# Corrosion Inhibition by an Ion Pair Reagent-Zn<sup>2+</sup> System

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**Abstract:** Inhibition of corrosion of carbon steel in dam water (Tamil Nadu, India) by Pentane sulphonicacid as its sodium salt (SPS)-Zinc ion system has been studied using weight-loss and potentiodynamic polarization methods. Results of weight loss method indicated that inhibition efficiency (IE) increased with increasing inhibitor concentration. Polarization study reveals that SPS- $Zn^{2+}$  system functions as a cathodic inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. The nature of protective film formed on the metal surface has been analysed by FTIR spectra and AFM analysis. From the results of above studies a suitable mechanism for corrosion inhibition is proposed.

Keywords: AFM, Carbon steel, Corrosion, Electrochemical techniques, FTIR.

# INTRODUCTION

Corrosion plays a very important role in diverse fields of industry and, consequently, in economics. The protection of metals and alloys is thus of particular interest. The goal of studying the processes of corrosion is to find methods of minimizing or preventing it. One approach is the use of corrosion inhibitors. Organic compounds have become widely accepted as effective corrosion inhibitors in various media. Most of the organic inhibitors containing nitrogen, oxygen, sulfur atoms, and multiple bonds in their molecules facilitate adsorption on the metal surface [1-3].

Organic compounds are recognized as effective inhibitors of the corrosion of many metals and alloys. The efficiency of an organic compound as a corrosion inhibitor is closely associated with the chemical adsorption [4-8]. Most of these organic compounds contain nitrogen, sulphur, oxygen and multiple bonds in the molecules which are adsorbed on the metal surface and the organic compound [9,10]. Much interest has been devoted to organic inhibitors such as polyethylene glycols [11], 1,2,3 benzotriazole [12], 2 mercaptobenzimidazole [13], ethoxylated fatty acids [14] and meta substituted aniline-N- sallicylidenes [15]. It was also found that the presence of hetero atom with unsaturated bonds in an organic compound caused inhibitive effect and reduces the metal dissolution. Protecting efficiency was also found to be improved with increasing the length of alkyl chain and the organic compound concentration [16]. A survey of the available literature reveals that the Corrosion inhibition of 2-naphthalenesulfonic acid, 2 naphthalenedisulfonic acid and 2-naphthol-3, 6-disulfonic acid on iron electrode in sulfuric acid has been investigated [17]. The inhibition action of 2-mercaptobenzoxazol, 2mercapto benzimidazole, N-cetyl pyridinium bromide and propargyl benzene\_sulphonate on the corrosion of carbon steel in acid media have also been studied [18]. Manickavasagam et al., have reported the corrosion inhibition of Poly (Styrene sulphonic acid) - doped poly aniline on carbon steel in acid media [19]. Aliev has described the influence of salts of Alkyl phenol Sulphonic acid on the corrosion of ST3 steel [20]. Shakkthivel and Vasudevan have studied the effect of Acrylic aciddiphenylamine sulphonic acid copolymer threshold inhibitor for sulphate and carbonate scales in cooling water systems [21].

Ion pair reagents are used as counter ions in ion pairs in reverse phase liquid chromatography and in reverse phase thin layer chromatography. They have been used in separation and quantitative determination of amino acids and peptides, of nucleosides, of vitamin B<sub>4</sub> compounds, of forensic drugs and of neuropeptides and various other pharmaceutical products. N-alkyl sulphonates interact specifically with biologicals, especially proteins, by virtue of electrostatic interaction along the polar chain or because of their stereochemistry in solution [22].

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 by the industries located downstream in cooling systems.

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The present work is undertaken

- to evaluate the inhibition efficiency (IE) of an ion pair reagent, Sodium pentane sulphonate (SPS) - Zn system, in controlling corrosion of carbon steel immersed in dam water in the absence and presence of Zn<sup>+</sup> by weight loss method,
- to study the mechanism of corrosion inhibition by Polarization study and AC impedance spectra,
- 3) to analyse the protective film by FTIR spectra and Atomic Force Microscope and
- 4) to propose the mechanism of corrosion inhibition based on the above results.

# MATERIALS AND METHODOLOGY

## **Preparation of the Specimen**

Carbon steel specimens of size 1.0 cm  $\times$  4.0 cm x 0.2 cm and chemical composition 0.026 % Sulphur, 0.06 % Phosphorous, 0.4 % Manganese, 0.1 % Carbon and the rest iron, (specimen density 7.87 gm/cm<sup>3</sup>), were polished to a mirror finish and degreased with trichloroethylene and used for the weight loss method and surface examination studies.

