# CORROSION MONITORING STUDIES AND SURFACE MORPHOLOGY OF MILD STEEL IN 0.5N HYDROCHLORIC ACID USING DIMETHYL SULPHIDE

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#### **ABSTRACT**

Dimethyl sulphide, more often known as DMS, acts as a corrosion inhibitor when applied to 0.5N mild steel. This property of DMS allows it to be used in this context. The mass loss method at room temperature is utilized as the major testing approach for analyzing hydrochloric acid. This technique is used to determine the concentration of the acid. The effectiveness of an inhibitor in preventing corrosion is proportional to the amount of that inhibitor present in the system. The corrosion pace may be slowed by raising the inhibitor's concentration in the system. Raising the concentration of the inhibitor solution on the surface of the mild steel makes it possible to prevent the creation of a protective layer on the surface of the mild steel, which would otherwise obstruct the active site. Electrochemical tests on mild steel, such as AC impedance and polarisation, have been performed to determine whether or not a protective coating has developed on the material. These tests aim to determine whether or not a coating has formed. Researchers working in the field of electrochemistry have found that using Fourier transform infrared spectroscopy has been beneficial to their work (FTIR). In order to conduct our studies on polished, corroded, and inhibitor-treated mild steel surfaces, a scanning electron microscope and an energy dispersive x-ray analyzer were used (EDAX). The surface's roughness and smoothness can be seen in the AFM micrographs.

Keywords: Corrosion, Dimethyl sulphide, EDAX, Mild steel, SEM, and AFM

#### INTRODUCTION

Corrosion is the gradual chemical or electrochemical breakdown of a metal or its elements. Corrosion may decrease the binding energy of metals. Corrosion causes metal atoms to oxidise, resulting in the loss of electrons. Corrosion is the process that essentially turns metal into powder. Material properties are diminished as a result of corrosion. Corrosion occurs when metals and nonmetals are in touch with their environments. There are consequences for the material systems and infrastructures. Steel rusts when oxygen and moisture combine in the air. Pipelines and structures wear out faster or slower depending on the soil's chemical and moisture level. Carbon steel and iron are prone to corrosion, both in theory and in practice<sup>1</sup>. Equipment and buildings suffer a greater loss in efficiency, consistency, and productivity due to corrosion than they do to the loss of metal. Some metal loss is inevitable, but even little loss might lead to

expensive repairs or replacements<sup>2</sup>. It doesn't matter how much metal is damaged, catastrophic failures will occur. Corrosion is the outcome of a chemical interaction between the metal and the air. This is a costly problem that never seems to go away. Corrosion inhibitors may protect against rust. Corrosion may transform refined metals into more chemically stable compounds like oxide, hydroxide, or sulphide. It is the progressive breakdown of materials over time due to chemical and/or electrochemical interactions<sup>3</sup>. This often affects metals. Inhibitors prevent metals from dissolving in situations that are acidic, neutral, or basic. Most industrial inhibitors have at least one functional group, which acts as the adsorption active centre. Researchers looked at how organic molecules fared in acidic, alkaline, and neutral conditions on a variety of metals and metal composites. Mass loss and electrochemical impedance spectroscopy were used to evaluate corrosion and inhibition. To learn more about the mild steel's protective coating, FTIR spectroscopy was used. The SEM is used to examine the surface morphology. SEM was used to examine the smoothness of mild steel with and without inhibitors. We used X-ray energy dispersive analysis to compare the polished, blank, and inhibitor systems of mild steel. Atomic force microscopy was used to examine the surface's roughness and smoothness.

#### MATERIALS AND METHODS

#### Mild steel specimen

Carbon - 0.1%, Sulphur - 0.026 %, Phosphorus - 0.06 %, Manganese - 0.4%, and the rest iron were polished to mirrors and degreased with acetone for weight reduction.

#### **Ingredients**

Analytical grade hydrochloric acid was diluted with double-distilled water (0.5 N HCl). ARgrade chemicals were also utilised.

### Stock solutions

Solution preparation required double-distilled water. Analytical-grade HCl was diluted to the necessary concentration. Organic inhibitors (Dimethyl sulphide) stock solution was made by dissolving inhibitors in ethanol and topping up with double-distilled water. The appropriate concentration of inhibitor stock solution was added to the hydrochloric acid solution.

