

Research Article

Surface Protection of Carbon Steel by Hexanesulphonic Acid-Zinc Ion System

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Inhibition of corrosion of carbon steel in dam water by hexanesulphonic acid as its sodium salt $C_6H_{13}SO_3Na$ (SHXS) in the absence and presence of a bivalent cation zinc ion (Zn^{2+}) has been investigated using weight loss method. Results of weight loss method indicate that inhibition efficiency (IE) increased with increase of inhibitor concentration. Polarization study reveals that SHXS- Zn^{2+} system controls the cathodic reaction predominantly. AC impedance spectra reveal that a protective film is formed on the metal surface. The nature of the metal surface has been analysed by Fourier Transform Infrared Spectroscopy (FTIR) and Atomic Force Microscopy (AFM).

1. Introduction

Corrosion is a naturally occurring phenomenon which deteriorates a metallic material or its properties because of a reaction with its environment. Corrosion can cause dangerous and expensive damage to everything from pipelines, bridges, and public buildings to vehicles, water, and wastewater systems, and even home appliances. It is one of the most serious problems in the oil and gas industry. The use of organic inhibitors is one of the most widely practical methods for protection of metals and alloys against corrosion. The efficiency of an organic compound as a corrosion inhibitor is closely associated with the chemical adsorption [1–4]. Most well-known organic inhibitors contain nitrogen, sulfur, and oxygen atoms due to their ability to form an adsorbed protective film at the metal/media interface. Studies report that the adsorption of organic inhibitors mainly depends on some physicochemical properties of the molecule, related to its functional groups, to the possible steric effects and electronic density of donor atoms: adsorption is supposed also to depend on the possible interaction of P-orbitals of the inhibitor with d-orbitals of the surface atoms, which

induce greater adsorption of the inhibitor molecules onto the surface of carbon steel, leading to the formation of a corrosion protective film [5].

A survey of the available literature reveals that the corrosion inhibition of 2-naphthalenesulfonic acid, 2, 7-naphthalenedisulfonic acid, and 2-naphthol-3, 6-disulfonic acid on Armco-iron electrode in sulfuric acid has been investigated by Vračar and Draži. The inhibition efficiency changes with the number of functional groups substituted on the benzene ring and increases with concentration [6]. The inhibition action of 2-mercaptobenzoxazol, 2-mercapto benzimidazole, N-cetyl pyridinium bromide, and propargyl benzene sulphonate on the corrosion of carbon steel in acid media has also been studied by Prakash et al. The corrosion inhibitors are used to reduce corrosion damage in subsurface equipment in oil well fields. The corrosion inhibition activity was studied by gravimetric and potentiostatic polarization methods in presence of 20% HCl [7]. Manickavasagam et al. have reported the corrosion inhibition of poly(Styrene sulphonic acid) - doped polyaniline on carbon steel in acid media. The polymer acts as an anodic inhibitor. The adsorption of the compound on the metal surface obeys Temkin's

adsorption isotherm [8]. Aliev has described the influence of salts of alkyl phenol sulphonic acid on the corrosion of ST3 steel. The protective effect increases with temperature. The investigated compounds inhibit corrosion of ST3 steel as a result of chemical adsorption [9]. Shakkthivel and Vasudevan have studied the effect of acrylic acid-diphenylamine sulphonic acid copolymer as threshold inhibitor for sulphate and carbonate scales in cooling water systems. The results show that the polymer acts as a very good antiscaling inhibitor both in the carbonate and sulphate brines. Copolymer of acrylic acid-diphenylamine sulphonic acid can be used safely in cooling water systems [10]. Perusal of several literatures reveals that there is no information regarding the use of hexanesulphonic acid (SHXS) in combination with zinc ion (Zn^{2+}) as corrosion inhibitor. This paper focuses on the IE of hexanesulphonic acid as its sodium salt (SHXS) in controlling corrosion of carbon steel immersed in dam water in the absence and presence of Zn^{2+} . The investigation is performed using weight loss method, polarization technique, and AC impedance spectroscopy. The morphology of the protective film was examined by FTIR and AFM techniques.

The medium which is used in the present study is dam water collected from Sothuparai dam in the state of Tamil Nadu, India, constructed across the Vaigai River, the water from which is used in cooling systems by the industries located downstream.

