Corrosion Resistance, Electrochemical and Surface Morphology Studies of Mild Steel in a Sulfuric Acid Medium by using Dibutyl Sulphide*

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Abstract
DBS corrosion resistance effect on MS immersed in a 0.25 mol/L sulphuric acid (H_2SO_4) solution has been evaluated at room temperature, using WL method, from which CR and IE(%) were obtained. The corrosion IE% increased with higher inhibitor concentrations. The CR decreased with higher DBS concentrations, since it blocked the MS active site, forming a protective film on its surface, which was confirmed by electrochemical studies and surface analysis techniques, such as FTIR and SEM. EDAX was used to analyze the elements present on the MS surface, after its immersion in 0.25 mol/L H_2SO_4, without and with DBD. The MS surface smoothness and roughness (polished and corroded) and the inhibitor have been evaluated by SEM and AFM.

Keywords: AFM, corrosion, DBS inhibitor, H_2SO_4, MS, SEM and WL.

Introduction

MS is used in making alloys for structural and industrial applications, and has become essential since the industrial revolution. New high strength alloys are usually more susceptible to a certain type of corrosive attack. Generally, metallic materials that use acids undergo corrosion, which is inevitable. The acids used as media in MS corrosion studies are important, due to their industrial applications, such as pickling, industrial cleaning, descaling, oil-well recovery and petrochemical processes [1-3]. The refining of crude oil has been carried out in a variety of aggressive conditions which cause the corrosion of equipments by a strong acid attack on its surface. Corrosion is a natural, gradual and costly destruction process of materials (usually metals), which converts a refined metal to a more chemically-stable form, such as its oxide, hydroxide, or sulfide, by chemical and/or electrochemical reactions with its environment [5]. Considerable

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[*] The abbreviations list is in pages 12-13.

progress towards the understanding of corrosion has been made. The consequences of corrosion are many and varied, and its effects on the safe, reliable and efficient operation of equipment or structures are often more serious than the simple loss of metal mass. Failures of various kinds and the need for expensive replacements may occur, even though the amount of metal destroyed is quite small [4]. It is a constant, continuous and costly problem, often difficult to eliminate completely. Therefore, it is required to prevent it, or at least control it. Corrosion is minimized by using inhibitors. Inhibitors are used in the industrial process, in order to control metal dissolution, especially in acidic, neutral and basic environments. Most of the efficient and well-known inhibitors used in industry are organic compounds containing hetero atoms, such as O, N, S and multiple bonds, which possess at least one functional group that is considered as the active center for the adsorption process. Several researchers studied the inhibition action of various organic compounds on Al, alloys, MS, CS and composites corrosion, in acidic, alkaline and neutral media [6, 7].

The main aim of the present study was to use DBS to inhibit the corrosion of MS immersed in a 0.25 mol/L H_2SO_4 solution. DBS, in terms of IE% against MS CR, has been evaluated by the WL method. The corrosion inhibition mechanism has been examined by electrochemical studies, such as ACIS and PDP studies. MS surface morphology has been characterized by FTIR, SEM, EDAX and AFM techniques.

Materials and methods

MS specimens with the composition of 0.026% S, 0.068% P, 0.36 % Mn, 0.13% C, 0.23% Zn and the remainder Fe, and the dimensions of 1.0 × 4.0 × 0.2 cm, were polished to mirror finish, degreased with acetone, and used for the WL method.

Preparation of stock solutions

The 0.25 mol/L H_2SO_4 solution was prepared by the dilution of an H_2SO_4 analytical grade with double distilled water, to the required concentration. The DBS stock solution desired concentration was prepared by dissolving it in a minimum amount of ethanol, making it up to the required volume with double distilled water and, then, adding it to the H_2SO_4 solution.

WL method

WL measurements were done according to the described method [8-10], for 2 h, by immersing the MS specimens in 0.25 mol/L H_2SO_4, without DBS and with it, at different concentrations (5, 10, 30, 50 and 100 mM). After the elapsed time, the specimens were taken out, washed, dried and accurately weighed. MS corrosion IE(%) by DBS was determined through the following equation:

$$\mathrm{IE}(\%) = \frac{W_O - W_i}{W_O} \times 100 \qquad (1)$$

where W_i and W_O are the WL values in g, with and without DBS, respectively.