#### Weight-Loss Method

Carbon steel specimens were immersed in 100 ml of the water containing various concentrations of the inhibitor (Sodium 1-Pentane Sulphonate) in the absence and presence of  $Zn^{2+}$  for 3 days. The molecular structure of Sodium 1-pentane sulphonate is given below.



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 concentrated Hydrochloric acid of specific gravity (1.9) [23].

The corrosion IE was calculated using the equation,

$$IE = 100 \left[ 1 - (W_2/W_1) \right] \%$$
 (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 the formula,

Mils penetration per year (mpy) = 
$$534 \text{ W} / \text{DAT}$$
 (2)

where Mils penetration per year is the rate of penetration in milli inches per year which is the customary unit for corrosion rate

W = weight loss in milligrams

 $D = density of specimen in g/cm^3$ 

A = area of specimen in square inches

T = exposure time in hours

#### **Surface Examination Study**

The carbon steel specimens were immersed in various test solutions for a period of 1 day. After 1 day, 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.

## **Fourier Transform Infrared Spectra**

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.

#### Atomic Force Microscopy Characterization (AFM)

The carbon steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and subjected to surface examination. Atomic force microscopy (Veeco dinnova model) was used to observe the surface of the samples in tapping mode, using cantilever with linear tips. The scanning area in the images was  $5 \ \mu m \times 5 \ \mu m$  and the scan rate was  $0.6 \ Hz$ /second

#### **Potentiodynamic Polarization**

Polarization studies were carried out in a CHIelectrochemical work station with impedance model 660A. It was provided with iR compensation facility. A three electrodes cell assembly was used. The working electrode was carbon steel. A SCE 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 study (LPR) was done. The scan rate (V/s) was 0.01. Hold time at Ef (s) was zero and quiet time (s) was two.

## **AC Impedance Spectra**

The instrument used for polarization study was used to record AC impedance spectra also. The cell set up was also the same. 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. AC impedance spectra were recorded with initial  $E_{(v)} = 0$ , high frequency (Hz) = 1x10<sup>5</sup>, low frequency (Hz) = 1, amplitude (V) = 0.005 and quiet time (s) = 2.

# **RESULTS AND DISCUSSION**

## Weight-Loss Study

The physicochemical parameters of dam water (Tamil Nadu, India) are given in Table 1.

The corrosion inhibition efficiencies and the corresponding corrosion rates (mpy) of Sodium pentane sulphonate (SPS)- $Zn^{2+}$  systems are given in Table 2.

The inhibition efficiencies of SPS-Zn<sup>2+</sup> systems are given in Table 2. It is found that the IE increases as the concentration of SPS increases. As the concentration of Zn<sup>2+</sup> increases, IE also increases. A synergistic effect exists between SPS and Zn<sup>2+</sup>. For example, 250 ppm of SPS has 22%IE. 30 ppm of Zn<sup>2+</sup> has 21% IE. However, interestingly, the formulation consisting of 250 ppm of SPS and 30 ppm of Zn<sup>2+</sup> has 68% IE. That is mixture of inhibitors shows better inhibition efficiency than the individual inhibitors [24].

 Table 1.
 Water Analysis (Sothuparai Dam Water, Tamil Nadu, India)

Parameters	Result		
Appearance	Brownish		
Total dissolved solids	100 ppm		
Electrical conductivity	140 μ mhos/cm		
pH	8.25		
Total hardness as CaCO <sub>3</sub>	50 ppm		
Calcium	10 ppm		
Magnesium	06 ppm		
Iron	1.2 ppm		
Nitrate	10 ppm		
Chloride	10 ppm		
Sulphate	02 ppm		

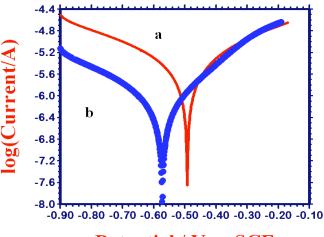
Table 2.Inhibition Efficiencies (IE) and the Corrosion Rates<br/>(CR) of Carbon Steel Immersed in Dam Water in<br/>the Presence of SPS-Zn<sup>2+</sup> System

	Zn <sup>2+</sup> (ppm)					
Inhibitor SPS (ppm)	0		15		30	
	IE %	CR mpy	IE %	CR mpy	IE %	CR mpy
0	-	4.4384	12	3.9058	21	3.5063
50	07	4.1277	16	3.7283	47	2.3524
100	11	3.9502	20	3.5507	49	2.2636
150	13	3.8614	17	3.6389	54	2.0416
200	19	3.5951	24	2.3732	61	1.7320
250	22	3.4620	26	3.2844	68	1.4203