2. Experimental Method

The chemicals used in this study, sodium hexanesulphonate (inhibitor) and $ZnSO_4 \cdot 7H_2O$ (Zn^{2+} ions) coinhibitor, were AR grade.

2.1. Preparation of the Specimen. Carbon steel specimens of size 1.0 cm × 4.0 cm × 0.2 cm, area 10 cm², and chemical composition 0.026% sulphur, 0.06% phosphorous, 0.4% manganese, 0.1% carbon, and the rest iron (density 7.87 gm/cm³) were polished to a mirror finish and degreased with trichloroethylene and used for the weight loss method and surface examination studies.

2.2. Weight Loss Method. Carbon steel specimens were immersed in 100 mL of the medium containing various concentrations of the inhibitor (sodium hexanesulphonate) in the absence and presence of Zn^{2+} for 3 days. The weights of the specimens before and after immersion were determined using a Digital Balance (Model AUY 220 SHIMADZU). The corrosion products were cleaned with Clarke's solution prepared by dissolving 20 gms of Sb_2O_3 and 50 gms of $SnCl_2$ in one litre of Conc. HCl of specific gravity (1.9) [11]. The corrosion IE was then calculated using the equation

$$IE = 100 \left[1 - \left(\frac{W_2}{W_1} \right) \right] \%, \quad (1)$$

where W_1 is the weight loss value in the absence of inhibitor and W_2 is the weight loss value in the presence of inhibitor.

Corrosion rate was calculated using formula [12] millimeter per year

$$(\text{mm/year}) = \frac{87.6W}{DAT}. \quad (2)$$

W = weight loss in milligrams, D = density of specimen in g/cm³, A = area of specimen in cm², and T = exposure time in hours.

2.3. Potentiodynamic Polarization. Polarization studies were carried out in a CHI—electrochemical work station with impedance model 660A. It was provided with iR compensation facility. A three-electrode cell assembly was used. The working electrode was carbon steel. Saturated calomel electrode was the reference electrode. Platinum was the counter electrode. From polarisation study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a , and cathodic = b_c were calculated and linear polarization resistance (LPR) was also calculated. The scan rate (V/S) was 0.01. Hold time at E_f (s) was zero and quiet time was two seconds.

2.4. AC Impedance Spectra. The instrument and cell set-up used for polarization study was used to record AC impedance spectra also. The real part (Z') and imaginary part (Z'') of the cell impedance were measured in ohms at various frequencies. Values of charge transfer resistance (R_t) and the double layer capacitance (C_{dl}) were calculated:

$$R_t = (R_s + R_t) - R_s, \quad (3)$$

where R_s = solution resistance

$$C_{dl} = \frac{1}{2} \pi R_t f_{\text{max}}, \quad (4)$$

where f_{max} = frequency at maximum imaginary impedance.

AC impedance spectra were recorded with initial $E_{(v)}$ = 0, high frequency (Hz) = 1×10^5 , low frequency (Hz) = 1, amplitude (V) = 0.005, and quiet time (s) = 2.

2.5. Surface Examination Study. The carbon steel specimens were immersed in various test solutions for a period of 1 day. After immersion, the specimens were taken out and dried. The nature of the film formed on the surface of the metal specimen was analyzed by various surface analysis techniques.

2.5.1. Fourier Transform Infrared Spectra (FTIR). These spectra were recorded in a Perkin-Elmer-1600 spectrophotometer using KBr pellet. The FTIR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr and making the pellet.

2.5.2. Atomic Force Microscopy Characterization (AFM). The carbon steel specimens immersed in blank and in the inhibitor solution for a period of one day were removed, rinsed with double distilled water, dried, and subjected to

TABLE 1: Water analysis.

Parameters	Result
Appearance	Brownish
Total dissolved solids	100 mol/L
Electrical conductivity	140 μ S/cm
pH	8.25
Total hardness as CaCO ₃	50 mol/L
Calcium	10 mol/L
Magnesium	06 mol/L
Iron	1.2 mol/L
Nitrate	10 mol/L
Chloride	10 mol/L
Sulphate	02 mol/L

TABLE 2: The corrosion inhibition efficiencies and the corresponding corrosion rates (mm/year) of SHXS-Zn²⁺ system.