CR determination

The weighed triplicate MS specimens were suspended by glass hooks in 100 mL of 0.25 mol/L H_2SO_4, without DBS and with it, in various concentrations, for 2 h. After that period, the specimens were taken out, washed in running water, dried and weighed. From the change in the specimen weights, CR values were calculated using the following relationship:

$$CR = \frac{87.6 \times WL\ (mg)}{\text{Surface area of the specimen } (dm^2) \times \text{period of immersion } (H) \times \text{density}}\ \text{myy} \qquad (2)$$

Corrosion IE% was then calculated using the equation:

$$IE\% = 100\ [1-(W_2/W_1)]\% \qquad (3)$$

where W_1 and W_2 are the CR values without and with inhibitor, respectively.

Electrochemical techniques

PDP study

PDP studies were carried out in a Princeton Applied Research vs. STAT MC electrochemical work station impedance analyzer model. A three electrode cell assembly was used. The WE was MS, with one constant 1cm^2 area exposed face, and the rest was shielded with red lacquer. Saturated calomel and a rectangular Pt foil were used as RE and CE, respectively. The CE area was much larger than that of the WE, which exerted a uniform potential field on the former. The WE and CE were immersed in 0.25 mol/L H_2SO_4, with and without DBS [11]. The RE was connected with the test solution, through a salt bridge.

EVs log I plots were recorded. The parameters, such as E_{corr}, I_{corr}, β_a and β_c, were determined from E vs. log I plots.

AC impedance measurements

AC impedance studies were carried out in an electrochemical work station impedance analyzer model Princeton Applied Research versa STATMC. The cell setup was the same as that used for PDP measurements.

A time interval of 5 to 10 min was given for the system to attain a steady state OCP. Then, over this steady state potential, an AC potential of 10 mV was superimposed. The AC frequency was varied from 100 KHz to 100 MHz.

The real part (z’) and imaginary part (z”) of the cell impedance were measured in ohms, for various frequencies.

The R_{ct} values were calculated. C_{dl} values were calculated using the following relationship [12]:

$$C_{dl} = \frac{1}{2 \times 3.14 \times R_t \times f_{max}} \qquad (4)$$

Surface examination techniques

The MS specimens were immersed in the solutions without and with DBS, for during a period of 2 h. After that, they were taken out and dried, and the nature of the film formed on their surface was analyzed by various analysis techniques.

Surface analysis by FTIR spectra

After an immersion period of 2 h in the solutions without and with DBS, the MS specimens were removed, and dried. Their surface films were carefully scratched, thoroughly mixed with KBr pellets, so as to make them uniform, and their FTIR spectra were recorded in a Perkin–Elmer 1600 spectrophotometer, with a resolving power of 4 cm^{-1} [13].

SEM

After an immersion period of 2 h in the solutions without and with DBS, the MS specimens were removed, rinsed with double distilled water, dried and observed in a CAREL ZEISS EVO 18, Hitachi computer controlled SEM, to assess their surface morphology [14].

EDAX

After an immersion period of 2 h in the solutions without and with DBS, the MS specimens were removed, rinsed with double distilled water, dried and observed in a Bruker computer controlled EDAX (Brucker Nano, GMBH, Germany), to assess the elements present on their surface [15].

AFM characterization

After an immersion period of 2 h in the solutions without and with DBS, the MS specimens were removed, rinsed with double distilled water and dried [16].
The MS surface morphology measurements were carried out by AFM, using an Agilent technology 5500 series model.

Results and discussion

The IE% and CR values (Table 1) of MS immersed in 0.25 mol/L H_2SO_4, without DBS inhibitor and with it, in different concentrations, were obtained by the WL method.

Table 1. CR and IE% data obtained from WL measurements for MS immersed in a 0.25 mol/L H_2SO_4 solution, without DBS and with it, in various concentrations (mM), for 2 h.

DBS (mM)	CR (mmy)	IE (%)
-	1.4478	-----
	0.8423	41.8
10	0.7061	51.2
30	0.6001	58.5
50	0.5456	62.3
100	0.3797	80.9

The CR was found to depend on the DBS concentration. IE% reached its maximum at 80.9%, with DBS concentration of 100 Mm. Consequently, the CR decreased, due to a wider MS surface coverage, which limited the metal dissolution, by blocking its corrosion sites. The S atom electron donating properties can be attributed to higher IE(%) values. These data are in good agreement with the results reported by many researchers [17].

Electrochemical analysis

The electrochemical measurements are a way for calculating MS CR. They also quickly assess the inhibitor performance, and the surface film durability. The following techniques were used for studying MS corrosion in 0.25 mol/L H_2SO_4, without and with DBS. They revealed if the inhibitor was of the cathodic, anodic or mixed type, and also formulated an appropriate mechanism for its action against corrosion.

