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Corrosion Inhibition of Carbon Steel using Dipropyl Sulphide as Inhibitor System in Sulphuric Acid Solution

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The corrosion inhibition effect of dipropyl sulphide (DPS) on carbon steel immersed in 0.5 N sulphuric acid has been evaluated at room temperature using mass loss method. The corrosion rate and inhibition efficiency was obtained from weight loss method. The corrosion inhibition efficiency increases with increase in concentration of an inhibitor. The corrosion rate decreases when increase in concentration of inhibitor. This is due to the higher concentration of inhibitor solution, which blocks the active site of a carbon steel and a protective film is formed on the carbon steel surface. Electrochemical studies have been used to confirm the formation of protective film over the carbon steel surface. This is further confirmed by surface analysis technique like FTIR spectroscopy and scanning electron microscopy. Energy dispersive analysis of X-ray was used to analyze the elements present over the carbon steel surface. Surface analysis of polished, corroded and inhibitor carbon steel surface has been evaluated by SEM. In order to study adsorption of dipropyl sulphide on carbon steel, *in situ* atomic force microscopy (AFM) measurements were performed in control, with and without dipropyl sulphide in 0.5 N H_2SO_4 solution.

Keywords: Acidic solutions, Carbon steel corrosion, Dipropyl sulphide, Scanning electron microscopy, Mass loss method.

INTRODUCTION

In carbon steel corrosion investigations, the utilization of an acidic medium is essential because of its industrial applications, such as acid pickling, industrial cleaning, petrochemical processes, acid descaling and oil-well acid in oil recovery [1-3]. The refining of crude oil were carried out in a variety of corrosive conditions. The major problems in the industrial use of acids are corrosion of the metal equipment, contamination of the circulating acid leads to the damage of equipments. Generally, acid using materials undergoes corrosion and it is unavoidable one. The corroded materials besides loss in weight and cross-section, can lead to hostile effects on the material properties. Therefore, it is required to prevent or reduce it by using inhibitors or additives [4].

Inhibitors are employed in industrial processes for controlling metal dissolution, especially in neutral, acidic and basic environments. For controlling corrosion, a few organic compounds having polar functional groups or electron-donating groups, aromatic rings with π-electrons and heteroatoms are widely used as potent corrosion inhibitors. These inhibitors are physically or chemically adsorbed onto metal surfaces, thereby creating a film on the surfaces and isolating the metal from corrosive ions existing in a medium. Corrosion causes various adverse effects. These effects on the reliable, safe and efficient operations of structures or equipment are more severe than material loss [5,6]

Several researchers made an attempt to study the inhibition action of various organic compounds on the corrosion of mild steel, aluminium alloys, carbon steel and composites in acids, alkaline and neutral media [7,8]. Even metal complex like copper complex of 1-(8-hydroxyquinolin-2-yl-methyl)thiourea has been used as corrosion inhibitors to control the corrosion of mild steel in sodium chloride solution. Literature survey reported that organic compounds could be used as corrosion inhibitors in controls the corrosion of metals and alloys. The corrosion inhibition of metals in acidic media by different types of organic compounds has been widely studied. The electron donating properties of sulphur atom can be attributed for higher inhibition efficiencies of dipropyl sulphide. The alkyl groups will activate the sulphur atom by increasing the electron density in dipropyl sulphide [9,10].

When compare to other inhibitors, it is simple, easily available inhibitors and will be more effective in controlling the corrosion of metals and alloys [11,12]. The main objective of the present study is to evaluate the effectiveness of dipropyl sulphide as inhibitor to control the corrosion of carbon steel immersed in 0.5N H_2SO_4. The influence of inhibitor in terms of corrosion rate and inhibition efficiency has been evaluated by weight loss method. The mechanistic aspects of corrosion inhibition is determined by electrochemical studies such as AC impedance spectra and polarization studies. The protective film was formed over the surface of carbon steel has been analyzed by Fourier transform infrared (FTIR) spectroscopy technique. The smoothness of carbon steel when compared to polished carbon steel, corroded carbon steel (blank) and carbon steel in inhibitor system has been characterized be scanning electron microscopy (SEM). The elements present on the carbon steel surface has been characterized by energy dispersive analysis of X-ray (EDAX). The roughness of carbon steel surface has been analyzed by atomic force microscopy (AFM) using various regimes.

EXPERIMENTAL

Carbon steel specimens (0.026% S, 0.068% P, 0.36 % Mn, 0.13 % C and the rest iron) of dimensions 1.0 cm × 4.0 cm × 0.2 cm were polished to finish and degreased with acetone and used for mass loss method. The solution (0.5N H_2SO_4) was prepared by dilution of an analytical grade sulphuric acid with double distilled water.

Preparation of stock solutions: Double distilled water was used wherever necessary in the preparation of solutions. Analytical grade H_2SO_4 ($\geq$ 99.9%) was diluted to the required concentrations. The required concentration of organic inhibitor (dipropyl sulphide) stock solution was prepared by dissolving dipropyl sulphide in the minimum amount of ethanol and making up to the desired volume with double distilled water. Then the required volume from the inhibitor stock solution was added to the sulphuric acid solution to obtain the desired concentration.