Inhibitor SHXS (mol/L) $\times 10^{-3}$	Zn ²⁺ (mol/L)					
	0		5.2×10^{-5}		1.04×10^{-4}	
	IE %	CR mm/year	IE %	CR mm/year	IE %	CR mm/year
0	—	0.1127	12	0.0992	21	0.0890
0.27	10	0.1014	26	0.0834	57	0.0642
0.54	13	0.0981	29	0.0801	64	0.0721
0.81	19	0.0913	32	0.0766	71	0.0327
1.08	26	0.0834	36	0.0721	74	0.0293
1.35	31	0.0778	40	0.0676	83	0.0192

the surface examination. Atomic Force Microscope (Veeco diInnova model) was used to observe the samples' surface in tapping mode, using cantilever with linear tips. The scanning area in the images was $5 \mu\text{m} \times 5 \mu\text{m}$ and the scan rate was 0.6 Hz.

3. Results and Discussion

3.1. Analysis of Weight Loss Study. The physicochemical parameters of dam water are given in Table 1.

Inhibition efficiency (IE) and the corrosion rates in millimeter per year (mm/year) of carbon steel immersed in dam water in presence of SHXS-Zn²⁺ system for a period of 3 days are given in Table 2.

It is found that the IE increases as the concentration of SHXS increases. As the concentration of Zn²⁺ increases, IE also increases. A synergistic effect exists between SHXS and Zn²⁺. For example, 1.35×10^{-3} mol/L of SHXS has 31% IE. 1.04×10^{-4} mol/L of Zn²⁺ has 21% IE. However, interestingly the formulation consisting of 1.35×10^{-3} mol/L of SHXS and 1.04×10^{-4} mol/L of Zn²⁺ has 83% IE. That is, the mixture of inhibitors shows better IE than the individual inhibitors. The improvement in the protection efficiency can be attributed to the formation of a complex between Zn²⁺ and SHXS which results from the synergistic effect between the two inhibitors,

TABLE 3: Synergism Parameter (S_I).

SHXS (mol/L) $\times 10^{-3}$	I_1	SHXS-Zn ²⁺ (5.2×10^{-5})		SHXS-Zn ²⁺ (1.04×10^{-4})	
		$I'_{(1+2)}$	S_I	$I'_{(1+2)}$	S_I
0.27	0.10	0.26	1.0703	0.57	1.6535
0.54	0.13	0.29	1.0783	0.64	1.9090
0.81	0.19	0.32	1.0482	0.71	2.2066
1.08	0.26	0.36	1.0175	0.74	2.2485
1.35	0.31	0.40	1.0120	0.83	3.2065

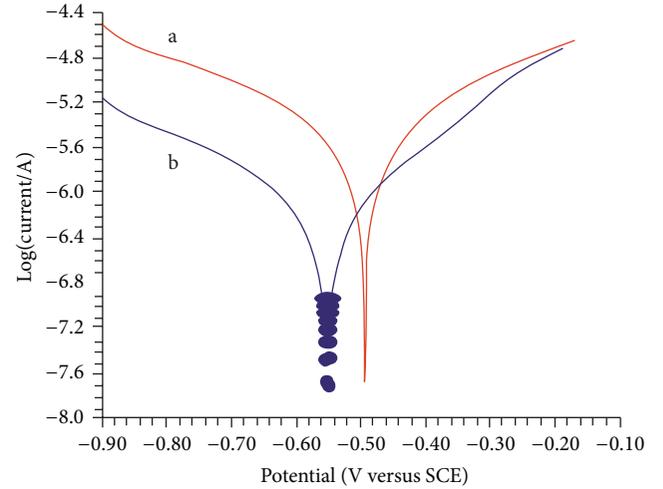


FIGURE 1: Polarisation curves of carbon steel immersed in various test solutions. (a) Dam water. (b) Dam water containing 1.35×10^{-3} mol/L of SHXS and 1.04×10^{-4} mol/L of Zn²⁺.

Zn²⁺ and SHXS. As a result of this complex formation, the inhibitor molecules are readily transported from the bulk to the metal surface [13].

