

## **Sodium Molybdate –Zn<sup>2+</sup> System as Corrosion Inhibitor for AISI 410 Stainless Steel in Sea Water**

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**Abstract:** *The inhibition efficiency of Sodium Molybdate (SM) in controlling corrosion of stainless steel 410 in sea water have been studied in the presence and absence of Zn<sup>2+</sup> has been evaluated by weight loss, Polarization, FTIR and SEM. SM alone shows good inhibition efficiency. Addition of Zn<sup>2+</sup> ions to the system increases the inhibition efficiency. A synergistic effect existing between SM and Zn<sup>2+</sup> ions Polarization study reveals that this formulation controls both anodic and cathodic reaction. The surface film was analyzed by FTIR. The surface morphology and roughness of the metal surface have been analyzed by atomic force microscopy (AFM) and scanning electron microscopy (SEM).*

**Keywords:** *Corrosion inhibition, Atomic force microscopy (AFM), Fourier Transform infrared spectroscopy, stainless steel, synergistic effect, Sodium Molybdate (SM).*

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### **1. INTRODUCTION**

AISI 410 stainless steel is one of the most popular steels amongst the 400 series martensitic stainless steels used in corrosion resistant and high strength applications [1-2]. 12 % Cr martensitic stainless steels like the 410 steel have found wide usage in gas and oil production applications, mine ladder rungs, because of their high corrosion resistance in CO<sub>2</sub> containing environments [3-4]. Many organic, inorganic and heterocyclic compounds containing hetero atoms like N, O, S and P have been proved to be an effective inhibitors for the corrosion of stainless steel in acid media [5]. Sodium Molybdate (SM) finds effective inhibition in controlling corrosion of stainless steel in various aqueous environment [6-7]. Sea water is normally used in storage reservoirs, cooling systems and water transporting pipe lines for injection system etc, exhibits corrosion problem using inhibitors is an important method for protecting materials against corrosion [8, 9, 10]. The aim of the present study was to investigate synergistic corrosion inhibition for the SM and Zn<sup>2+</sup> combination to stainless steel 410 in sea water collected from Bay of Bengal at Cuddalore port, Tamilnadu, India. The inhibition efficiency (IE) was calculated using weight loss method and polarization study. The protective film formed on the metal surface characterized using surface morphological studies such as Fourier Transform Infrared spectra (FTIR), scanning electron microscopy (SEM) and atomic force microscopy (AFM).

### **2. METHODS**

#### **2.1. Preparation of Specimens**

AISI 410 Stainless steel specimens (0.095 % C, 0.001 % P, 6.03.4 % Mn, 0.340 % Si, 13 % Cr, 0.710 % Ni and the rest iron) of dimensions 1.0 cm x 4.0 cm x 0.2 cm were polished to a mirror finish and degreased with trichloroethylene.

#### **2.2. Weight-Loss Method**

Stain Stainless steel specimens in triplicate were immersed in 100 ml of the solutions containing various concentrations of the inhibitor in the presence and absence of Zn<sup>2+</sup> for seven days. The

weight of the specimens before and after immersion was determined using a Shimadzu balance, model AY62. The corrosion products were cleansed with Clarke's solution [11]. The inhibition efficiency (IE) was then calculated using the equation (1)

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

Where  $W_1$ ,  $W_2$  are corrosion rate in the absence and presence of the inhibitor respectively.

The corrosion rate (CR) was calculated using the equation (2).

$$CR = \text{Weight loss in mg} / (0.11 \text{ dm}^3 \times \text{Immersion period}) \quad (2)$$

### **2.3. Synergism Parameter (SI)**

Synergism parameters ( $S_i$ ) are used to know the synergistic effect existing between two inhibitors [12-15]. Synergism parameter (SI) can be calculated using the following relationship (3).

$$SI = (1 - \theta_{1+2}) / (1 - \theta'_{1+2}) \quad (3)$$

Where

$$\theta_{1+2} = \theta_1 + \theta_2 - \theta_1 \cdot \theta_2,$$

$$\theta_1 = \text{Surface coverage by sodium molybdate}$$

$$\theta_2 = \text{Surface coverage by Zn}^{2+}$$

$$\theta'_{1+2} = \text{Surface coverage by both sodium molybdate and Zn}^{2+}$$

$$\theta = \text{Surface coverage} = IE \% / 100$$

### **2.4. Polarization Study**

Polarization studies were carried out in an H & CH electrochemical workstation impedance analyser, model CHI 660A. A three-electrode cell assembly was used. The working electrode was stainless steel. A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode.