### **Techniques**

#### Measure mass loss

Mild steel specimens were immersed in 0.5N hydrochloric acid for 2 hours with and without organic inhibitors (5 mM, 10 mM, 30 mM, 50 mM, 100 mM). The specimen was then removed, cleaned, dried, and measured. The equation below calculates inhibitory efficiency (IE%).

IE (%) = 
$$\frac{W_0 - W_j}{W_0}$$
 X 100

Where Wi and Wo are weight loss values in gram with and without the organic inhibitor (dimethyl sulphide).

#### Potentiodynamic polarization

CHI 6608 electrochemical workstation impedance analyzer with a three-electrode cell assembly was used for polarization investigations. The working electrode was mild steel with a 1cm2 exposed surface and red lacquer shielding. The reference electrode was a saturated calomel electrode (SCE), and the counter electrode was a platinum foil. Counter electrode has more surface area than working electrode. This produces a homogeneous potential field at the counter

electrode. Working and platinum electrodes were submerged in 0.5 N hydrochloric acid with and without inhibitors. A saturated calomel electrode was bound to the test fluid via a salt bridge. Plots of potential (E) vs. log current (I) were made. The results such as corrosion potential (Ecorr) and Icorr Tafel slopes ba, bc were determined from E Vs log I plots.

#### Measure AC impedance

Princeton Applied Research versa electrochemical workstation impedance analyzer STATMC analyzed AC impedance. Cell arrangement was comparable to polarization testing. The gadget had 5-10 minutes to attain steady-state open circuit potential. Over this steady-state potential, 10 mV AC was overlaid. AC frequency was changed from 100 kHz to 100 MHz, and real (Z) and imaginary (z") cell impedance was computed in ohms. Cdl and Rt were determined. Cdl was measured using the following relationship.

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

# Surface studies

Mild steel was soaked in blank and organic inhibitor solutions for 2 hours. After 2 hours, the samples were dried. Various approaches were employed to analyze the zinc film.

### FTIR spectra analysis

FTIR spectra were recorded using a Perkin-Elmer 1600 spectrophotometer. After mixing the film with KBr and forming pellets, the FTIR spectra were recorded. After 2 hours in test solutions, samples were removed and dried. The surface film was scraped and blended to maintain uniformity. Perkin–Elmer 1600 FTIR spectrophotometer was used to record the KBr pellet's FTIR spectrum.

#### **SEM**

After 2 hours in blank and inhibitor solutions, the mild steel specimen was removed, washed with double-distilled water, cleaned, and inspected under a SEM. Mild steel's surface morphology was studied using a CAREL ZEISS EVO 18, Hitachi SEM.

#### **EDAX**

The mild steel specimen was immersed in blank and inhibitor solution for 2 hours, washed with double distilled water, cleaned, and analyzed with EDAX to identify the surface elements [4,5]. Bruker computer-controlled Energy Dispersive Analysis of X-Rays analysed the mild steel surface (Brucker Nano, GMBH, Germany).

#### **AFM**

After 2 hours in blank and inhibitor solutions, the mild steel specimen was removed, washed with double-distilled water, cleaned, and inspected [6]. Using atomic force microscopy, Agilent 5500 series model measured mild steel surface morphology (AFM).

#### RESULTS AND DISCUSSION

Mass loss method analysis

Mass loss technique is used to measure mild steel corrosion rates (CR) in 0.5 N hydrochloric acid and inhibition efficiencies (IE) in the absence and presence of dimethyl sulphide inhibitor. Table 1 shows corrosion rate and inhibition efficiency. 100 mM dimethyl sulphide inhibits 69.9%. As dimethyl sulphide concentration rises, corrosion rate drops. A larger dosage of the inhibitor increases surface coverage, slowing mild steel disintegration. Sulfur's electron-donating characteristics increase inhibition efficiency. This monitoring confirms several studies' findings <sup>7</sup>.

Table 1: Corrosion rates (CR) of mild steel is immersed in 0.5N HCl absence and presence of inhibitor systems at various concentration and the inhibition efficiency (IE %) obtained by mass loss method.