Synergism Parameter (S_I). Synergism parameters are indications of synergistic effect existing between inhibitors [14, 15]:

$$S_I = \frac{1 - I_{1+2}}{1 - I'_1 - I'_2}, \quad (5)$$

where $I_{1+2} = (I_1 + I_2) - (I_1 I_2)$, I_1 = surface coverage of inhibitor (SHXS), I_2 = surface coverage of inhibitor (Zn²⁺), and I'_{1+2} = combined surface coverage of inhibitors (SHXS) and (Zn²⁺)

$$\text{Surface coverage} = \frac{\text{IE}}{100}. \quad (6)$$

I_2 for Zn²⁺ (5.2×10^{-5} mol/L) = 0.12 and I_2 for Zn²⁺ (1.04×10^{-4} mol/L) = 0.21.

The results are given in Table 3.

S_I value is found to be greater than one, indicating the synergistic effect existing between Zn²⁺ of concentrations 5.2×10^{-5} mol/L and 1.04×10^{-4} mol/L with various concentrations of SHXS.

TABLE 4: Corrosion parameters of carbon steel immersed in dam water in the presence and absence of inhibitor obtained by polarization method.

SHXS (mol/L)	Zn ²⁺ (mol/L)	E_{corr} (mV versus SCE)	I_{corr} (A/cm ²)	b_a (mV/dec)	b_c (mV/dec)	LPR (Ω cm ²)
0	0	-494	2.66×10^{-6}	166	203	2.05×10^4
1.35×10^{-3}	1.04×10^{-4}	-572	5.31×10^{-7}	178	157	7.52×10^4

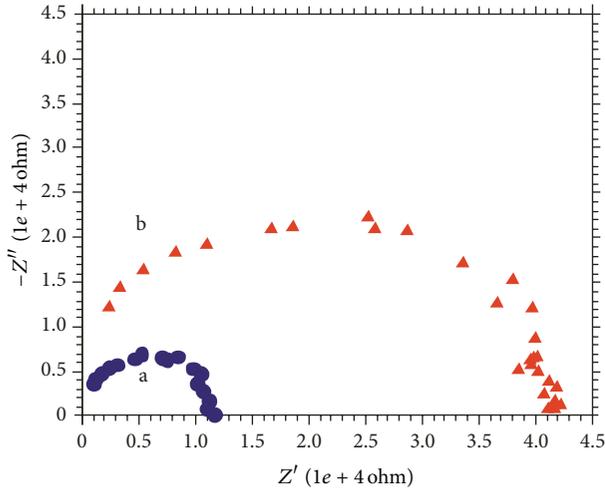


FIGURE 2: AC impedance spectra of carbon steel immersed in various test solutions. (a) Dam water. (b) Dam water containing 1.35×10^{-3} mol/L of SHXS and 1.04×10^{-4} mol/L of Zn²⁺.

3.2. Analysis of Polarization Curves. Figure 1 represents the potentiodynamic polarization curves of carbon steel in dam water in the absence and presence of the inhibitor system. The cathodic branch represents the oxygen reduction reaction, while the anodic branch represents the iron dissolution reaction. The electrochemical parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes (b_a and b_c), and linear polarization resistance (LPR) are given in Table 4.

When carbon steel is immersed in dam water, the corrosion potential is -494 mV versus SCE. The formulation consisting of SHXS (1.35×10^{-3} mol/L)-Zn²⁺ (1.04×10^{-4} mol/L) shifts the corrosion potential to -572 mV versus SCE. It is observed that the corrosion potential is shifted to the cathodic side. It is also observed that the shift in the cathodic slope (from 203 mV/dec to 157 mV/dec) is higher than the shift in the anodic slope (from 166 mV/dec to 178 mV/dec). Hence, it can be said that the inhibitor system predominantly controls the cathodic reaction [16]. The corrosion current value and LPR value for dam water are 2.66×10^{-6} A/cm² and 2.05×10^4 Ω cm².

For the formulation of SHXS (1.35×10^{-3} mol/L)-Zn²⁺ (1.04×10^{-4} mol/L), the corrosion current value has decreased to 5.31×10^{-7} A/cm², and the LPR value has increased to 7.52×10^4 Ω cm². The fact that the LPR value increases with decrease in corrosion current indicates adsorption of the inhibitor on the metal surface to block the active sites and inhibit corrosion and reduce the corrosion rate [17, 18].