### **2.5. Surface Examination Study**

The stainless steel specimens were immersed in various test solutions for a period of seven days. After seven days, the specimens were taken out and dried. The nature of the film formed on the surface of the metal specimens was analyzed for surface analysis technique by FTIR spectra and fluorescence spectra.

#### *2.5.1. FTIR Spectra*

The film formed on the metal surface was carefully removed and mixed thoroughly with KBr. The FTIR spectra were recorded in a Perkin Elmer 1600 spectrophotometer.

#### *2.5.2. Atomic Force Microscopy (AFM)*

Atomic Force Microscope (AFM) is an exciting new technique that allows surface to be imaged at higher resolutions and accuracies than ever before. The microscope used for the present study was PicoSPM I Molecular Imaging, USA make. Polished specimens prior to the initiation of all corrosion experiments were examined through an optical microscope to find out any surface defects such as pits or noticeable irregularities like cracks, etc. Only those specimens, which had a smooth pit-free surface, were subjected for AFM examination. The protective films formed on the stainless steel specimens after immersion in the inhibitor systems for different time durations were examined for a scanned area of  $30 \times 30 \mu\text{m}^2$  and  $15 \times 15 \mu\text{m}^2$ . The two-dimensional and three-dimensional topography of surface films gave various roughness parameters of the film.

#### *2.5.3. Scanning Electron Microscopic Studies (SEM)*

The stainless steel specimen was immersed in blank and in the inhibitor solution. After one day it 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 stainless steel were examined by using Hitachi S-3000 H computer controlled scanning electron microscope.

### 3. RESULTS AND DISCUSSION

#### 3.1. Analysis of Result of Weight Loss Method

Corrosion rates (CR) of stainless steel 410 immersed in sea water in the absence and presence of inhibitor (Sodium molybdate) are given in table 1. It is observed that sodium molybdate shows some inhibition efficiency. 50 ppm of sodium molybdate has 27 percent IE. As the concentration of sodium molybdate increases, the IE increases. This is due to the fact that as the concentration of sodium molybdate increases the protective film formed on the surface controls the anodic reaction and protects the metal. When the concentration of sodium molybdate increases the IE increases. Similarly for a given concentration of sodium molybdate the IE increases as the concentration of Zn<sup>2+</sup> increases. It is also observed that a synergistic effect exists between sodium molybdate and Zn<sup>2+</sup>. For example 250 ppm of SM has 55 percent IE; 50 ppm of Zn<sup>2+</sup> alone has 51 percent IE. But the combination of 250 ppm of SM and 50 ppm of Zn<sup>2+</sup> has a high IE, namely 96 percent.

In the presence of Zn<sup>2+</sup> more amount of sodium molybdate is transported towards the metal surface. On the metal surface Fe- molybdate complex is formed on the anodic sites of the metal surface. Thus the anodic reaction is controlled. The cathodic reaction is the generation of OH<sup>-</sup>, which is controlled by the formation of Zn(OH)<sub>2</sub> on the cathodic sites of the metal surface. Thus the anodic reaction and cathodic reaction are controlled effectively. This accounts for the synergistic effect existing between Zn<sup>2+</sup> and Sodium molybdate.[16-18].

**Table 1.** Inhibition efficiencies (IE) and Corrosion rates (CR) obtained from SM-Zn<sup>2+</sup> system, when stainless steel is immersed in sea water.