PDP study results

PDP study has been used to confirm the formation of a protective film on the MS surface, during the corrosion inhibition process. If a protective film is formed on the MS surface, the LPR values increase and the I_{corr} decreases [18-20]. PDP curves of MS immersed in 0.25 mol/L H_2SO_4, and also DBS IE%, are shown in Fig. 1 (a, b).

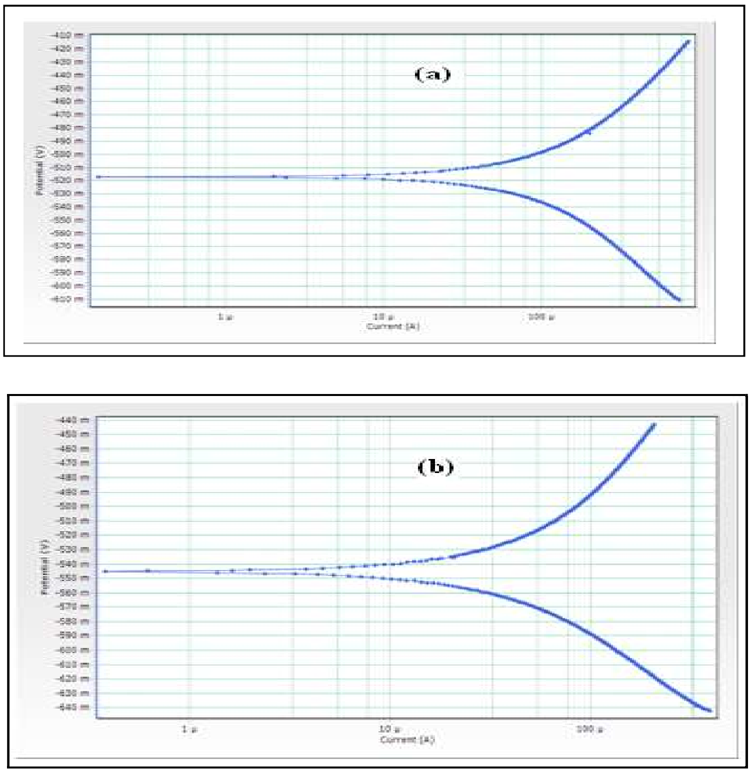

Figure 1. PDP curves of MS immersed in test solutions: **(a)** 0.25 mol/L H_2SO_4 (blank); **(b)** 0.25 mol/L H_2SO_4 + 100 mM DBS.

The corrosion parameters are given in Table 2.

Table 2. Corrosion parameters, obtained by PDP, for MS immersed in 0.25 mol/L, without and with DBS.

Systems	**E_{corr} *vs* SCE (mV)**	**I_{corr} (A/cm^2)**	**β_a (mV/dec)**	**β_c (mV/dec)**	**LPR (ohm/ cm^2)**
0.25 mol/L H_2SO_4	-518	- 617	136	112	-0.4323
0.25 mol/L H_2SO_4 + 100 mM DBS	-545	- 666	192	131	0.5102

When MS was immersed in 0.25 mol/L H_2SO_4, its E_{corr} was -518 mV vs SCE. When 100 mM DBS were added to the solution, MS E_{corr} shifted to the cathodic side, at -545 mV *vs.* SCE. This indicates that the protective film was formed on the MS surface cathodic sites. This film controlled the MS dissolution cathodic reaction, by forming a Fe^{2+}-DBS complex on the metal surface cathodic sites. Further, the LPR value increased from -0.4323 to -0.5102 ohm/cm^2, and I_{corr} decreased from -617 to -666 μA. Thus, PDP study confirmed the formation of a protective film on the MS surface.

Analysis of ACIS results

ACIS analysis has been used to confirm the formation of a protective film on the MS surface [21-22]. If a protective film is formed on the MS surface, R_{ct} and the impedance log (z/ohm) values increase, and C_{dl} decreases. ACIS of MS immersed in 0.25 mol/L H_2SO_4, without and with DBS, are shown in Fig. 2 (a, b), and their values are given in Table 3.