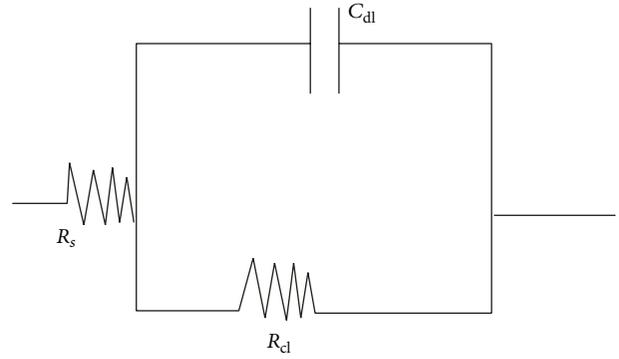


FIGURE 3: Equivalent electrical circuit diagram. R_s = solution resistance. R_{ct} = charge transfer resistance. C_{dl} = double layer capacitance.

3.3. Analysis of AC Impedance Spectra. AC impedance spectra have been used to detect the formation of film on the metal surface. If a protective film is formed, the charge transfer resistance (R_t) increases and double layer capacitance (C_{dl}) value decreases [19, 20]. Nyquist representations of carbon steel in dam water in the absence and presence of the inhibitor system are shown in Figure 2.

It is clear from the plots that the impedance response of carbon steel was significantly changed after addition of the inhibitors. The impedance diagrams obtained have an almost semicircular appearance. This indicates that the corrosion of carbon steel in aqueous solution is mainly controlled by a charge transfer process [21]. The deviation from the perfect semicircle shape (depression) is due to the frequency dispersion of interfacial impedance. This anomalous behavior is generally attributed to the inhomogeneity of the metal surface arising from surface roughness or interfacial phenomena [22]. The AC impedance parameters, namely, charge transfer resistance (R_t) and double layer capacitance (C_{dl}), are given in Table 5.

When carbon steel is immersed in dam water, R_t value is 1.08×10^4 Ω cm² and C_{dl} value is 8.36×10^{-10} F/cm². When SHXS (1.35×10^{-3} mol/L)-Zn²⁺ (1.04×10^{-4} mol/L) are added to dam water, R_t value increases from 1.08×10^4 Ω cm² to 3.93×10^4 Ω cm² and the C_{dl} decreases from 8.36×10^{-10} F/cm² to 2.30×10^{-10} F/cm². This suggests that a protective film is formed on the surface of the metal. Results obtained from impedance measurements can be interpreted in terms of the equivalent circuit of the electrical double-layer presented in Figure 3, which was used previously to model other interaction involving metal/solutions interface [23, 24].

TABLE 5: Impedance parameters of carbon steel in dam water in the presence and absence of inhibitor obtained by AC impedance method.

SHXS (mol/L)	Zn ²⁺ (mol/L)	R _t Ω cm ²	C _{dl} F/cm ²
0	0	1.08 × 10 ⁴	8.36 × 10 ⁻¹⁰
1.35 × 10 ⁻³	1.04 × 10 ⁻⁴	3.93 × 10 ⁴	2.30 × 10 ⁻¹⁰

This type of circuit is known as Randle circuit. Many authors also suggested Randle circuit for similar graphs [25, 26].

3.4. Analysis of FTIR Spectra. Earlier researchers have confirmed that FTIR spectrometer is a powerful instrument that can be used to determine the type of bonding for organic inhibitors adsorbed on the metal surface [27]. FTIR spectra have been used to analyze the protective film formed on metal surface. FTIR spectrum of pure SHXS is given in Figure 4(a). The FTIR spectrum of the film formed on the metal surface after immersion in the dam water for one day containing 250 ppm of SHXS and 30 ppm of Zn²⁺ is shown in Figure 4(b). The S=O stretching frequency has decreased from 1193 cm⁻¹ to 1120 cm⁻¹. This indicates that the oxygen atom of S=O group has coordinated with Fe²⁺ formed on the metal surface resulting in the formation of Fe²⁺-SHXS complex on the anodic sites of the metal surface. The peak at 1386 cm⁻¹ is due to Zn-O stretching. The stretching frequency due to -OH appears at 3463 cm⁻¹. So, it is concluded that Zn(OH)₂ is formed on cathodic sites of the metal surface [28].