Weight loss method: Weight loss measurements were done according to the described method [13-15]. Weight loss measurements were performed for 2 h by immersing the carbon steel specimens in 0.5N H_2SO_4 without and with different concentration (5 mM, 10 mM, 30 mM, 50 mM and 100 mM) 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:

$$\text{IE }(\%) = \frac{W_o - W_i}{W_o} \times 100 \tag{1}$$

where W_i and W_o are the weight loss values in g in presence and absence of dipropyl sulphide inhibitor.

Determination of corrosion rate: Carbon steel specimens were weighed in triplicate and suspended in 100 mL of 0.5N H_2SO_4 by using glass hooks in presence and absence of different concentrations of dipropyl sulphide (DPS) inhibitors for 2 h. After 2 h immersion, these specimens were removed, washed under running water, dried, and weighed. Corrosion rates (CRs) were determined from the weight change of the specimen with the following relationship:

$$\text{Corrosion rate} = \frac{87.6 \times \text{Loss in weight (mg)}}{\begin{array}{c}\text{Surface area of the specimen (dm}^2) \times \\ \text{Period of immersion (h)} \times \text{Density}\end{array}} \text{mmy} \tag{2}$$

Corrosion inhibition efficiency (IE) was then calculated using eqn. 3:

$$\text{IE }(\%) = 100\left(1 - \frac{W_2}{W_1}\right) \tag{3}$$

where, W_1 is the corrosion rate in the absence of the inhibitor and W_2 is the corrosion rate in the presence of the inhibitor.

Electrochemical techniques

Potentiodynamic polarization study: Polarization investigations were performed on an electrochemical work station impedance analyser model Princeton Applied Research versa STAT MC. A cell assembly of three electrodes was employed. Carbon steel having an area of 1 cm^2 constantly exposed and the remaining part shielded with the red lacquer was used as working electrode. A saturated calomel electrode (SCE) and rectangular platinum foil were the reference and counter electrodes, respectively. The area of the counter electrode was considerably larger than that of the working electrode. For the counter electrode, this difference can provide a uniform potential field [16]. In 0.5 N sulphuric acid, platinum and working electrodes were immersed in presence and absence of inhibitors. SCE was connected to the test solution with a salt bridge. The plots of log current (I) *versus* potential (E) were recorded. The corrosion potential (E_{corr}), Tafel slopes b_a and b_c, and I_{corr} were determined from these plots.

AC impedance measurements: On an electrochemical work station impedance analyzer model Princeton Applied Research versa STATMC, AC impedance was studied. The same cell setup of polarization estimation was used. For the system to achieve an open circuit potential of the steady state, 5-10 min time interval was provided. Subsequently, on the steady state potential, 10 mV AC potential was superimposed. AC frequency was changed between 100 KHz and 100 MHz. For various frequencies, the real (z') and imaginary (z'') parts of cell impedance were estimated in ohms. The C_{dl} (double layer capacitance) and R_t (charge transfer resistance) were calculated. The following relationship was used to calculate C_{dl} [17]:

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

Surface examination techniques: In blank and organic inhibitor solutions, the carbon steel samples were immersed for 2 h. Then, the specimens were removed and dried. The film produced on the carbon steel surface was characterized using different analysis techniques.

Surface analysis by FTIR spectra: FTIR spectra were obtained using a Perkin-Elmer 1600 spectrophotometer. The acquired film was removed and thoroughly mixed with KBr. This film was pressed into pellets and its FTIR spectra were recorded. After 2 h immersion in various environments, these specimens were removed from test solutions and then dried. The formed film was scratched and thoroughly mixed to render it uniform [18]. The FTIR spectra of the powder of KBr pellets were recorded on the Perkin-Elmer 1600 FTIR spectrophotometer with a resolution of 400 cm^{-1}.

Scanning electron microscopic (SEM) analysis: The carbon steel samples immersed in inhibitor and blank solutions for 2 h were removed, rinsed using double-distilled water and dried. Their surface morphology was analyzed through scanning electron microscopy. The surface morphology of carbon steel was examined using the CAREL ZEISS EVO 18, Hitachi computer controlled scanning electron microscope [19].

Energy dispersive analysis of X-ray (EDAX): The samples immersed in inhibitor and blank solutions for 2 h were removed, rinsed using double-distilled water and dried. The energy dispersive analysis of X-ray (EDAX) was performed to study the elements available on the sample surface [20] by using the Bruker computer-controlled EDAX instrument (Brucker Nano, GMBH, Germany).

Atomic force microscopic (AFM) analysis: The samples immersed in inhibitor and blank solutions for 2 h were removed, rinsed using double-distilled water and dried. The surface of these samples was examined [21]. The surface morphology of the samples was studied using atomic force microscopy (AFM) with Agilent technologies 5500 series mode.