#### **Analysis of Polarization Curves**

The potentiodynamic polarization curves of carbon steel immersed in various test solutions are shown in Fig. (1). The corrosion parameters are given in Table 3. When carbon steel is immersed in dam water, the corrosion potential is -494 mV vs SCE. The formulation consisting of 250 ppm SPS + 30 ppm Zn<sup>2+</sup> shifts the corrosion potential to -573 mV vs SCE. This suggests that the cathodic reaction is controlled predominantly. The corrosion current value and LPR value for dam water are  $2.66 \times 10^{-6} \text{ A/cm}^2$  and  $2.05 \times 10^4 \Omega \text{ cm}^2$ .

For the formulation of SPS (250 ppm) +  $Zn^{2+}$  (30 ppm), the corrosion current value has decreased to  $4.96 \times 10^{-7}$  A/cm<sup>2</sup>, and the LPR value has increased to  $7.70 \times 104 \ \Omega \ cm^2$ . This indicates that a protective film is formed on the metal surface. When a protective film is formed on the metal surface LPR value increases and corrosion current value decreases [25-29].



**Potential / V vs SCE** 

Fig. (1). Polarisation curves of carbon steel immersed in various test solutions. a) Dam water. b) Dam water containing 250 ppm of SPS and 30 ppm of  $Zn^{2+}$ .

## 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<sub>t</sub>) increases and double layer capacitance (C<sub>dl</sub>) value decreases [30-34]. The AC impedance spectra of carbon steel immersed in various solutions are shown in Fig. (2). The AC impedance parameters, namely charge transfer resistance (R<sub>t</sub>) and double layer capacitance (C<sub>dl</sub>) are given in Table 4. When carbon steel is immersed in dam water, R<sub>t</sub> value is  $1.084 \times 10^4$   $\Omega$  cm<sup>2</sup> and C<sub>dl</sub> value is  $8.24 \times 10^{-10}$  F/cm<sup>2</sup>. When SPS and Zn<sup>2+</sup> are added to dam water, R<sub>t</sub> value increases from  $1.084 \times 10^4$   $\Omega$  cm<sup>2</sup> to  $1.396 \times 10^4 \Omega$  cm<sup>2</sup> and the C<sub>dl</sub> decreases from  $8.24 \times 10^{-10}$  F/cm<sup>2</sup> to  $6.38 \times 10^{-10}$  F/cm<sup>2</sup>. This suggests that a protective film is formed on the surface of the metal. This is further supported by the increase in impedance value from 4.062 to  $4.128 \log(z/ohm)$  (Fig. 3). This accounts for the very high IE of SPS-Zn<sup>2+</sup> system.

## **Analysis of FTIR Spectra**

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 [33-37]. FTIR spectra have been used to analyze the

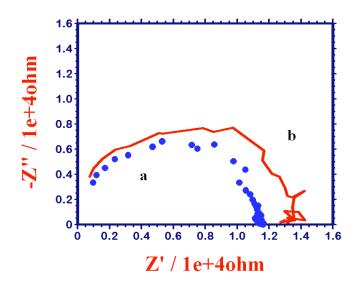
 Table 3.
 Corrosion Parameters of Carbon Steel Immersed in Dam Water in the Presence and Absence of Inhibitor Obtained by Polarization Method

Inhibitor SPS (ppm)	Zn <sup>2+</sup> (ppm)	Ecorr mV vs SCE	Icorr A/cm <sup>2</sup>	ba mV/dec	bc mV/dec	LPR Ω cm <sup>2</sup>
0	0	-494	2.66×10 <sup>-6</sup>	166	203	$2.05 \times 10^{4}$
250	30	-573	4.96×10 <sup>-7</sup>	181	152	$7.70 \times 10^4$

protective film formed on metal surface. FTIR spectrum of pure SPS is shown in Fig (**4a**). The FTIR spectrum of the film formed on the metal surface after immersion in the dam water for one day containing 250 ppm of SPS and 30 ppm of Zn<sup>2+</sup> is shown in Fig (**4b**). The S=O stretching frequency of SPS has decreased from1194 cm<sup>-1</sup> to 1123 cm<sup>-1</sup>. This indicates that the oxygen atom of S=O group has coordinated with Fe<sup>2+</sup> formed on the metal surface resulting in the formation of Fe<sup>2+</sup>-SPS complex on the anodic sites of the metal surface. The peak at 1397 cm<sup>-1</sup> is due to Zn-O stretching. The stretching frequency due to –OH appears at 3419 cm<sup>-1</sup>. So, it is concluded that Zn (OH)<sub>2</sub> is formed on cathodic sites of the metal surface [38-40].