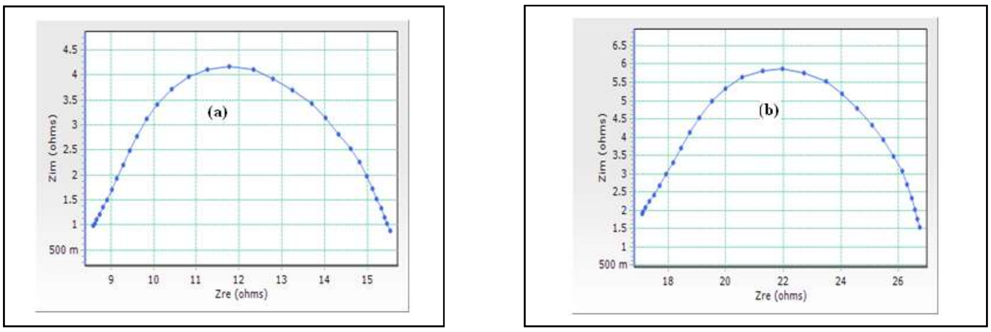

Figure 2. **a.** ACIS of MS immersed in 0.25 mol/L H_2SO_4, without DBS; **b**. ACIS of MS immersed in 0.25 mol/L H_2SO_4, without DBS and with it.

Table 3. Corrosion parameters, obtained from ACIS, of MS immersed in 0.25 mol/L H_2SO_4, without and with DBS.

System	**Nyquist plot**		**Bode plot**
	R_t (Ω/cm^2)	**C_{dl} (F/cm^{-2})**	**Impedance [log (Z/ohm^{-1})]**
0.25 mol/L H_2SO_4	14	8.6412×10^{-5}	0.1180
0.25 mol/L H_2SO_4 + 100 mM DBS	21.97	9.317×10^{-6}	1.053

It is was observed that, when the inhibitor (100 mM DBS) was added to MS immersed in 0.25 mol/L H_2SO_4, R_{ct} increased from 14 to 21.97 Ω cm^2, and C_{dl} value decreased from 8.6412×10^{-5} F cm^{-2} to 9.317×10^{-6} F/ cm^{-2}. The impedance value [log (z/ohm)] increased from 0.1180 to 1.053. These results led to the conclusion that a protective film was formed on the MS surface.

FTIR spectra analysis

FTIR spectra have been used to analyze the protective film formed on the MS surface [23-24]. DBS structure is shown in Fig. 3.

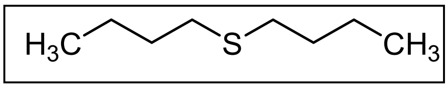

Figure 3. DBS structure.

DBS FTIR spectrum is shown in Fig. 4a. The CH and CS stretching frequencies appear at 2836.25 and 780.96 cm^{-1}, respectively.

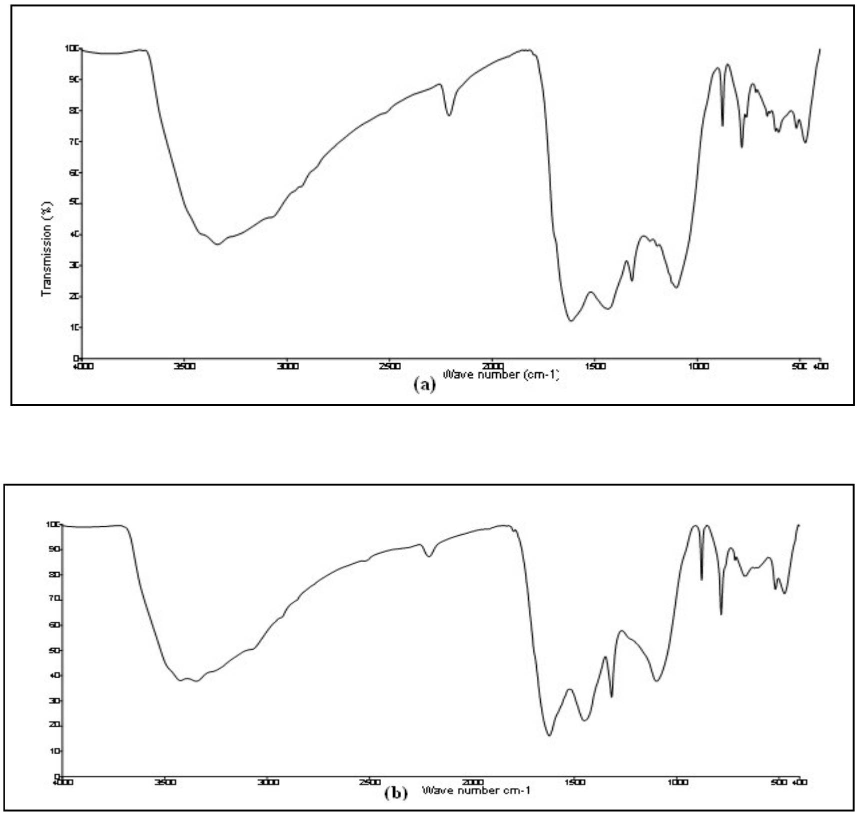

Figure 4. (a) FTIR spectrum of pure DBS; **(b)** FTIR spectrum of the film (Fe^{2+}-DBS complex) formed on the MS surface after immersion in 0.25 mol/L H_2SO_4 containing 100 mM DBS.