RESULTS AND DISCUSSION

In presence and absence of various dipropyl sulphide (DPS) inhibitor concentrations, mass loss was measured in 0.5 N H_2SO_4. The corrosion rates (CRs) and inhibition efficiencies (IEs) of the carbon steel samples in presence and absence of DPS inhibitor were obtained using the mass loss method. The IE and CR values are presented in Table-1. The corrosion rate (CR) depended on the DPS concentration. With the increase of DPS concentration, IE increases, reaching the maximum at 100 mM concentration. Notably, 100 mM DPS offered 73.7% IE. The increase of IE and decrease of CR are because of the increase in surface coverage at the high inhibitor concentration, which limited carbon steel dissolution by blocking corrosion sites. Hence, CR decreased with the increasing IE when the DPS concentrations increased. The electron-donating characteristics of sulphur attributed to high inhibition efficiency. This findings are in a strong agreement with results reported in literature [22].

TABLE-1
CORROSION RATES (CR) AND INHIBITION EFFICIENCY (IE %) DATA OBTAINED FROM MASS LOSS MEASUREMENTS OF CARBON STEEL IMMERSED IN 0.5N SULPHURIC ACID WITHOUT AND WITH VARIOUS CONCENTRATION OF DPS
Inhibitor system: Dipropyl sulphide (mM); Immersion period: 2 h

Dipropyl sulphide (mM) inhibitor	CR (mmy)	IE (%)
–	1.3372	–
5	0.8501	36.0
10	0.7185	46.2
30	0.6010	55.0
50	0.4844	63.7
100	0.2554	73.7

Electrochemical analysis: The electrochemical measurements provide an approach to calculate the corrosion rate (CR) of mild steel and rapid evaluation of inhibitor performance, surface film durability and corrosion rate.

In 0.5N H_2SO_4 in the presence and absence of the DPS inhibitor, following methods were employed to determine the carbon steel corrosion, to analyze whether the inhibitor is cathodic, anodic or mixed-type inhibitor and to obtain the appropriate mechanism for its corrosion inhibition action.

Potentiodynamic polarization: Polarization study confirmed that the protective film formed during corrosion inhibition on the carbon steel surface. When a protective films forms on the carbon steel surface, the corrosion current (I_{corr}) values decrease and linear polarization resistance values (LPR) increase [23-25].

Fig. 1a-b shows IE for the presence and absence of the inhibitor and the potentiodynamic polarization curves of carbon steel immersed in 0.5 N sulphuric acid. Table-2 lists the corrosion parameters. The corrosion potential was –518 mV *versus* SCE, for carbon steel immersed in 0.5 N sulphuric acid. When 100 mM DPS was incorporated into this system, this corrosion potential shifted to –519 mV *versus* SCE, that is, to the cathodic side, which indicated that the protective film formed at the cathodic sites on the sample surface. The film controlled the cathodic carbon steel dissolution reaction by producing the Fe^{2+}-DPS complex at the cathodic sites of the sample surface.

Linear polarization resistance (LPR) and the corrosion current increased and decreased, respectively, from –0.4323 to –0.6736 ohm cm^2 and from –617 to –633 µA, respectively. Therefore, polarization study confirmed protective film production on the surface of carbon steel.

AC impedance: AC (electrochemical) impedance spectra were used to prove the protective film formation on the sample surface. When a protective film forms on the carbon surface, charge transfer resistance (R_t), double layer capacitance (C_{dl}), and impedance log (z/ohm) increases, decreases and increases,

TABLE-2
CORROSION PARAMETER OF CARBON STEEL IMMERSED IN 0.5N SULPHURIC ACID AND INHIBITION EFFICIENCIES (IE) IN ABSENCE AND PRESENCE OF INHIBITOR SYSTEM 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)
0.5N H_2SO_4	-518	-617	136	112	-0.4323
0.5N H_2SO_4 + 100 mM DPS	-519	-633	229	223	-0.6736