3.5. Atomic Force Microscopy Characterization. AFM is a powerful technique to investigate the surface morphology at nano- to microscale and has become a new choice to study the influence of inhibitor on the generation and the progress of the corrosion at the metal/solution interface [29–32]. The three-dimensional (3D) AFM morphology and the AFM cross-sectional profile for polished carbon steel surface (reference sample), carbon steel surface immersed in dam water (blank sample), and carbon steel surface immersed in dam water containing SHXS (1.35 × 10⁻³ mol/L)-Zn²⁺ (1.04 × 10⁻⁴ mol/L) are shown in Figures 5 and 6.

3.6. 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) [29]. Table 6 is a summary of (R_q), (R_a), and (P-V) value for carbon steel surface immersed in different environment.

In Figures 5(a) and 6(a) the surface topography of uncorroded metal surface is shown. The value of R_q, R_a, and

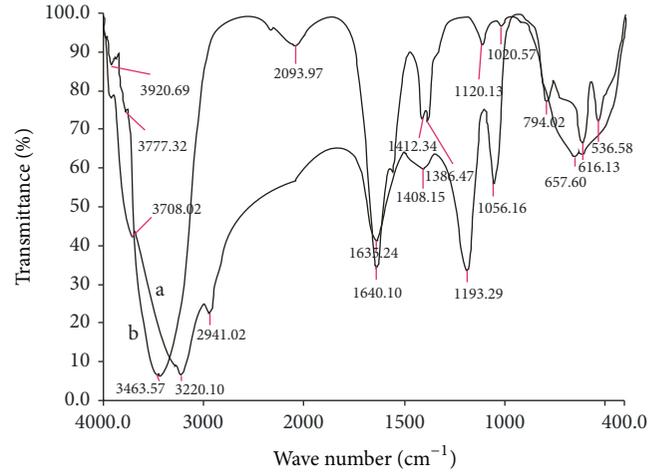


FIGURE 4: (a) FTIR spectrum of pure sodium hexanesulphonate. (b) FTIR spectrum of film formed on metal surface after immersion in dam water containing 1.35 × 10⁻³ mol/L of SHXS and 1.04 × 10⁻⁴ mol/L of Zn²⁺.

P-V height for the polished carbon steel surface (reference sample) is 4.33 nm, 3.41 nm, and 35.28 nm, respectively. The slight roughness observed on the polished carbon steel surface is due to atmospheric corrosion.

Figures 5(b) and 6(b) show the pitted, corroded metal surface in the absence of the inhibitor immersed in dam water. The (R_q), (R_a), and (P-V) height values for the carbon steel surface are 31.9 nm, 24.9 nm, and 420.3 nm, respectively. These data suggest that carbon steel surface immersed in dam water has a greater surface roughness than the polished metal surface, which shows that the unprotected carbon steel surface is rougher and is due to the corrosion of the carbon steel in dam water environment.