The FTIR spectrum of the film complex (Fe^{2+}-DBS) formed on the MS surface, after its immersion in 0.25 mol/L H_2SO_4 with 100 mM DBS, is shown in Fig. 4 b. The CH and CS stretching frequencies have shifted from 2836.25 to 2925.32 cm^{-1}, and from 780.96 to 712.65 cm^{-1}, respectively. A new peak appeared in the region of 490 cm^{-1}, since the DBS S atom has coordinated with Fe^{2+}, forming a complex on the MS surface.

MS surface SEM analysis

To understand the MS surface nature, and its CR without and with the inhibitor system, its SEM micrographs were examined. The SEM images of the MS specimen immersed in 0.25 mol/L H_2SO_4, in the inhibitor system absence and presence, for 2 h, are shown in Fig. 5 (a, b, c, d, e and f).

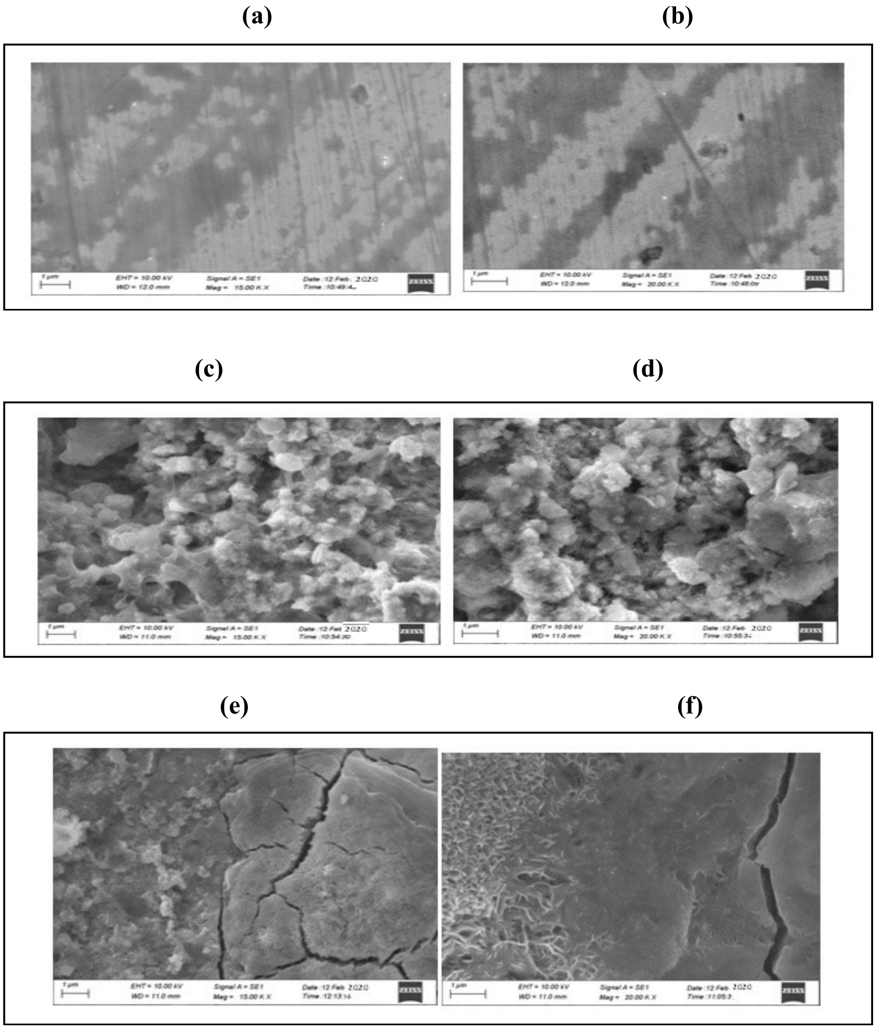

Figure 5. SEM analysis of MS: **(a)** magnification kX 15.00 (control); **(b)** magnification kX 20.00 (control); **(c)** in 0.25 mol/L H_2SO_4, magnification kX 15.00 **(d)** 0.25 mol/L H_2SO_4, magnif. kX 20.00; **(e)** 0.25 mol/L H_2SO_4 + 100 mM DBS, magnif. kX 15.00 (control); and **(f)** 0.25 mol/L H_2SO_4 + 100 mM DBS, magnif. kX 20.00kX 20.00 (control).