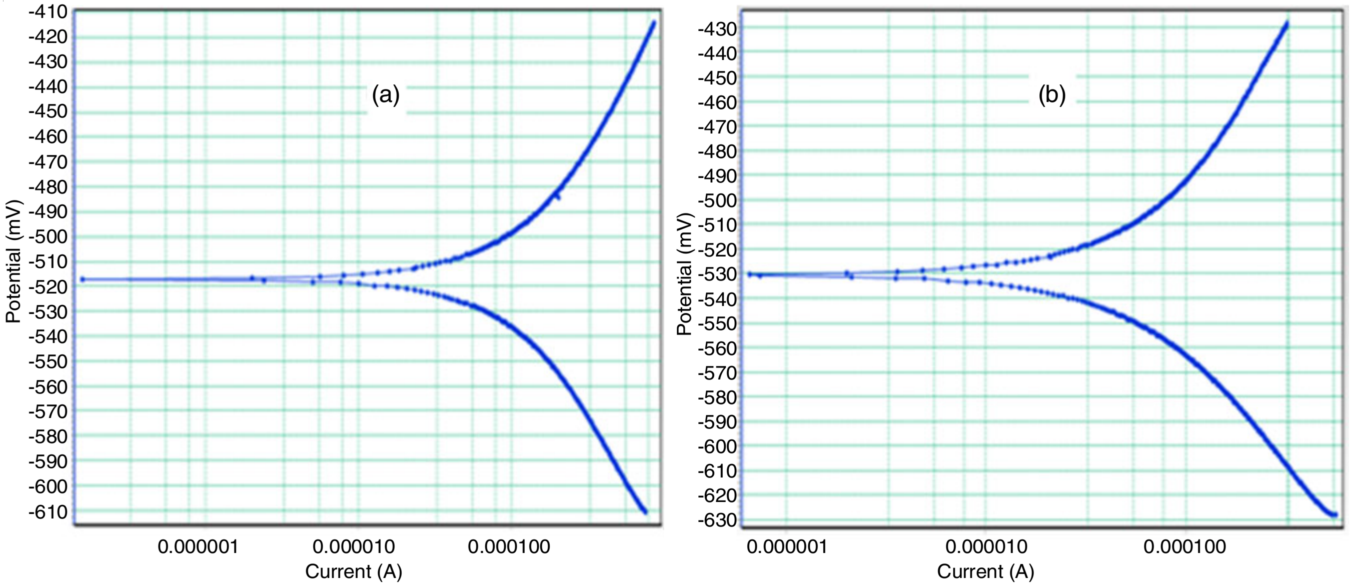

Fig. 1. Polarization curves of carbon steel immersed in test solutions (a) 0.5 N H_2SO_4 (blank), (b) 0.5 N H_2SO_4 + 100 mM dipropyl sulphide

respectively [26,27]. Fig. 2a-b present the AC impedance spectra of the carbon steel sample.

It is observed that when the inhibitor (100 mM of DPS) is added to the above system, the charge transfer resistance (R_t) increases from 14 Ω cm^2 to 19 Ω cm^2 and the C_{dl} value decreases from 8.6412×10^{-5} F cm^{-2} to 19.806×10^{-6} F cm^{-2}. The impedance value [log (z/ohm)] increases from 0.1180 to 0.2154 (Table-3). These results lead to the conclusion that a protective film was formed on the carbon steel surface.

FTIR spectra: FTIR spectra have been used to analyze the protective film formed over on the carbon steel surface [28,29]. The FTIR spectrum (KBr) of pure dipropyl sulphide is shown in Fig. 3a. The CH stretching frequency appears at 2856.11 cm^{-1}. The CS stretching frequency appears at 712.76 cm^{-1}. The FTIR spectrum (KBr) of film formed on the carbon steel surface after immersion in 0.5N H_2SO_4 and 100 mM of dipropyl sulphide is shown in Fig. 3b. The CH stretching frequency has shifted from 2752.10 cm^{-1} to 2823.09 cm^{-1}. The CS stretching frequency has shifted from 611.65 cm^{-1} to 771.15 cm^{-1}. A new peak appears at 490 cm^{-1} is due to formation of Fe^{2+}-complex formed on the carbon steel surface. The sulphur atom of DPS has coordinated with Fe^{2+} and form Fe^{2+}-DPS

TABLE-3
CORROSION PARAMETERS OF CARBON STEEL IMMERSED IN 0.5N H_2SO_4 SOLUTION IN ABSENCE AND PRESENCE OF INHIBITOR SYSTEM BY AC IMPEDANCE SPECTRA

Systems	Nyquist plot		Bode plot
	Rt (Ω cm^2)	C_{dl} (F cm^{-2})	Impedance Lg (Z ohm^{-1})
0.5N H_2SO_4	14	8.6412×10^{-5}	0.1180
0.5N H_2SO_4 + 100 mM DPS	19	19.806×10^{-6}	0.2154