Inhibitor system SM- Zn<sup>2+</sup>

Immersion period: 7 days

Na <sub>2</sub> MoO <sub>4</sub> ppm	Zn <sup>2+</sup> (0 ppm)		Zn <sup>2+</sup> (25 ppm)		Zn <sup>2+</sup> (50 ppm)	
	IE %	CR (mdd)	IE %	CR (mdd)	IE %	CR (mdd)
50	27	4.93	39	4.12	51	3.31
100	34	4.46	42	3.92	59	2.77
150	42	3.92	49	3.44	71	1.96
200	49	3.44	54	3.11	82	1.22
250	55	3.04	62	2.57	96	0.27

#### 3.2. Synergism Parameters (SI)

The synergism parameters of Sodium molybdate – Zn<sup>2+</sup> system are given in Table 2. For different concentrations of inhibitors. S<sub>I</sub> approaches 1 when no interaction between the inhibitor compounds exists. When SI > 1, it points to synergistic effects. In the case of S<sub>I</sub> < 1, it is an indication that the synergistic effect is not significant. From table 2, it is observed that value of synergism parameters (S<sub>I</sub>) calculated from surface coverage were found to be one and above. This indicates that the synergistic effect exists between sodium molybdate and Zn<sup>2+</sup>. Thus, the enhancement of the inhibition efficiency caused by the addition of Zn<sup>2+</sup> ions to sodium molybdate is due to the synergistic effect.

**Table 2.** Inhibition efficiencies and synergism parameters for various concentrations of Sodium molybdate– Zn<sup>2+</sup> (50 ppm) system, when stainless steel is immersed in sea water.

Inhibitor system SM- Zn<sup>2+</sup> (0 ppm)

Immersion period: 7 days

SM ppm	IE%	Θ 1	Zn <sup>2+</sup> ppm	IE %	Θ2	Combined IE%	Θ' 1+2	S <sub>I</sub>
50	27	0.27	50	25	0.25	51	0.51	1.12
100	34	0.34	50	25	0.25	59	0.59	1.21
150	42	0.42	50	25	0.25	71	0.71	1.50
200	49	0.49	50	25	0.25	82	0.82	2.13
250	55	0.55	50	25	0.25	96	0.96	8.44

### 3.3. Analysis of Potentiodynamic Polarization Study

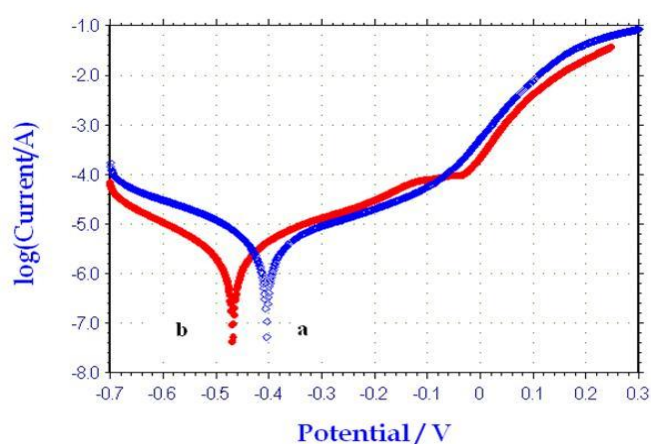
Polarization study has been used to study the formation of protective film formed on the metal surface during corrosion inhibition process. Protective film is formed on the metal surface, the linear polarization resistance value (LPR) increases and the corrosion current value ( $I_{corr}$ ) decreases. The potentiodynamic polarization curves of stainless steel in sea water in the absence and presence of inhibitors are shown in Fig .1. The corrosion parameters namely corrosion potential ( $E_{corr}$ ), tafel slopes  $b_c$ ,  $b_a$  linear polarization resistance (LPR) and corrosion current ( $I_{corr}$ ) are given in the Table 3.

When stainless steel is immersed in sea water the corrosion potential is  $-405$  mV vs SCE .When sodium molybdate (250 ppm) and  $Zn^{2+}$  (50 ppm ) are added to the above system the corrosion potential shifted to  $-468$  mV vs SCE. The LPR value increases from  $9.57 \times 10^3$  ohm  $cm^2$  to  $15.95 \times 10^3$  ohm  $cm^2$ . The corrosion current decreases from  $4.48 \times 10^{-6}$  A  $cm^{-2}$  to  $2.231 \times 10^{-6}$  A  $cm^{-2}$ . This indicates the surface film formed on the protective film on the metal surface. This film controls the anodic reaction of metal dissolution by forming Fe-Molybdate complex on the anodic site of the metal surface.