Inhibitor System: Dimethyl sulphide (mM); Immersion period: 2 Hours Method: Mass loss method.			
Hydrochloric acid (N)	Dimethyl ulphide (mM)	CR (my)	IE (%)
0.5	Blank	1.4478	
0.5	5	1.9087	30.3
0.5	10	0.8505	41.2
0.5	30	0.7318	49.4
0.5	50	0.6131	57.6
0.5	100	0.4351	69.9

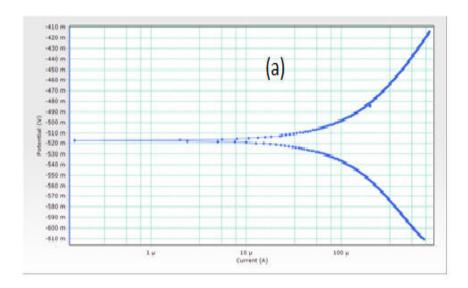
#### **Electrochemistry**

### Potentiodynamic polarizations

During corrosion inhibition, polarization analysis confirmed a protective coating on mild steel. LPR increases when a protective coating builds on mild steel, whereas Icorr drops. <sup>8,9</sup>. Figures 1a and 1b show the potentiodynamic polarization curves of mild steel in 0.5N hydrochloric acid and inhibition efficiencies (IE) with and without an inhibitor. Table 2 describes corrosion parameters. Mild steel corrodes at -517 mV versus SCE in 0.5N hydrochloric acid. 100 mM dimethyl sulphide changed the device's corrosion potential to cathodic (-530 mV vs SCE). This film controls mild steel dissolving by generating the Fe2+-DMS complex on its cathodic sites. LPR increases from 1054717 to 1640465 ohm cm², whereas corrosion current lowers from -151 to -185 A/cm³v. Polarization analysis verifies production of a protective coating on mild steel.

Table 2. Corrosion parameters of mild steel are immersed in 0.5 N hydrochloric acid and inhibition efficiencies (IE) in the absence and presence of inhibitor system obtained by potentiodynamic polarization method

C 4	E <sub>corr</sub> vs SCE (mV)	I <sub>corr</sub> A / cm <sup>2</sup>	ba (mV/dec)	bc (mV/dec)	LPR (ohm cm <sup>2</sup> )
0.5 N HCl	-517	-151	133	139	1054717
0.5 N HCl +					
100 mM DMS	-530	-185	174	125	1640465



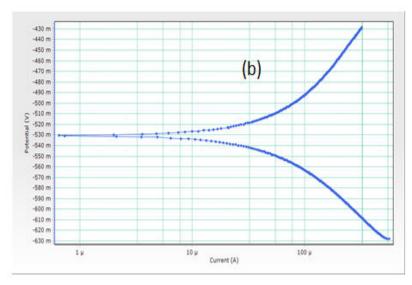


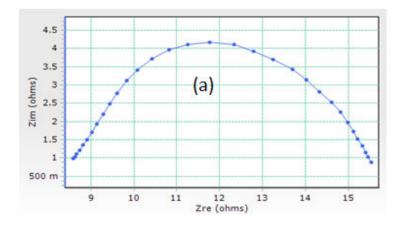
Fig. 1: Polarization curves of mild steel immersed in test solutions: (a) 0.5~N~HCl (blank) (b) 0.5~N~HCl+100~mM dipropylsulphide

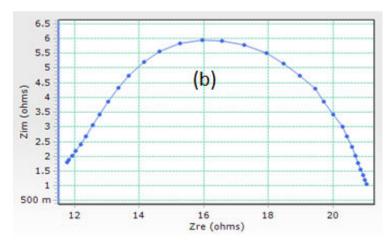
AC impedance spectra analysis

Electrochemical impedance spectra indicate the production of a protective coating on mild steel<sup>10-12</sup>. As a protective coating forms on mild steel, charge transfer resistance (Rt), double layer capacitance (Cdl), and impedance log(z/ohm-1) rise. Figure 2a and 2b show the AC impedance spectra of mild steel submerged in 0.5 N HCl with and without inhibitor (DMS). When 100 mM of DMS is introduced to the aforementioned solution, charge transfer resistance (Rt) rises from 8.03 cm2 to 11.38 cm2 and Cdl reduces from 6.53110-5 Fcm-2 to 68.749 10-6 Fcm-2. [log (z/ohm-1)] rises from 0.762-1.392. These results suggest a protective coating on mild steel.