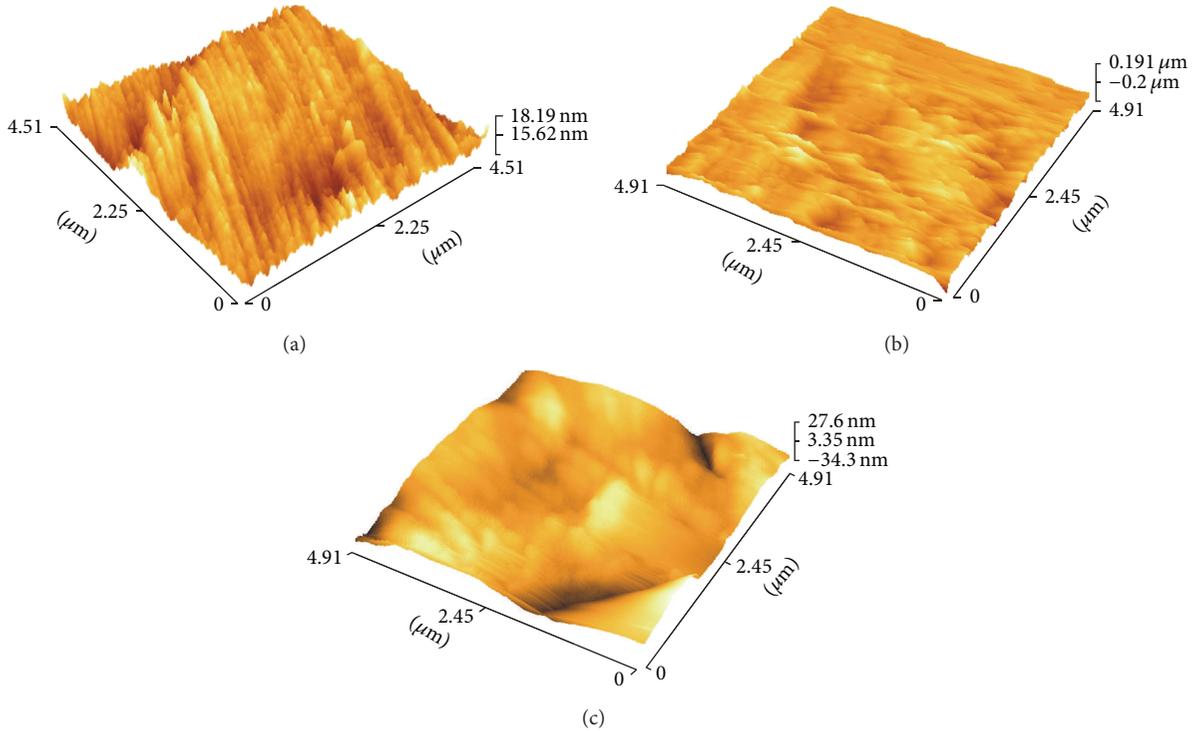
Figures 5(c) and 6(c) show the steel surface after immersion in dam water containing SHXS (1.35 × 10⁻³ mol/L)-Zn²⁺ (1.04 × 10⁻⁴ mol/L). The (R_q), (R_a), and (P-V) height values for the carbon steel surface are 4.38 nm, 3.71 nm, and 27.21 nm, respectively. The (R_q), (R_a), and (P-V) height values are considerably less in the inhibited environment compared to the uninhibited environment. These parameters confirm that the surface is smoother. The smoothness of the surface is due to the formation of a compact protective film of Fe²⁺-SHXS complex and Zn(OH)₂ on the metal surface thereby inhibiting the corrosion of carbon steel [29].

3.7. Mechanism of Corrosion Inhibition. With these discussions, a mechanism may be proposed for the corrosion inhibition of carbon steel immersed in dam water containing SHXS (1.35 × 10⁻³ mol/L)-Zn²⁺ (1.04 × 10⁻⁴ mol/L).

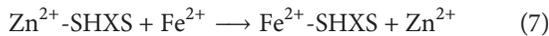
When the formulation consists of SHXS (1.35 × 10⁻³ mol/L)-Zn²⁺ (1.04 × 10⁻⁴ mol/L) in dam water, there is formation of SHXS-Zn²⁺ complex in solution.

TABLE 6: AFM data for carbon steel surface immersed in inhibited and uninhibited environment.

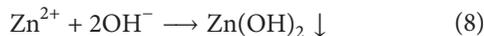
Samples	RMS (R_q) roughness (nm)	Average (R_a) roughness (nm)	Maximum peak-to-valley height (nm)
1. Polished carbon steel	4.33	3.41	35.28
2. Carbon steel immersed in dam water (blank)	31.9	24.9	420.3
3. Carbon steel immersed in dam water + SHXS (1.35×10^{-3} mol/L) + Zn^{2+} (1.04×10^{-4} mol/L)	4.38	3.71	27.21

FIGURE 5: Three-dimensional AFM images of the surface of (a) polished carbon steel (control); (b) carbon steel immersed in dam water (blank); (c) carbon steel immersed in dam water containing 1.35×10^{-3} mol/L of SHXS and 1.04×10^{-4} mol/L of Zn^{2+} .