The SEM micrographs of Fig. 5 a and b show the polished MS surface (control), without any corrosion products; Fig. 5 c and d show the rough MS surface immersed in 0.25 mol/L H_2SO_4, whith its highly corroded area; Fig. 5 e and f show the MS surface almost free from corrosion, in 0.25 mol/L H_2SO_4 with 100 mM DBS, due to the formation of an insoluble complex. [25].

MS surface EDAX spectra

The EDAX spectra were used to determine the elements present on the MS surface, before and after its exposure to the inhibitor system. The aim of this section was to confirm, with the results obtained from chemical and electrochemical measurements, that an inhibitor surface film was formed on the MS surface. [26-29]. MS EDAX spectrum is shown in Fig. 6a.

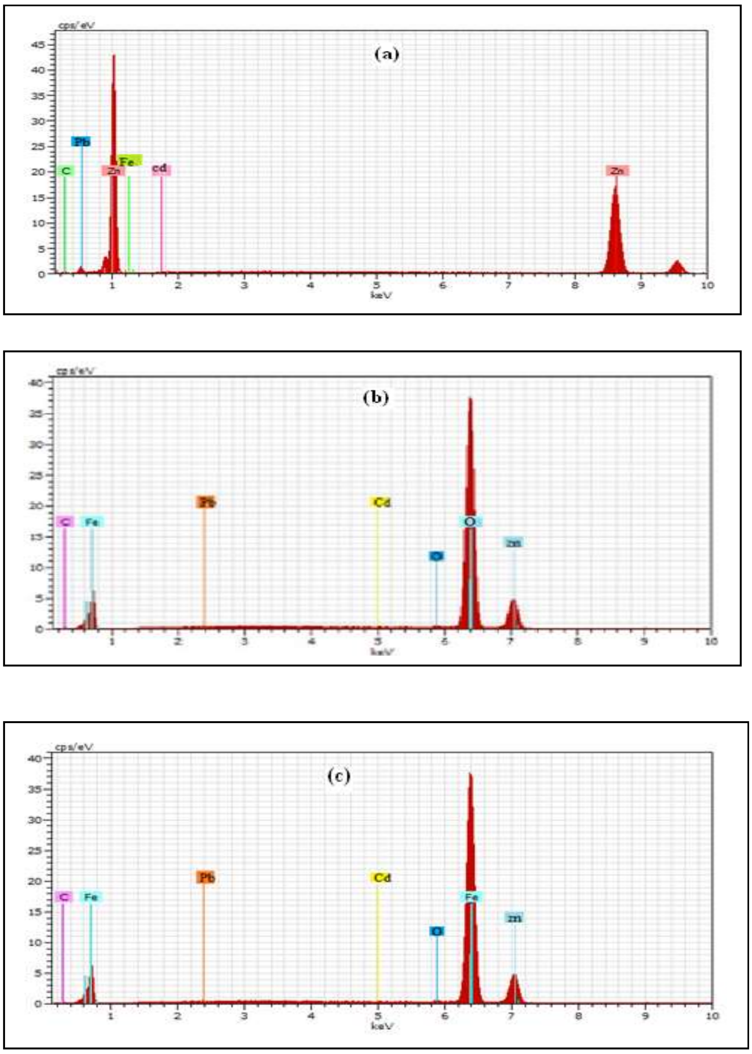

Figure 6. EDAX spectrum of: **(a)** MS specimen (control); **(b)** MS after immersion in 0.25 mol/L H_2SO_4; **c)** MS specimen after immersion in 0.25 mol/L H_2SO_4 + 100 mM DBS.