Table 4.Impedance Parameters of Carbon Steel in Dam<br/>Water in the Presence and Absence of Inhibitor<br/>Obtained by AC Impedance Method

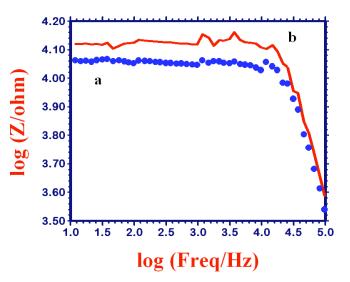
SPS (ppm)	Zn <sup>2+</sup> (ppm)	$rac{R_t}{\Omega cm^2}$	$\frac{C_{dl}}{F/cm^2}.$	Impedance log(z/ohm)
0	0	1.084×10 <sup>4</sup>	8.24×10 <sup>-10</sup>	4.062
250	30	1.3960×10 <sup>4</sup>	6.38×10 <sup>-10</sup>	4.128



**Fig. (2).** AC impedance spectra of carbon steel immersed in various test solutions. **a)** Dam water. **b)** Dam water containing 250 ppm of SPS and 30 ppm of  $Zn^{2+}$ .

#### **Atomic Force Microscopy Characterization**

AFM is a powerful technique to investigate the surface morphology at nano-to micro-scale 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 [41-43]. The two dimensional (2D), three dimensional (3D) AFM morphologies and the AFM crosssectional 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 the formulation of 250 ppm of SPS and 30 ppm of Zn<sup>2+</sup> are shown as Fig. (5) images (a, d, g), (b, e, h), (c, f, i) respectively.



**Fig. (3).** AC impedance spectra of carbon steel immersed in various test solutions. (Frequency Bode plots). **a**) Dam water. **b**) Dam water containing 250 ppm of SPS and 30 ppm of  $Zn^{2+}$ .

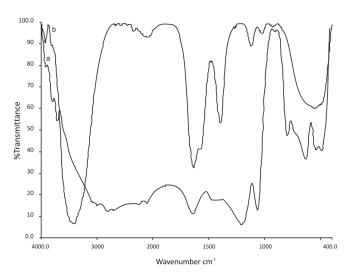
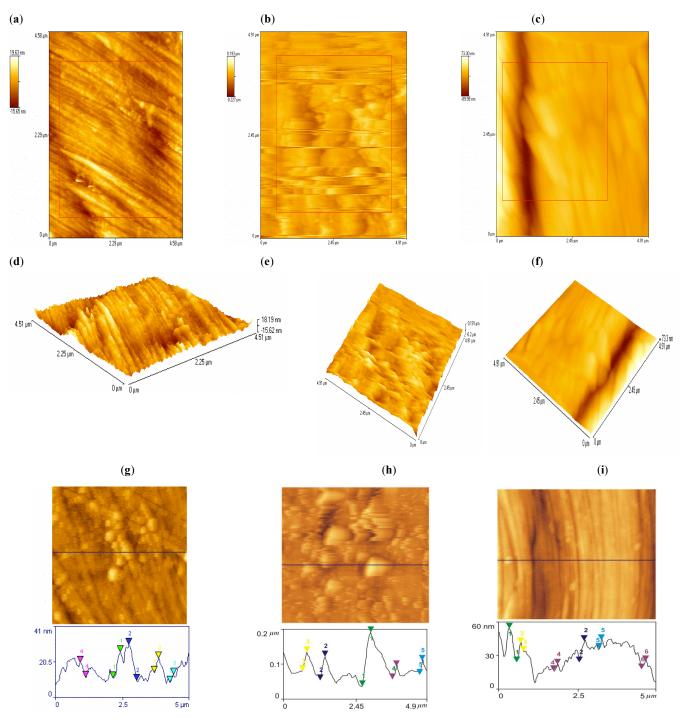


Fig. (4). FTIR spectra **a**) pure sodium pentane sulphonate, **b**) film formed on metal surface after immersion in dam water containing 250 ppm of SPS -30 ppm  $Zn^{2+}$ .

