTIR spectra reveal that the protective film consists of Fe^{2+} -2-(benzylidene-amino)-benzenethiol complex. SEM micrographs show the smoothness of carbon steel surface like polished carbon steel. The AFM microscopes confirm the roughness and smoothness of carbon steel surface.

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