The EDAX spectrum of MS immersed in 0.25 mol/L H_2SO_4 is shown in Fig. 6b. It indicates that the Fe signal characteristic peaks was reduced, and that the O signal has increased in intensity. This means that MS underwent a corrosion attack by H_2SO_4.
The EDAX spectrum of MS immersed in 0.25 mol/L H_2SO_4 and 100 mM DBS is shown in Fig. 6c. It shows the additional line characteristic of the O signals reduced intensity, and the Fe signal increased intensity. These data show that the MS surface has been covered with Zn, Pd, Cd and Fe atoms. This layer was undoubtedly due to the inhibitor system.
The Fe peaks observed in 0.25 mol/L H_2SO_4 with DBS were considerably suppressed, compared to those observed in the solution without the inhibitor. This suppression occurred due to the overlying inhibitor film, i.e., an inhibitor adsorbed layer that protected MS against corrosion, suggesting that DBS S atom has coordinated with Fe^{2+}, resulting in the complex formation on the MS surface.

Analysis of the AFM results

AFM is a powerful technique for gathering roughness statistics from a variety of surfaces. It is becoming an accepted method of roughness investigation. All AFM images were obtained on an Agilent technology 5500 series mode that operated on the contact mode in air. The scan size of all the AFM images is 5 x 5 µm, at a scan rate of 2.4 lines per s [30-32].
Table 4 shows the various AFM parameters obtained for the MS surface immersed in different environments.

Table 4. AFM data for MS immersed in 0.25 mol/L H_2SO_4, without and with the inhibitor systems.

Environment	AFM parameters		
	Average R_a (nm)	RMS value R_q (nm)	Maximum P/V height (nm)
Polished MS	423	367	2112
Polished MS + 0.5 N H_2SO_4	709	606	2604
Polished MS + 0.5 N H_2SO_4 + 100 mM dibutylsulphide	589	459	2188

The two and three dimensional AFM morphologies and the AFM cross sectional profile, for MS polished surface (reference sample) immersed in 0.25 mol/L H_2SO_4 and 0.25 mol/L H_2SO_4 with 100 mM DBS, are shown in Fig. 7 (a, b, c), (d, e, f) and (g, h, i).
Fig. 7 2D, 3D and broken line (a, d, g) shows the AFM images and cross section analysis of the polished MS surface, with R_a, RMS surface roughness and maximum P/V height values of 423, 367 and 2112 nm, respectively.
Fig.7, 2D, 3D and broken line (b, e, h) shows that MS, after immersion in 0.25 mol/L H_2SO_4 without DBD, had R_a, RMS roughness and maximum P/V height values of 709, 606 and 2604 nm, respectively, indicating the formation of Fe oxides and high

roughness of the corroded metal surface. The surface microstructure shows many smaller and larger corrosion product deposits.

Fig. 7 2D, 3D and broken line (c, f, i) shows that MS, immersed in 0.25 mol/L H_2SO_4 + 100 mM DBS, had R_a, RMS surface roughness and maximum P/V height values of 589, 459 and 2188 nm, respectively, which indicates the formation of a protective film on its surface.