- (1) When carbon steel is immersed in this solution SHXS- Zn^{2+} complex diffuses from the bulk of the solution towards the metal surface.
- (2) SHXS- Zn^{2+} complex is converted into SHXS- Fe^{2+} complex on the anodic sites of the metal surface with the release of Zn^{2+} ion:



The released Zn^{2+} combines with OH^- to form $Zn(OH)_2$ on the cathodic sites of the metal surface.



- (3) Thus the protective film consists of SHXS- Fe^{2+} complex and $Zn(OH)_2$.

- (4) This accounts for the synergistic effect of SHXS- Zn^{2+} system.

4. Conclusions

The present study leads to the following conclusions.

The inhibition efficiency (IE) of SHXS in controlling corrosion of carbon steel immersed in dam water in the absence and presence of Zn^{2+} has been evaluated by weight loss method. The formulation consisting of 1.35×10^{-3} mol/L of SHXS and 1.04×10^{-4} mol/L of Zn^{2+} has 83% IE. Polarization study reveals that SHXS- Zn^{2+} system controls the cathodic reaction predominantly. AC impedance spectra and AFM study reveal that a compact protective film is formed on the metal surface.

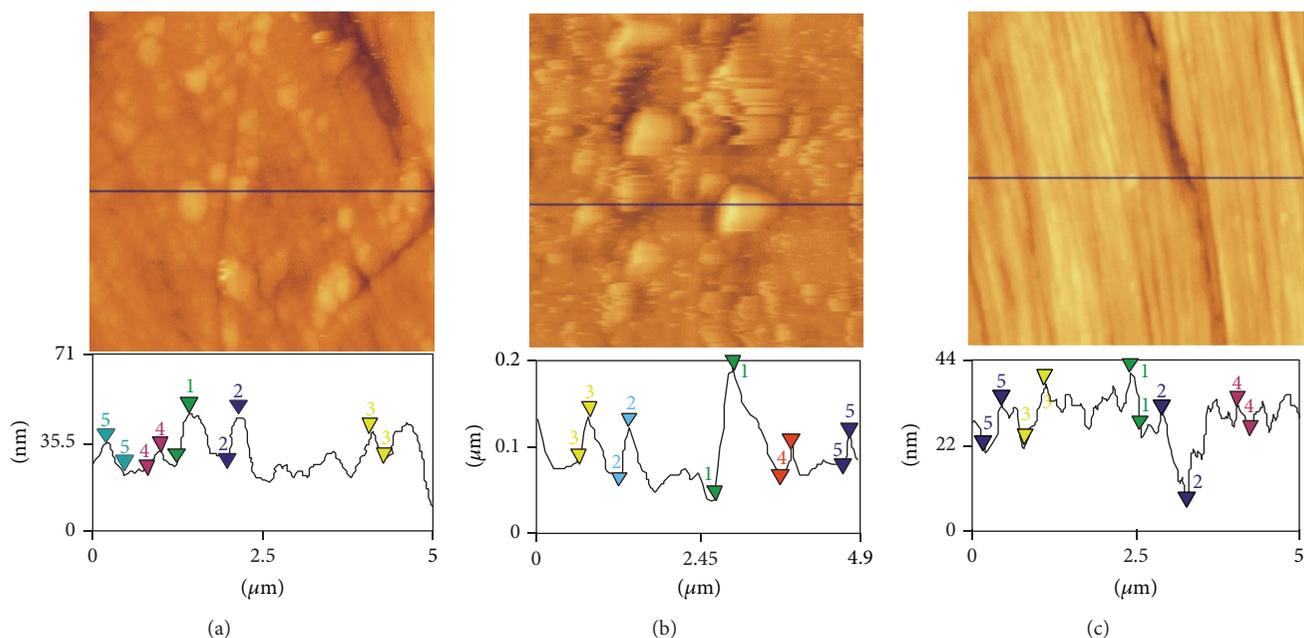


FIGURE 6: AFM cross-sectional images of the surface of (a) polished carbon steel (control); (b) carbon steel immersed in dam water (blank); (c) carbon steel immersed in dam water containing 1.35×10^{-3} mol/L of SHXS and 1.04×10^{-4} mol/L of Zn^{2+} .

Conflict of Interests

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

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