# 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) [41]. Table **5** is a summary of ( $R_q$ ), ( $R_a$ ), (P-V) value for carbon steel surface immersed in different environment.

Fig. (5a, d, g) displays the surface topography of uncorroded metal surface. The values of  $R_q$ ,  $R_a$  and P-V height for the polished carbon steel surface (reference sample) are 4.3 nm, 3.4 nm and 35.3 nm respectively. The



**Fig. (5).** Two dimensional AFM images of the surface of: **a**) As polished carbon steel(control); **b**) carbon steel immersed in dam water (blank); **c**) carbon steel immersed in dam water containing SHXS (250 ppm) +  $Zn^{2+}$  (30ppm). Three dimensional AFM images of the surface of: **d**) As polished carbon steel(control); **e**) carbon steel immersed in dam water (blank); **f**) carbon steel immersed in dam water containing SHXS (250 ppm) +  $Zn^{2+}$  (30ppm). AFM cross-sectional images of the surface of: **g**) As polished carbon steel (control); **h**) carbon steel immersed in dam water (blank); **i**) carbon steel immersed in dam water containing SHXS (250 ppm) +  $Zn^{2+}$  (30ppm).

slight roughness observed on the polished carbon steel surface is due to atmospheric corrosion.

Fig. (**5b**, **e**, **h**) displays the corroded metal surface with few pits in the absence of the inhibitor immersed in dam water. The ( $R_q$ ), ( $R_a$ ), (P-V) height values for the carbon steel surface are 31.9nm, 24.9nm and 420.3 nm respectively. These data suggests 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 was due to the corrosion of the carbon steel in dam water environment.

Fig. (5c, f, i) displays the steel surface after immersion in dam water containing 250 ppm of SPS and 30 ppm of  $Zn^{2+}$ . The (R<sub>q</sub>), (R<sub>a</sub>), (P-V) height values for the carbon steel

Samples	RMS (Rq) Roughness (nm)	Average (R <sub>a</sub> ) Roughness (nm)	Maximum Peak –to- Valley Height (nm)
Polished carbon steel	4.3	3.4	35.3
Carbon steel immersed in dam water (blank)	31.9	24.9	420.3
Carbon steel immersed in dam water +SPS (250ppm) + Zn <sup>2+</sup> (30ppm)	10.28	8.01	76.65

#### Table 5. AFM Data for Carbon Steel Surface Immersed in Inhibited and Uninhibited Environment

surface are 10.28nm, 8.01nm and 76.65nm respectively The  $(R_{q})$ ,  $(R_{a})$ , (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 Fe<sup>2+</sup>- SPS complex and  $Zn(OH)_2$  on the metal surface thereby inhibiting the corrosion of carbon steel [41].

## **Mechanism of Corrosion Inhibition**

With these discussions, a mechanism may be proposed for the corrosion inhibition of carbon steel immersed in dam water by 250 ppm SPS and 30 ppm  $Zn^{2+}$  system.

When the formulation consisting of 250 ppm of SPS and 30 ppm of  $Zn^{2+}$  in dam water is prepared there is formation of  $SPS - Zn^{2+}$  complex in solution

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

$$Zn^{2+} - SPS + Fe^{2+} - SPS + Zn^{2+}$$

- 3) The released  $Zn^{2\scriptscriptstyle +}$  combines with OH  $^-$  to form  $Zn(OH)_2$ on the cathodic sites of the metal surface  $Zn^{2+} + 2$  OH  $\rightarrow$  Zn(OH)<sub>2</sub>  $\downarrow$
- 4) Thus the protective film consists of SPS-  $Fe^{2+}$  complex and  $Zn(OH)_2$ .
- 5) This account for the synergistic effect of SPS  $Zn^{2+}$ system.

Similar mechanism has been proposed by Noreen Antony et al., [36] while studying Inhibition and biocide actions of Sodium dodecyl sulfate- Zn<sup>2+</sup> system for the corrosion of carbon steel in chloride solution and by Sathiyabama et al., [44] while studying Methyl orange as corrosion inhibitor for carbon steel in well water.

## **CONCLUSIONS**

The present study leads to the following conclusions

The inhibition efficiency (IE) of SPS 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 250 ppm SPS and 30 ppm  $Zn^{2+}$  has 68% IE. Polarization study reveals that SPS –  $Zn^{2+}$  system functions as a cathodic inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. FTIR spectra reveal that the protective film consists of  $Fe^{2+}$  – SPS complex and Zn(OH)<sub>2</sub>.

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### **CONFLICT OF INTEREST**

None declared.

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