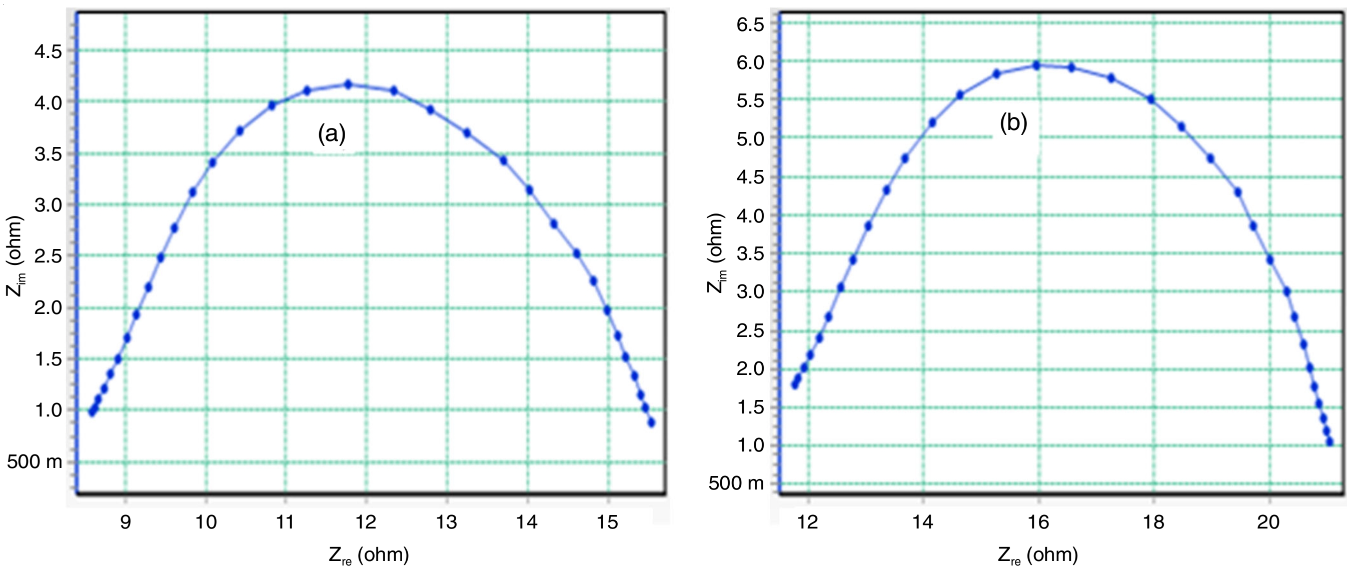

Fig. 2. AC impedance spectra of carbon steel immersed in 0.5N H_2SO_4 (blank) (a) and 0.5 N H_2SO_4 (blank) + 100 mM dipropyl sulphide (b)

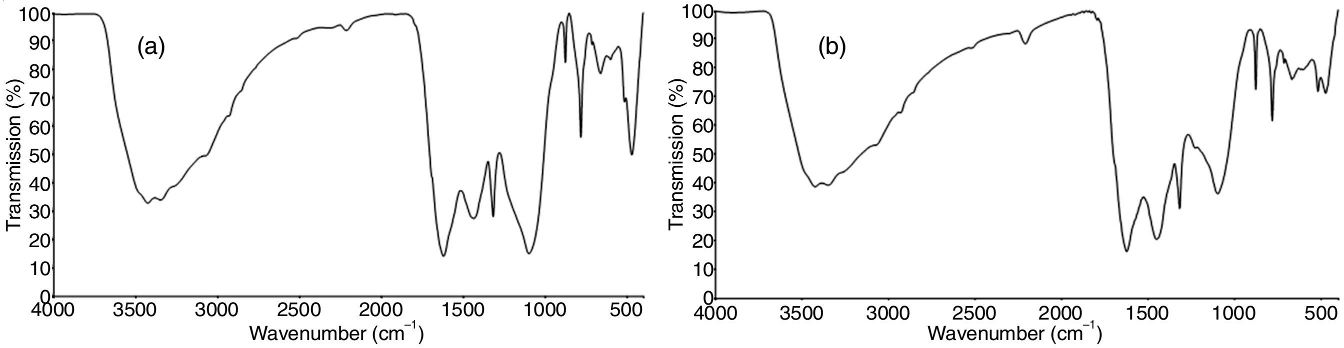

Fig. 3. (a) FTIR spectra of pure dipropyl sulphide (b) film formed on the carbon steel surface after immersion in 0.5N H_2SO_4 solution containing 100 mM dipropyl sulphide

complex on the surface of carbon steel. Thus, the FTIR spectral study leads to the conclusion that the protective film consists of Fe^{2+}-DPS complex.

SEM studies: To determine the nature of the surface film with and without inhibitors and the extent of sample corrosion, the SEM micrographs of the surface were analyzed. Fig. 4a-f present the SEM images of the specimen immersed in 0.5N H_2SO_4 for 2 h with and without the inhibitor. The SEM micrographs presented in Fig. 4a-b illustrate the smooth carbon steel surface, which indicates the lack of corrosion products or inhibitor complexes on the sample surface [30,31].

According to the SEM micrograph of the immersed sample surface (Fig. 4c-d), the carbon steel surface was rough, which revealed the considerably corroded area of the sample in 0.5N H_2SO_4. Fig. 4e-f shows that with inhibitor (100 mM DPS) corrosion rate suppressed, which was observed from a decrease in the corroded areas. The sample surface was almost corrosion free because of the generation of the insoluble complex on it. With DPS, the surface was covered with a thin inhibitor layer, which effectively controlled carbon steel dissolution [32].

Energy dispersive analysis of X-ray (EDAX) studies: The EDAX spectrum of sample obtained before and after inhibitor solution exposure was employed to explore the elements present on the sample surface to confirm the results of electrochemical and chemical measurements, *i.e.* a protective film of the inhibitor was formed on the surface [33,34]. The EDAX analysis of the carbon steel surface was conducted with and without the inhibitor.