**Table 3.** Corrosion parameters of stainless steel immersed in sea water in the presence and absence of inhibitor system obtained from potentiodynamic polarization study

System	$E_{corr}$ mV vs SCE	$b_a$ mv/decade	$b_c$ mV/decade	LPR ohm $cm^2$	$I_{corr}$ A/ $cm^2$
Sea water	-405	227	174	$9.57 \times 10^3$	$4.48 \times 10^{-6}$
Seawater + SM (250 ppm) + $Zn^{2+}$ (50 ppm)	-468	175	164	$15.95 \times 10^3$	$2.231 \times 10^{-6}$

It is observed that the corrosion potential shifted to more negative site. This suggests that the cathodic reaction controlled predominantly. The anodic reaction is controlled by the transmission of  $Fe^{2+}$ - Molybdate complex on the anodic sites of the metal surface. The cathodic reaction controlled by the formation of  $Zn(OH)_2$  on the cathodic sites of the metal surface. It is observed from the figure 1, the protective film is stable up to a particular potential. Afterwards the film is broken due to the attack of corrosive ions such as  $Cl^-$  present in the sea water. Hence the corrosion current increases abruptly after this potential. It is also observed that break down potential increases in presence of inhibitor.[19,20]



**Fig .1** Polarization curves of stainless steel immersed in various test solutions (a) sea water (blank) (b) sea water + SM (250 ppm) +  $Zn^{2+}$  (50 ppm)

### 3.4. Analysis of FTIR spectra

FTIR spectra have been used to analyze the protective film formed on the metal surface. The FTIR spectrum (KBr) of pure sodium molybdate is shown in Fig 2 .The  $Na_2MoO_4$  - stretching frequency of sodium molybdate appears at  $906$   $cm^{-1}$

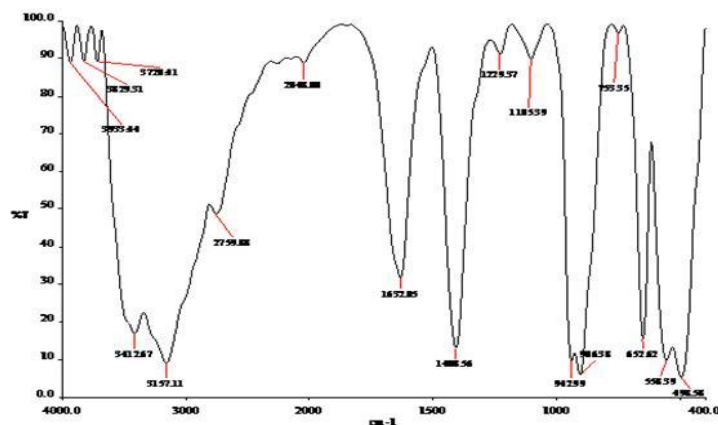


Fig 2. FTIR spectra of Sodium molybdate

The FTIR spectrum of the film formed on the metal surface after immersion in the solution containing 250 ppm of SM and 50 ppm of Zn<sup>2+</sup> is shown in the figure 3. The SM- stretching frequency has shifted from 964 cm<sup>-1</sup> to 1064 cm<sup>-1</sup>. This observation suggests that sodium molybdate has coordinated with Fe<sup>-2+</sup> through the oxygen atom of the molybdate group. The peak at 1372 cm<sup>-1</sup> and 671 cm<sup>-1</sup> corresponds to Zn-O stretching. The peak at 3426 cm<sup>-1</sup> is due to OH stretching. This confirms that Zn (OH)<sub>2</sub> is formed on the cathodic sites of metal surface. Thus the FTIR spectral study lead to the conclusion that the protective film consists of Fe<sup>2+</sup> Molybdate complex and Zn (OH)<sub>2</sub> [21-23].