Table 3: Corrosion parameters of mild steel immersed in 0.5 N HCl solution in the absence and presence of inhibitor system obtained from AC impedance spectra

	Impedance		
Systems	$R_t \Omega cm^2$	C <sub>dl</sub> F/cm <sup>-2</sup>	Log (Z ohm <sup>-1</sup> )
0.5 N HCl	08.03	6.531×10 <sup>-5</sup>	0.762
0.5 N HCl + 100 mM DMS	11.38	68.749×10 <sup>-6</sup>	1.392





# Fig. 2: AC impedance spectra of mild steel immersed in various test solutions a) 0.5 N HCl(blank) b) 0.5 N HCl + 100 mM dipropylsulphide

# Surface analysis

# FTIR spectra analysis

FTIR spectra were used to study the protective coating on mild steel. Figure 3 shows dimethyl sulfide's structure. Figure 4a shows the dimethyl sulphide FTIR spectrum (KBr). CH stretching frequency is 2875.91cm-1; CS is 758.44cm-1. Figure 4b shows the FTIR spectrum (KBr) of the zinc metal film produced in 0.5 N HCl and 100 mM dimethyl sulphide. A Protective thin film is formed on the surface of the carbon steel immersed in A shift of the C-H stretching from 3410.13 to 3417.67 cm<sup>-1</sup> indicates that the molecular adsorption 13-15. The shift in frequency from 2720.16 to 2865.42 cm<sup>-1</sup> are noticed for S-H group. The shift in frequency from 1650.10 to 1579.44 cm<sup>-1</sup> are noticed for C=N group. The C-C frequency is shifted from 1014.13 cm<sup>-1</sup> to 1060.64 cm<sup>-1</sup>. The peaks at 1590.02 cm<sup>-1</sup> for C-N group is shifted to 1399.08 cm<sup>-1</sup>. The band 585.86 cm<sup>-1</sup> considerably originate mainly from Fe-complex (Table 4). All the above bands clearly indicate the formation of a complex on the carbon steel surface. The sulphur atom of DMS coordinates with Fe2+ on mild steel's surface. FTIR spectrum study shows that the protective coating is Fe+2 – DMS.

Figure 3. Structure of Dimethyl sulphide

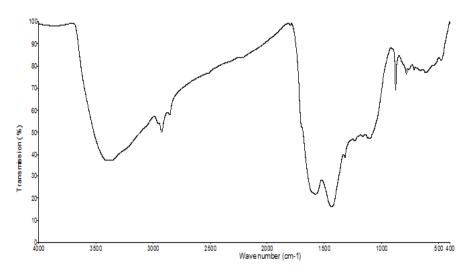


Fig. 4a: FTIR spectrum of pure methyl sulphide

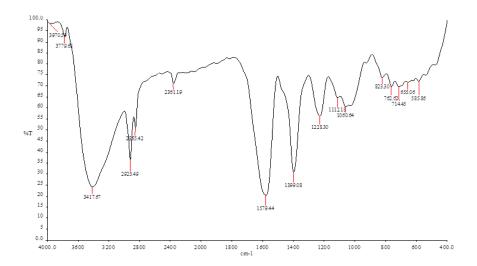


Fig. 4b: FTIR spectrum of the film formed on the mild steel surface after immersion in 0.5 N HCl solution containing 100 mM DMS.

Table – 4: FT-IR spectral data mild steel in 0.5n hydrochloric acid using dimethyl sulphide

IR bands	IR Bands of film from mild steel surface	Frequency assignment to functional groups
3410.13	3417.67	C-H stretching
1650.10	1579.44	C=N stretching
1014.13	1060.64	C-C stretching
2720.16	2865.42	SH
1490.36	1399.08	C-N
-	585.86	Y-Fe <sub>2</sub> 0 <sub>3</sub>

#### SEM steel Surface analysis

SEM can view mild steel's surface (SEM). SEM micrographs were used to examine the surface film structure in the absence and presence of inhibitors and the degree of mild steel corrosion <sup>16</sup>. Figure 5a shows SEM micrographs of polished mild steel (control). No corrosion products or inhibitor compounds had developed on the mild steel surface. Figure 5b shows the roughness of mild steel in 0.5 N HCl. 0.5 N HCl significantly corrodes mild steel. As seen in Figure 5c, corrosion has decreased. Inhibitors slow rusting (100 mM DMS). The mild steel surface is practically corrosion-free due to an insoluble compound. In the presence of DMS, inhibitors cover the surface, inhibiting mild steel dissolution <sup>17</sup>.