Fig. 5a shows the EDAX spectra of carbon steel. The spectra present the characteristic peaks of some elements of the sample. Fig. 5b presents the EDAX spectra for carbon steel immersed in 0.5N H_2SO_4. The characteristic peaks of Fe decreased and

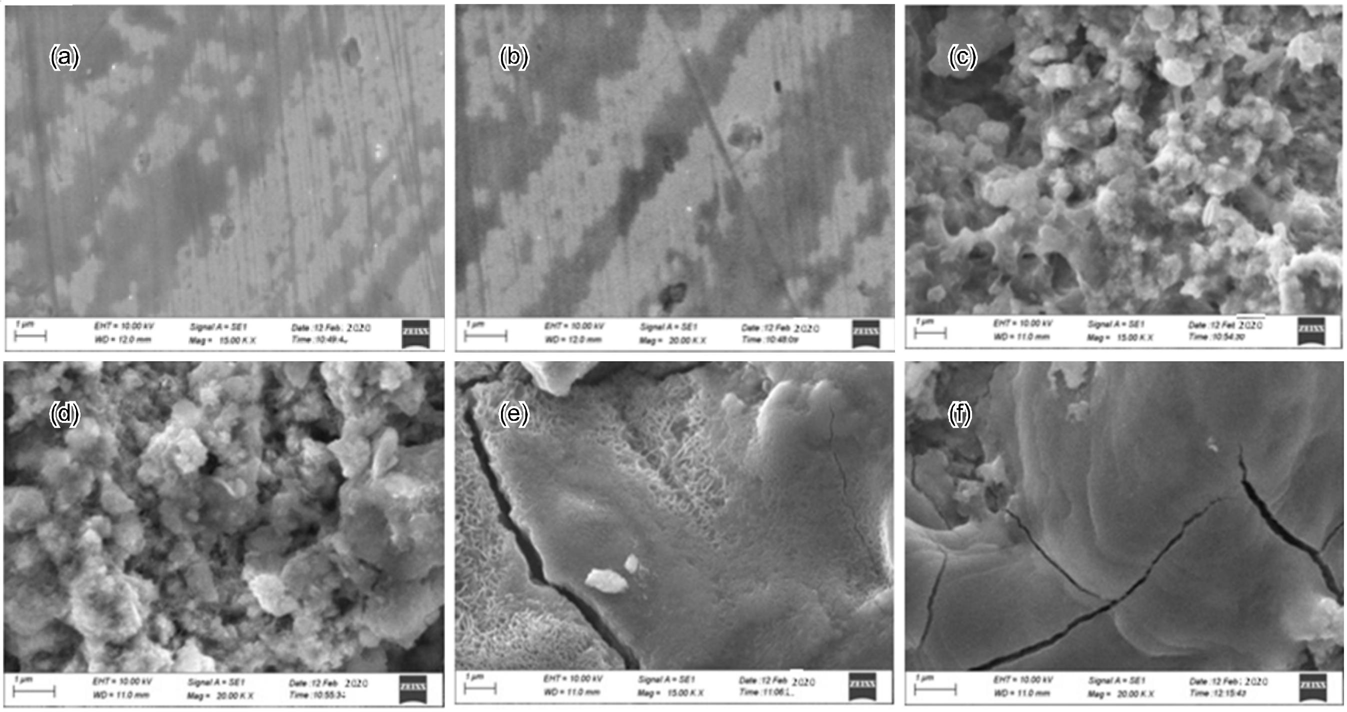

Fig. 4. SEM images of (a) polished carbon steel; Magnification Kx 15.00 (control); (b) polished carbon steel; Magnification Kx 20.00 (control); (c) carbon steel in 0.5N H_2SO_4 solution; Magnification Kx 15.00 (blank); (d) carbon steel in 0.5N H_2SO_4 solution; Magnification Kx 20.00 (blank); (e) carbon steel in 0.5N H_2SO_4 solution + 100 mM DPS solution magnification Kx 15.00; (f) carbon steel in 0.5N H_2SO_4solution + 100 mM DPS solution magnification Kx 20.00