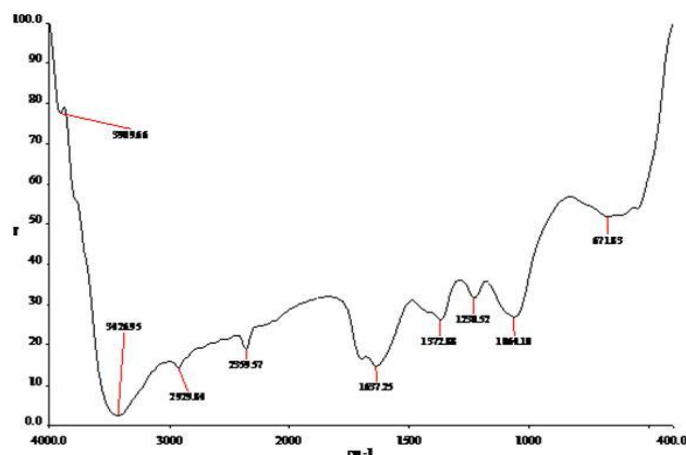
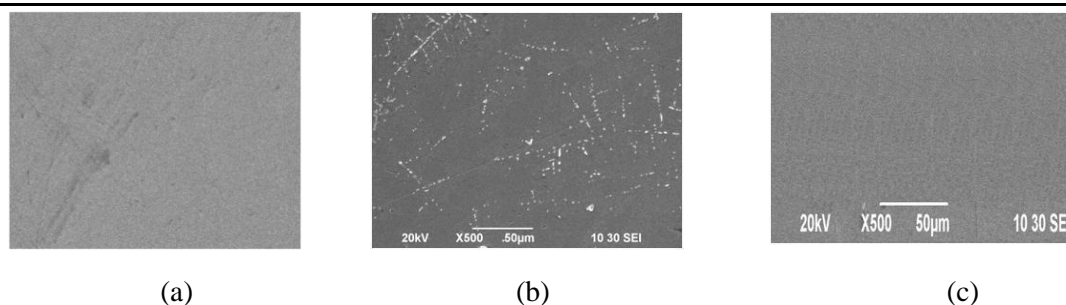


Fig 3. Film formed on stainless steel after immersion in aqueous solution containing sodium molybdate (250 ppm) + Zn<sup>2+</sup> (50 ppm) Complex

### 3.5. SEM Analysis of Metal Surface

SEM provides pictorial representation of the surface. To understand the nature of the surface film in the absence and presence of inhibitors and extent of corrosion of stainless steel, the SEM micrographs of the surface are examined. The SEM images of x 500 magnification of stainless steel specimen immersed in sea water for 7 days in the absence and presence of inhibitor system are shown in Fig 4 (a), (b), (c) respectively.

The SEM micrographs of polished stainless steel surface (control) in Fig 4 (a) shows the smooth surface of the metal. This shows the absence of any corrosion products or inhibitor complex formed on the metal surface. The SEM micrographs of stainless steel surface immersed in sea water Fig 4 (b) shows the roughness of the metal surface which indicates the corroded area of stainless steel in sea water. However Fig 4(c) indicates the rate of corrosion is suppressed, as can be seen from the decrease of corroded areas. The metal surface almost free from corrosion due to the formation of insoluble complex on the surface of the metal. In the presence of SM and Zn<sup>2+</sup>, the surface is covered by thin layer of inhibitors, which effectively controls the dissolution of stainless steel. [12, 19, 20].



**Fig 4.** SEM micrographs of Stainless steel (magnification  $\times 500$ ) immersed in (a) sea water control ; ( b) stainless steel immersed in sea water (Blank); (c) stainless steel immersed in sea water + SM (250 ppm) + 50 ppm of  $Zn^{2+}$

### 3.6. Atomic Force Microscopy

Atomic Force microscopy is a powerful technique for the gathering of roughness statistics from a variety of surfaces [24]. AFM is becoming an accepted method of roughness investigation [25, 26, 27]. All atomic force microscopy images were obtained in a VECCO lab incorporation AFM instrument operating in tapping mode in air. The scan size of all the AFM images are  $05 \mu m \times 05 \mu m$  areas at a scan rate of  $6.685 \mu m/second$ . The three dimensional (3D) AFM morphologies and the AFM cross –sectional profile for polished stainless steel surface (reference sample), stainless steel immersed in sea water (blank) ,and stainless steel surface immersed in sea water containing the formation of SM 250 ppm and 50 ppm of  $Zn^{2+}$  are shown in the figure 5 (a,b),(c,d) and (e,f.) respectively.