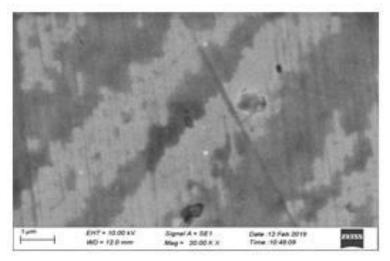


Fig. 5 (a)

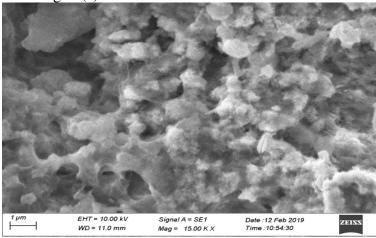


Fig. 5 (b)

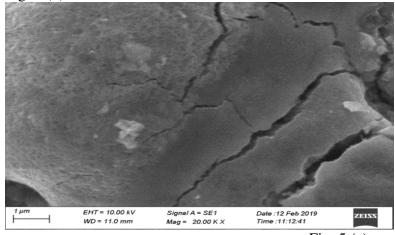


Fig. 5 (c)

Fig.5: SEM analysis of (a) Mild steel; Magnification (control); (b) Mild steel in 0.5 N HCl solution (blank); (c) Mild steel in 0.5 N HCl solution + 100 mM DMS solution

# EDAX of mild steel surface

EDAX (Energy dispersive analysis of X-rays) spectra were utilized to examine the mild steel surface elements before and after exposure to the inhibitor. This section confirmed that a protective surface coating of inhibitor formed on mild steel using chemical and electrochemical studies. EDAX tests were done on mild steel surfaces with and without an inhibitor unit <sup>18,19</sup>. Figure 6a shows mild steel's EDAX spectrum while Figure 6b shows it in 0.5 N HCl. The lowered Fe signal and increased O signal indicate that 0.5N HCl corrodes mild steel. They also display typical peaks of zinc metal's components. Figure 6c displays zinc metal in 0.5 N HCl and 100 mM dimethyl sulphide. It demonstrates that O signal strength is lowered and Fe signal intensity is enhanced. According to these results, mild steel is covered with Fe, Pd, Cd, and Fe. This layer is caused by inhibition. Fe signal and substantial O and C contribution are missing on mild steel subjected to 0.5 N HCl. Inhibitor reduces Zn peaks compared to 0.5 N HCl. Inhibitor film suppresses Fe peaks. This implies a corrosion-protecting inhibitor-adsorbed layer on mild steel. These data imply that DMS's sulphur atom coordinates with Fe2+, forming the Fe2+-DMS complex on mild steel.

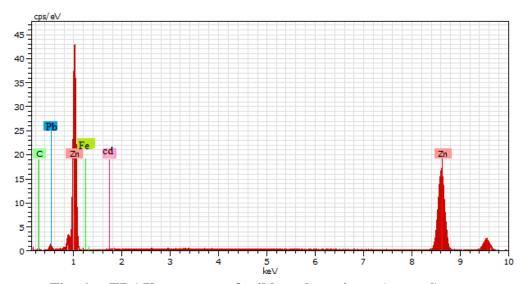


Fig. 6a: EDAX spectrum of mild steel specimen (control)

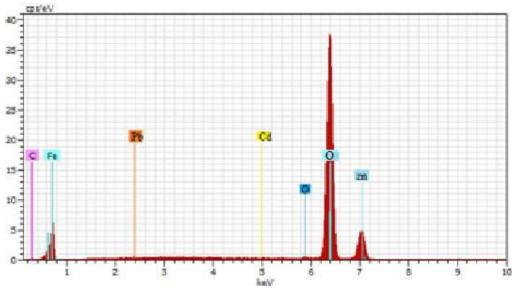


Fig. 6b: EDAX spectrum of mild steel specimen after immersion in 0.5 N HCl solution (blank)