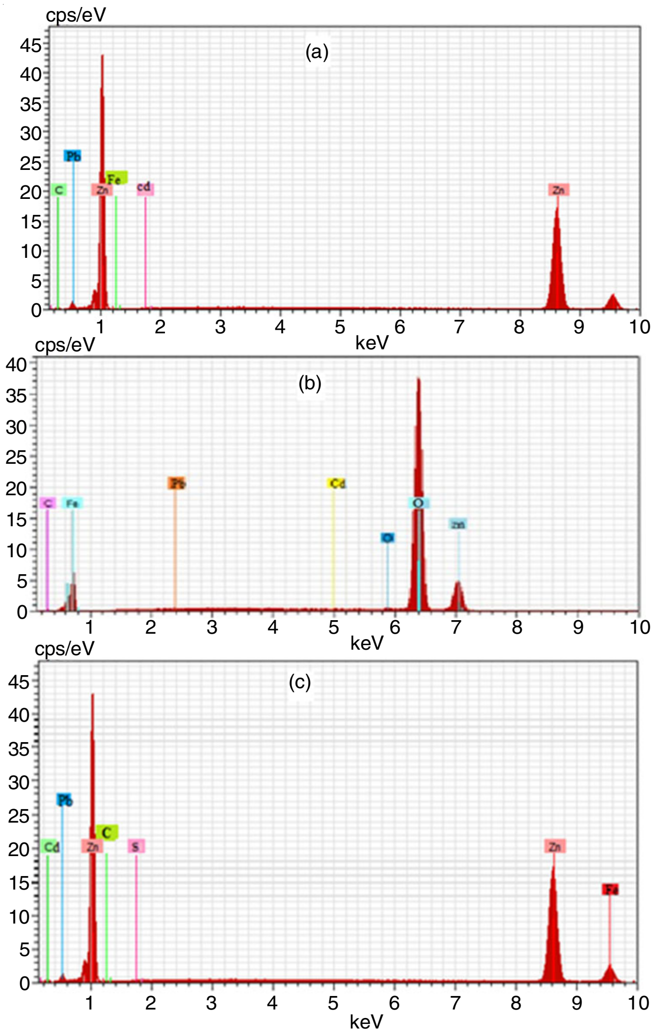

Fig. 5. EDAX spectra of (a) carbon steel specimen (control); (b) carbon steel specimen after immersion in 0.5N H_2SO_4 solution (blank) and (c) carbon steel specimen after immersion in 0.5N H_2SO_4 + 100 m MDPS

the oxygen signal intensity increased, which indicated that carbon steel underwent corrosion by 0.5N H_2SO_4. The spectra present the characteristic peaks of some elements of the sample. Fig. 5c presents the EDAX spectra of carbon steel immersed 0.5N H_2SO_4 and 100 mM DPS; this spectra show additional line characteristics. The oxygen signal intensity decreased, and the Fe signal intensity increased. This data showed that the carbon steel surface was covered with Pb, C, Pd, Zn, Cd and Fe atoms due to the inhibitor system.

The Fe peaks obtained with the inhibitor are considerably suppressed compared with those observed for 0.5N H_2SO_4 (blank solution) (Fig. 5c). The Zn peaks were suppressed due to the overlying inhibitor film, which indicated the presence of an adsorbed inhibitor layer protecting the zinc metal against corrosion. The sulphur atom of DPS coordinated with Fe^{2+}, leading to Fe^{2+}-DPS complex formation on the carbon steel surface.

Atomic force microscopic (AFM) studies: AFM is a powerful tool for obtaining the roughness statistics from the various surfaces. All the AFM images were acquired in the Agilent technologies 5500 series mode. The AFM instrument was operated in air in the contact mode. The size for all AFM images was 5 µm × 5 µm and a scan rate was 2.4 lines per second [35-37].

Fig. 6a-c, d-f, and g-i show two dimensional, three dimensional AFM morphologies and the AFM cross sectional profiles, respectively, for the polished carbon steel surface (reference sample), carbon steel surface immersed in 0.5N H_2SO_4 (blank sample) and carbon steel surface immersed in 0.5N H_2SO_4 with 100 mM DPS, respectively.

The surface topographies recorded in 2D cross sectional profile diagram, 2D and 3D images were examined. Surface roughness (RMS), average roughness (R_a) and maximum peak-to-valley height were estimated using the images. Table-4 presents various AFM parameters for the sample surface immersed in different solutions. The cross-section analysis of the polished zinc metal surface with a R_a value 423 nm, RMS value 367 nm and maximum peak to valley height value of 2112 nm (Fig. 6a, d and g). After immersion in 0.5N H_2SO_4 in the absence of the inhibitor, with an increased R_a value 709 nm, RMS value 606 nm and maximum peak to valley height value of 2604 nm, indicating the formation of iron oxides (Fig. 6b, e and h) . The root-mean-square (RMS) roughness is found to be 606 nm, which clearly indicates the high roughness of the corroded carbon steel surface. The microstructure of the surface shows many smaller and larger corrosion product deposits.

However, when zinc metal immersed in inhibitor consisted of 0.5N H_2SO_4 + 100 mM DPS show a decreased Ra value 590 nm, RMS value of 461 nm and maximum peak to valley height is 2190 nm, which indicates the formation of a protective film on the carbon steel surface (Fig. 6c, f and i).

On the carbon steel surface, the corrosion product could not deposit. Furthermore, the differences in optical cross section analyses verified the results. The carbon steel sample surface was coated with the protective film, which formed a barrier against the attack from aggressive ions in a corrosive environment. With inhibitor addition, the average roughness decreased to 590 nm, which revealed that the inhibitor film was formed on the surface of carbon steel.

TABLE-4
AFM DATA OF CARBON STEEL IMMERSED IN THE PRESENCE AND ABSENCE OF INHIBITOR SYSTEMS

Environment	AFM parameters		
	Average roughness R_a (nm)	RMS value R_q (nm)	Maximum peak-to-valley height (nm)
Polished carbon steel surface	423	367	2112
Polished carbon steel + 0.5 N H_2SO_4	709	606	2604
Polished carbon steel + 0.5 N H_2SO_4 + 100 mM dipropyl sulphide	590	461	2190