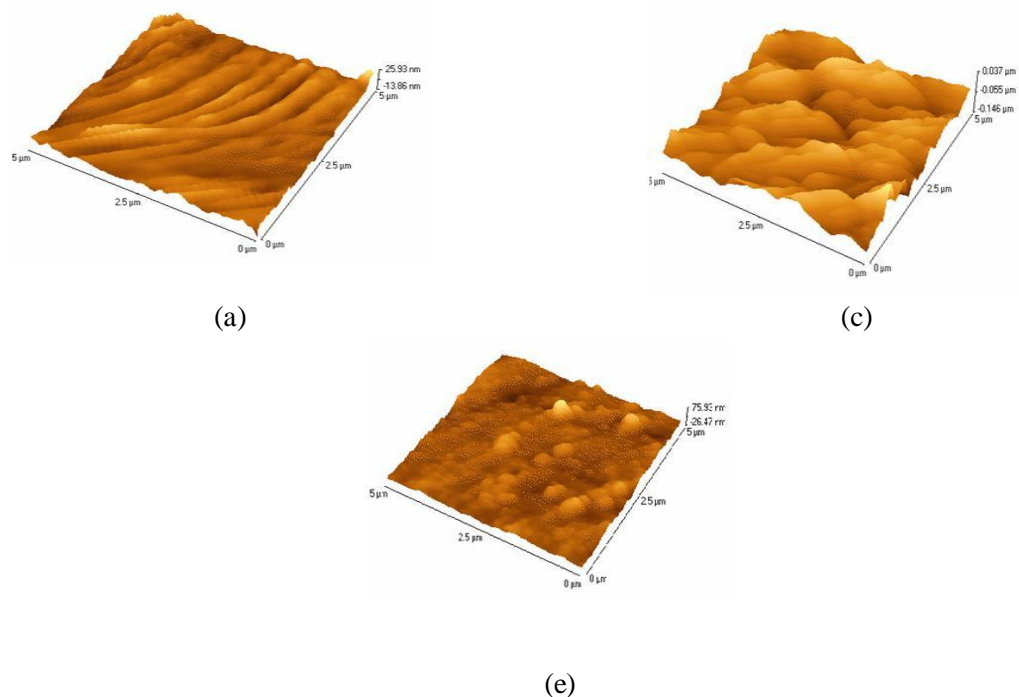
### 3.7. Root Mean Square Roughness, Average Roughness and Peak to Valley Value

AFM image analysis was performed to obtain the average roughness,  $R_a$  (the average deviation of all points roughness profile from a mean line over the evaluation length), root –mean – square roughness,  $R_q$  (the average of the measured height deviations taken within the evaluation length and measured from the mean line) and the maximum peak to valley (P-V) height values (largest single peak-to-valley height in five adjoining sampling heights)[28] . $R_q$  is much more sensitive than  $R_a$  to large and small height deviations from the mean

**Table 4.** AFM data for stainless steel surface immersed in inhibited and uninhibited environment

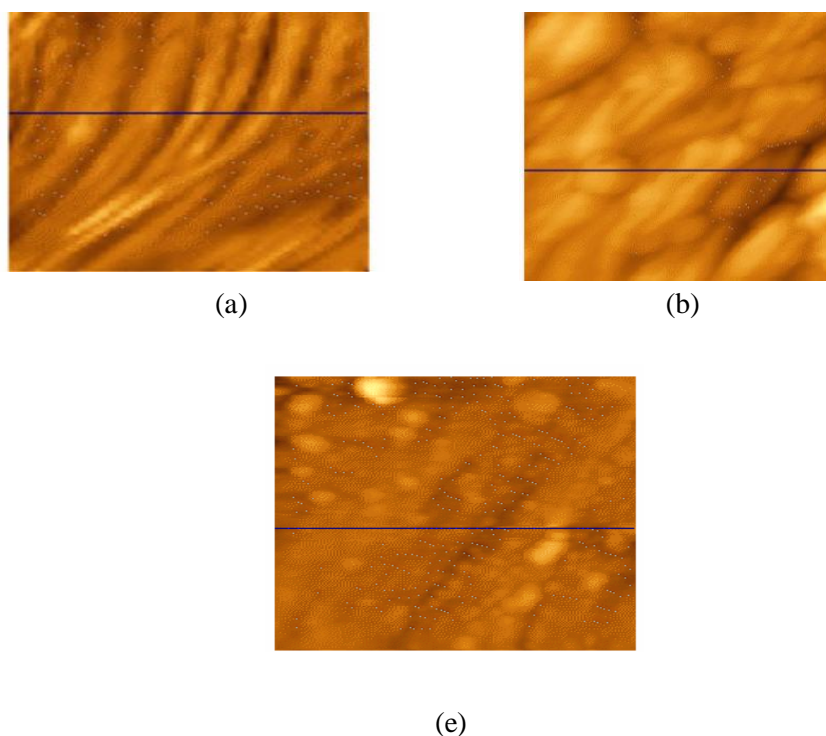
Samples	RMS( $R_q$ ) Roughness (nm)	Average ( $R_a$ ) Roughness (nm)	Maximum peak to valley height (nm)
Polished stainless steel (Control)	4.1	3.3	39.78
Stainless steel immersed in sea water (blank)	31	24	215.3
Stainless steel immersed in sea water containing 250 ppm SM and 50 ppm $Zn^{2+}$ .	6.8	9.6	102.4