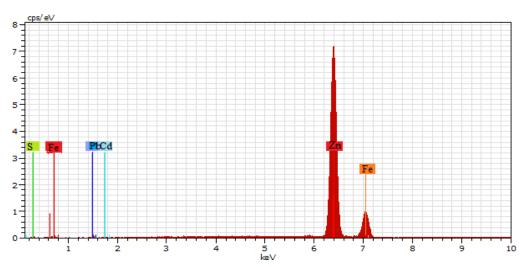


Fig. 6c: EDAX spectrum of mild steel specimen after immersion in  $0.5\ N\ HCl+100\ mM$  DMS

### AFM Analysis of mild steel surface

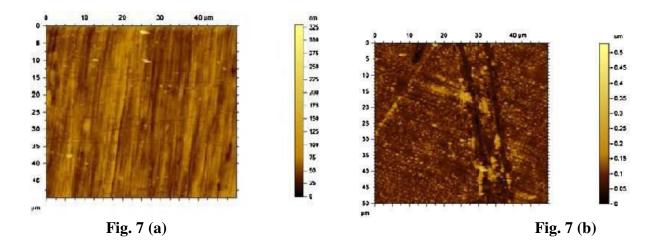
Atomic force microscopy (AFM) is a high-resolution scanning probe and one of the most powerful techniques to investigate surface morphology from nano to microscale. It has become a choice to study the influence of inhibitors on the generation and progress of corrosion at the metal/solution interface<sup>20</sup>. Average roughness (Ra), surface roughness (RMS), and maximum peak-to-valley height were calculated using 2D, 2D cross-sectional profile diagram, and 3D surface pictures. Table 5 gives AFM parameters for zinc in different settings. 2D, 3D, broken line displays AFM pictures and cross-section analysis of a polished zinc metal surface with a Ra value of 421 nm, RMS value of 365 nm, and maximum peak to valley height of 2110nm. 8h After immersion in 0.5 N HCl without an inhibitor, a 2D,3D broken line is visible with an

enhanced Ra value of 708nm, RMS value of 605 nm, and maximum peak to valley height of 2603 nm, suggesting the generation of Fe oxides. The corroded mild steel surface's RMS roughness is 605 nm. Surface microstructure exhibits tiny and massive corrosion deposits (Fig. 6,7,8). 2D, 3D, broken line reveals that mild steel submerged in 0.5 N HCl + 100 mM DMS displays a lowered Ra value of 600 nm, RMS value of 470 nm, and maximum peak to valley height of 2200 nm, indicating the production of a protective coating on the mild steel surface.

The corrosion product didn't settle on mild steel. Optical cross-section research variances confirm these conclusions. A protective coating was placed to mild steel to guard it from corrosive ions. The inhibitor created a layer over the mild steel surface, reducing average roughness to 600 nm<sup>21</sup>.

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

	AFM parameters		
Environment	Rougnness,	Da (nm)	Maximum peak -to - valley Height ,(nm)
Polished mild steel	421	365	2110
Mild steel + 0.5 N HCl		605	2603
Mild steel + 0.5 N HCl + 100 mM dimethyl sulphide	600	470	2200



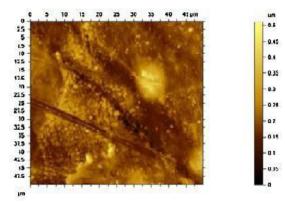
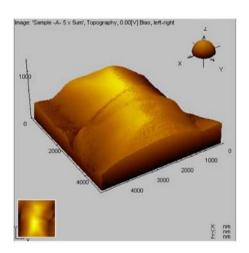


Fig. 7 (c)

Fig. 7: 2D AFM images of the surface of a) Polished mild steel (control) b) Mild steel immersed in 0.5 N HCl (blank) c) Mild steel immersed in 0.5 N HCl containing 100 mM **DMS** 



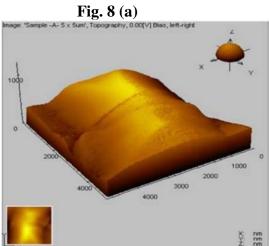


Fig. 8 (c)

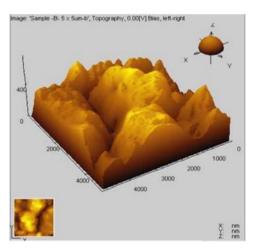


Fig. 8 (b)

Fig. 8: 3D AFM images of the surface of a) Polished mild steel (control) b) Mild steel immersed in 0.5 N HCl(blank) c) Mild steel immersed in 0.5 N HCl containing 100 mM