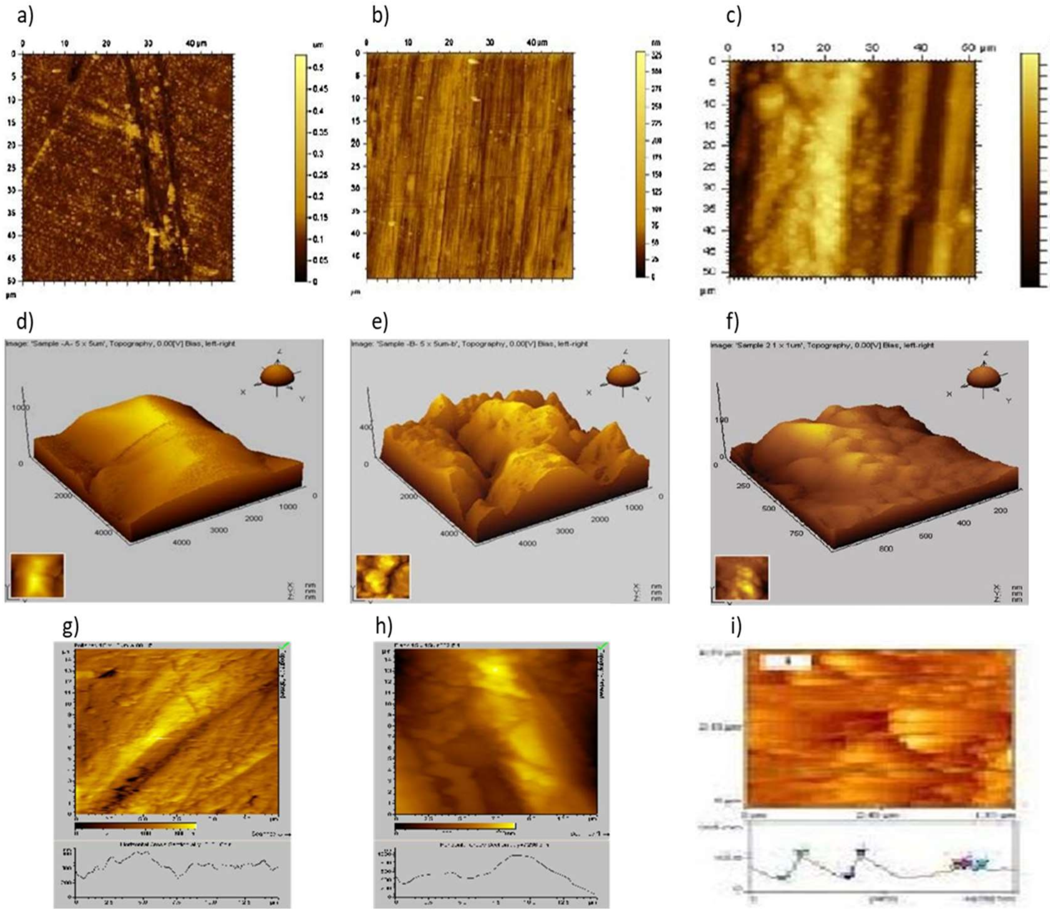

Figure 7. AFM images of MS at different conditions: polished surface (control) – **a)** 2D, **d)** 3D and **g)** cross-sectional profiles; immersed in 0.25 mol/L H_2SO_4 – **b)** 2D, **e)** 3D and **h)** cross-sectional profiles; and immersed in 0.25 mol/L H_2SO_4 + 100 mM DBS – **c)**: 2D, **f)** 3D and **i)** cross-sectional profiles.

The corrosion product was not deposited on the MS surface. Further, these results were confirmed by the clearly visible differences among the optical cross section analyes. The MS surface was covered with a protective film, forming a barrier against the attack by aggressive ions from the corrosive environment. With the inhibitor addition, R_a was reduced to 589 nm, which suggested the inhibitor film formation on the MS surface [33-34].

Conclusion

In this present study, the DBS compound was used as a corrosion inhibitor to control the corrosion of MS immersed in 0.25 mol/L H_2SO_4. The present study led to the following conclusions:

- DBS acted as an inhibitor against corrosion of MS immersed in 0.25 mol/L H_2SO_4, and showed good corrosion IE(%).
- PDP study indicates that the effective DBS system functioned as a cathodic inhibitor, predominantly controlling the cathodic reaction.
- The WL technique showed the IE of 80.9%.
- EIS measurements indicate an increase in R_{ct}, a decrease in C_{dl} and I_{corr} values, due to the adsorbed layer increased thickness.
- FTIR spectra revealed that the protective film composition was the Fe^{2+}-DBS complex.
- SEM micrographs showed a polished and smooth MS surface without the aggressive media, and in it with DBS. The AFM microscopes confirmed the MS surface roughness in 0.25 mol/L H_2SO_4, and its smoothness in the solution with DBS.

Authors' contributions

P. Vijayakumar: selected the problem; made the literature survey; has incorporated new ideas with available literature; has done the experimental part. **S. Valarselvan**: helped in the spectral characterization; suggested the interpretation report; guided in the paper writing. **S. S. Syed Abuthahir**: gave outlines about how to write the paper; helped in the interpretation and analysis part; suggested this Journal.

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Abbreviations

AC: alternating current
ACIS: alternating current impedance spectroscopy
AFM: atomic force microscopy
β_a: anodic Tafel slope
β_c: cathodic Tafel slope
C_{dl}: double layer capacitance
CE: counter electrode
CR: corrosion rate
DBS: dibutyl sulphide
E: potential
E_{corr}: corrosion potential

EDAX: energy dispersive analysis of X-rays
FTIR: Fourier transform infrared
H_2SO_4: sulphuric acid
I: current density
I_{corr}: corrosion current density
IE(%): inhibition efficiency
KBr: potassium bromide
LPR: linear polarization resistance values
MS: mild steel
OCP: open circuit potential
PDP: potentiodynamic polarization
P/V: peak to valley
R_a: average roughness
RE: reference electrode
RMS: root-mean-square
R_{tc}: charge transfer resistance
SEM: scanning electron microscopy
WE: working electrode
WL: weight loss

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