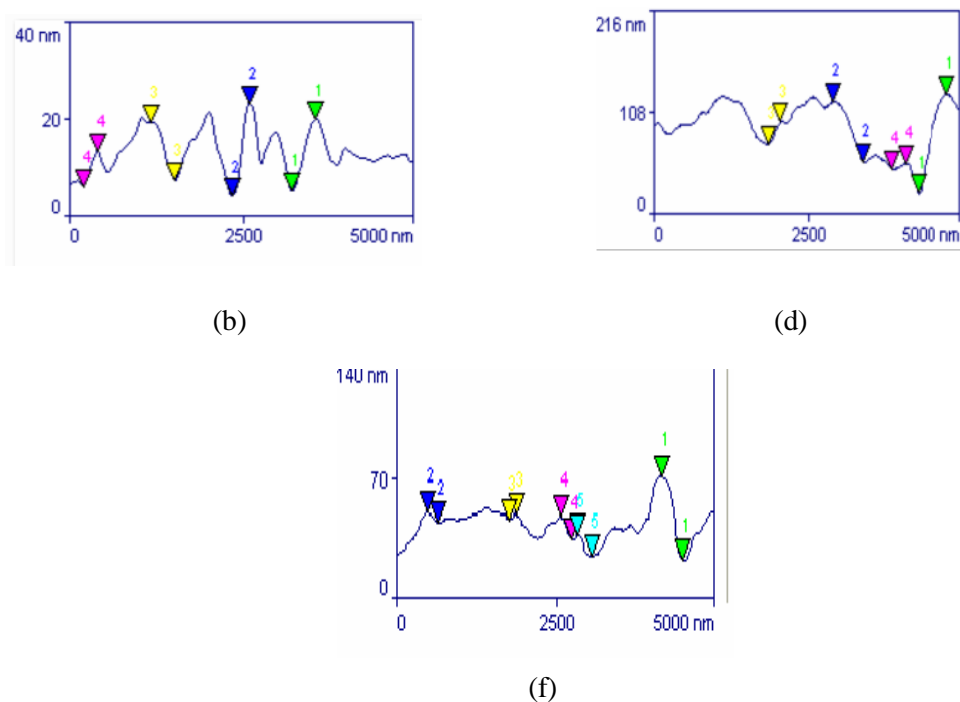
Table 4 is a summary of the average roughness ( $R_a$ ), rms roughness ( $R_q$ ), maximum peak to valley height (P-V) value for stainless steel surface immersed in different environments. The value of  $R_q$ ,  $R_a$  and P-V height for the polished stainless steel surface (reference sample) are 4.1 nm 3.3 nm and 39.78 nm respectively. This shows that the surface is more homogenous, with some places where the height is lower than the average depth [29]. Fig.5 (a, b) displays the non-corroded metal surface. From the Fig 5 (c, d), the slight roughness observed on the polished stainless steel surface is due to atmospheric corrosion. The rms roughness, average roughness and P-V height values for the stainless steel surface immersed in sea water are 31 nm, 24 nm and 215.3 nm respectively. These values suggest that stainless steel surface immersed in sea water has a greater surface roughness than the polished metal surface, indicating that the unprotected stainless steel surface is rougher and were due to the corrosion of stainless steel in sea water environment.