#### **DMS**

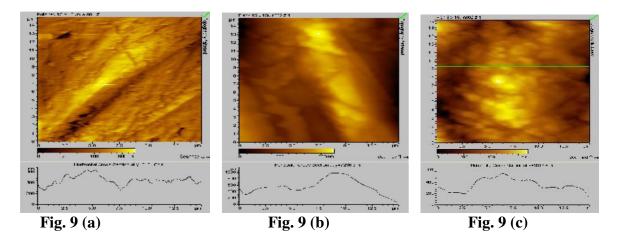


Fig. 9: The cross-sectional profiles correspond to the broken lines in AFM images of the surface of a) Polished mild steel (control) b) Mild steel immersed in 0.5 N HCl (blank) c) Mild steel immersed in 0.5 N HCl containing 100 mM DMS.

#### Corrosion inhibition mechanism

Organic inhibitor molecules block by adsorbing on mild steel. The adsorption process is influenced by the inhibitors' chemical structure, the zinc's charged surface, and the inhibitor's charge distribution. Complex inhibitor adsorption and inhibition prevent a single adsorption mode on mild steel. Organic inhibitor compounds may be adsorbed on mild steel by: Electrostatic reaction between charged molecules and zinc metal. Molecule electron pairs interact with mild steel 22,23.

The inhibitory efficiency may be due to sulphur- and alkyl-rich electrons. It might be an alkyl or protonated molecule in acid solutions (cation). Alkyl groups adsorb on mild steel in one or more ways. Electrostatic interactions of protonated alkyl groups with adsorbed chloride ions, (ii) donor-acceptor interactions between aliphatic compound - electrons and surface mild steel d orbitals, and (iii) interactions between unshared aliphatic electron pairs and zinc d orbitals. Alkyl groups may be adsorbed on iron by chemisorption, which involves displacing water molecules from mild steel and sharing electrons with Fe. Donor-acceptor interactions between -electrons of the aliphatic chemical and Fe's unoccupied d-orbital allow inhibitor molecules to adsorb on mild steel. 100mM DMS has a 69.9% IE in controlling mild steel corrosion in 0.5 N HCl, according to the weight loss data. Polarization investigations show that this formulation inhibits anodization. AC impedance spectra demonstrate a protective coating on mild steel. FTIR spectra show that the protective coating is Fe<sup>2+</sup>-DMS. To demonstrate these subtleties, consider<sup>24</sup>. 0.5 N HCl and DMS solution. ionised DMS Immersing mild steel in this solution converts ionised DMS to a more stable Fe2+- DMS complex<sup>25</sup>. The protective coating is Fe2+-DMS. FTIR confirms this. EDAX and SEM micrographs confirm a protective layer on zinc. AFM validates mild steel's roughness and smoothness.

#### **CONCLUSION**

It has been shown that dimethyl sulphide mitigates the corrosive effects of 0.5N HCl on mild steel. To lessen the effects of 0.5N HCl on mild steel, dimethyl sulphide is added to the solution. Increasing the concentration of the inhibitor has a positive effect on the inhibition process. The best inhibitory concentration is 100 mM. Using the mass loss method and electrochemical experiments, it is shown that the compound inhibits mild steel in 0.5N HCl. The gasometric method reveals an inhibitory efficacy of 71.7%, whereas the weight loss strategy only exhibits 69.9%. The corrosion parameters Icorr, Ecorr, ba, bc, Rct, and Cdl were calculated using electrochemical tests. Experiments with potentiodynamic polarization shown that dimethyl sulphide adsorbs into 0.5N HCl, suggesting it may block anodic reactions and processes on the surface of mild steel. The findings for Icorr, Cdl, and Rt all reduced as the adsorbed layer became thicker, demonstrating that the inhibitor was, indeed, inhibitive. The electrochemical impedance measurements demonstrate that the double-layer capacitance (Cdl) and corrosion current are reduced with increasing adsorbed layer thickness (Icorr). Fe<sup>2+</sup>-DMS serves as an anticorrosion coating. As FTIR tests show, this is really the case. Micrographs taken with an EDAX and SEM demonstrate the presence of a protective layer on the mild steel surface. Roughness and smoothness of mild steel are confirmed by AFM.

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