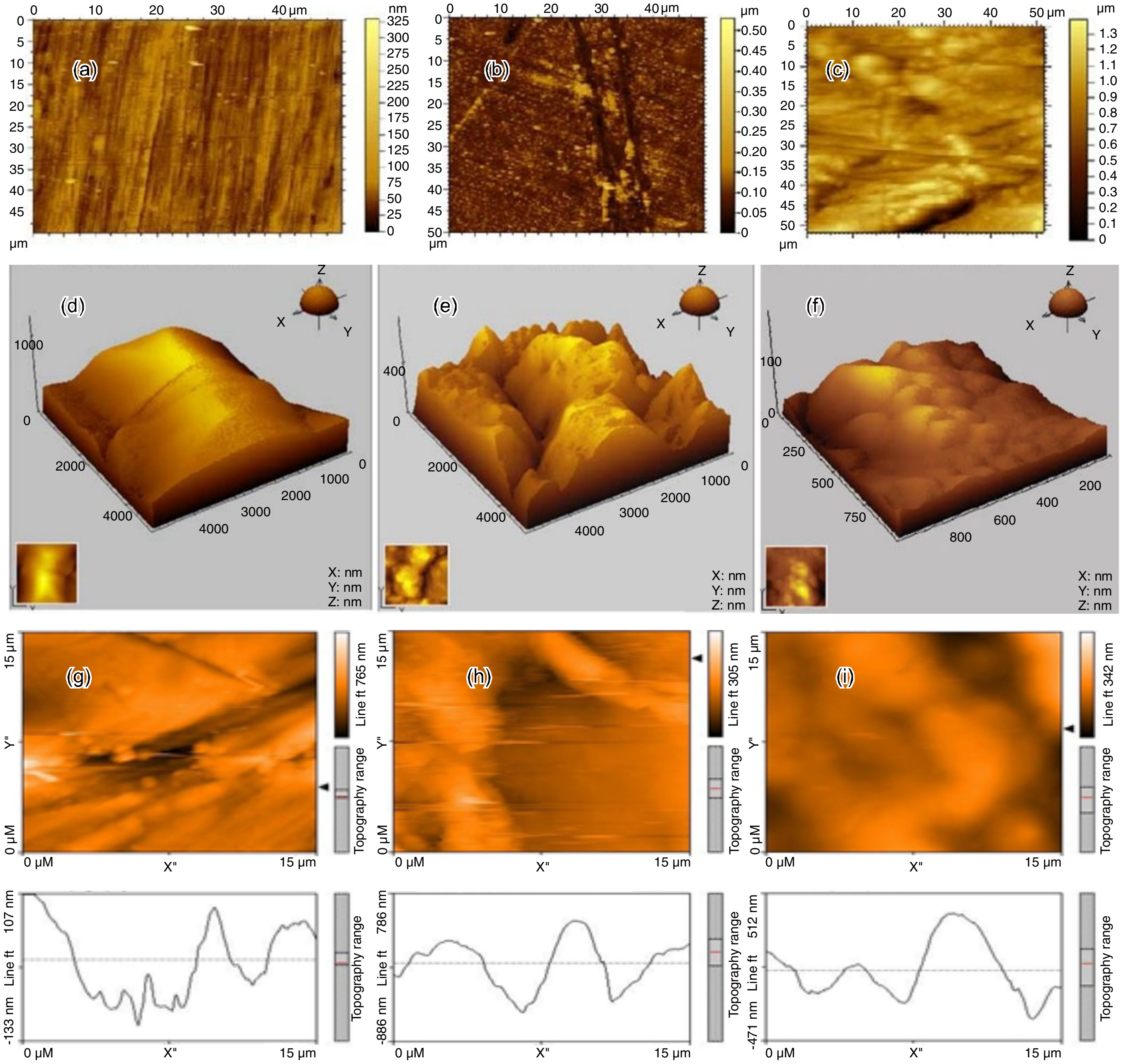

Fig. 6. 2D AFM images of the surface of (a) polished carbon steel (control); (b) carbon steel immersed in 0.5N H_2SO_4 (blank); (c) carbon steel immersed in 0.5N H_2SO_4 containing 100 mM DPS; 3D AFM images of the surface of (d) carbon steel metal (control); (e) carbon steel immersed in 0.5N H_2SO_4 (blank); (f) carbon steel immersed in 0.5N H_2SO_4 containing 100 mM DPS; the cross-sectional profiles, which are corresponding to as shown broken lines in AFM images of the surface of (g) polished carbon steel (control); (g) carbon steel immersed in 0.5N H_2SO_4 (blank) and (i) carbon steel immersed 0.5N H_2SO_4 containing 100 mM DPS

Conclusion

In this study, dipropyl sulphide has been used as a corrosion inhibitor to put off the corrosion of carbon steel engrossed in 0.5N H_2SO_4. The results revealed that dipropyl sulphide inhibitor acted as a good corrosion inhibition efficiency in controlling the corrosion of carbon steel is immersed in 0.5N H_2SO_4. The polarization study showed that the effective dipropyl sulphide systems function as cathodic inhibitor controlling the cathodic reaction predominantly. The mass loss technique shows the inhibition efficiency is 73.7%. The electrochemical impedance measurements indicate that an increase the charge transfer resistance (R_t), decrease the double layer capacitance (C_{dl}) and corrosion current (I_{corr}) values owing to the increased thickness of adsorbed layer. FTIR spectra revealed that the protective film consists of Fe^{2+}-DPS complex. The SEM micrographs exhibit the smoothness of carbon steel surface like polished carbon steel, moreover, the AFM microscopic images also confirmed the roughness and smoothness of carbon steel surface.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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