**Fig 5.** Three dimensional images of surface of (a) polished stainless steel (control); (b) Stainless steel immersed in sea water (blank); (c) Stainless steel immersed in sea water containing 250 ppm SM and 50 ppm Zn<sup>2+</sup>

The formulation consisting of 250 ppm SM and 50 ppm of Zn<sup>2+</sup> in sea water shows Rq value of 6.8 nm reduced from 31 nm and the average roughness is significantly reduced to 9.6 nm when compared with 24 nm for stainless steel surface immersed in sea water. The maximum peak to valley height was also reduced to 102.4 nm. These parameters confirm that the surface appears smoother. Fig 5 (e,f) shows the smoothness of the surface is due to the formation of a protective film of Fe<sup>2+</sup> - Molybdate complex and Zn (OH)<sub>2</sub> on the metal surface thereby inhibiting the corrosion of stainless steel. The above parameters are also somewhat greater than the AFM data of polished metal surface, which confirms the formation of film on the metal surface, which is protective in nature.





**Fig 5** The cross-sectional profiles (shown as broken lines) corresponding to the AFM images of surface of (b)Polished stainless steel (control); (d)stainless steel immersed in sea water (blank); (f)stainless steel immersed in sea water containing 250 ppm SM and 50 ppm Zn<sup>2+</sup>.

#### 4. MECHANISM OF CORROSION INHIBITION

Analysis of the results of weight-loss method reveals that the formulation consisting of sea water, 250 ppm of SM and 50 ppm of Zn<sup>2+</sup> offers an IE of 98%. Results of polarization study suggest that the formulation functions as cathodic inhibitor. FTIR spectra reveal that the protective film consists of Fe<sup>2+</sup>-Molybdate complex and Zn (OH)<sub>2</sub>. In order to explain all these observations in a holistic way, the following mechanism of corrosion inhibition is proposed.

- When stainless steel specimen is immersed in an aqueous solution,  
The anodic reaction is  $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$   
Cathodic reaction is  $2\text{H}_2\text{O} + \text{O}_2 + 4\text{e}^- \rightarrow 4\text{OH}^-$
- When the system containing 250 ppm of STPP and 50 ppm of Zn<sup>2+</sup> is prepared, there is formation of Zn<sup>2+</sup>-Molybdate complex.  
 $\text{Zn}^{2+} + \text{SM} \rightarrow \text{Zn}^{2+}\text{-SM}$
- When stainless steel is immersed in the solution, the Zn<sup>2+</sup>-SM diffuses from the bulk of the solution to the metal surface.
- On the surface of metal, Zn<sup>2+</sup>-SM complex is converted into Fe<sup>2+</sup> - SM complex at the local regions. The stability of Fe<sup>2+</sup>-SM complex is higher than the corresponding zinc complex.  
 $\text{Zn}^{2+}\text{-SM} + \text{Fe}^{2+} \rightarrow \text{Fe}^{2+}\text{-SM} + \text{Zn}^{2+}$
- The released Zn<sup>2+</sup> ions combine with OH<sup>-</sup> ions to form Zn (OH)<sub>2</sub> on the cathodic sites.
- $\text{Zn}^{2+} + 2\text{OH}^- \rightarrow \text{Zn}(\text{OH})_2 \downarrow$

Thus the protective film consists of Fe<sup>2+</sup> - SM complex and Zn (OH)<sub>2</sub>.

#### 5. CONCLUSION

- The inhibition efficiency (IE) of SM in controlling corrosion of stainless steel in sea water in the presence of Zn<sup>2+</sup> has been evaluated by weight loss method.



- The formulation consists of 250 ppm of SM and 50 ppm of Zn<sup>2+</sup> has 96% IE.
- IE of SM increases with the addition of Zn<sup>2+</sup>.
- A synergistic effect exists between SM and Zn<sup>2+</sup>.It has been confirmed by synergism parameter.
- Polarization study reveals that SM-Zn<sup>2+</sup> system controls the cathodic reaction predominantly.
- FTIR spectra reveal that the protective film consists of SM-Fe<sup>2+</sup> complex and Zn(OH)<sub>2</sub>.
- The SEM micrographs and AFM images confirms the formation of protective layer on the metal surface.

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