

Synergistic effect of ethylphosphonic acid–Zn²⁺ system in controlling corrosion of carbon steel in chloride medium

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The inhibition efficiency of ethylphosphonic acid (EPA) in controlling corrosion of carbon steel, immersed in an aqueous solution containing 120 ppm of Cl⁻ and sulphate ion, has been evaluated by weight loss method in the absence and presence of Zn²⁺. Weight loss study reveals that the formulation consisting of 250 ppm of EPA, 50 ppm of Zn²⁺ and 73.58 ppm of sulphate ion has 84% inhibition efficiency in controlling corrosion of carbon steel, immersed in an aqueous solution containing 120 ppm of Cl⁻. Synergism parameters suggest that a synergistic effect exists between EPA and Zn²⁺. Polarization study reveals that this system functions as an anodic inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. FTIR spectra reveal that the protection film consists of Fe²⁺–EPA complex. This is confirmed by UV-visible reflectance spectra.

Key words: Carbon steel, corrosion inhibition, phosphonic acid, UV spectra, synergistic effect, F-test

INTRODUCTION

Inhibition of corrosion and scaling can be done by the application of inhibitors. It is noted that the effect of corrosion inhibitors is always caused by change in the state of surface, being protected due to adsorption or formation of hardly soluble compounds with metal cations. Several phosphonic acids have been used as corrosion inhibitors along with metal cation such as Zn²⁺ [1–5]. Phosphonic acids are the inhibitors which have been widely used due to their stability, ability to form complexes with metal cations and scale inhibiting properties [6–8]. Electrochemical techniques have been used in corrosion inhibition studies of carbon steel by phosphonic acids [9, 10]. The electrochemical techniques have provided only macroscopic details of the redox reaction and no mechanistic information [11–13]. To understand the mechanism of the effect of inhibitors on the metal surface analytical techniques for surface must be used [14, 15]. The aim of the present study is to investigate synergistic corrosion inhibition for the EPA and Zn²⁺ combination to carbon steel immersed in aqueous solution containing 120 ppm of Cl⁻ and 29.43 ppm of sulphate ion. The corrosion inhibition efficiency has been evaluated using weight loss method. Electrochemical studies such as polarization and AC impedance spectra have been employed. The protective film formed on the metal surface was

characterized with the help of surface analytical techniques such as UV-visible reflectance spectra and Fourier transform infrared (FTIR) spectroscopy.

METHODS AND MATERIALS

Carbon steel (0.026% S, 0.06% P, 0.4% Mn, 0.1% C and the rest Fe) specimens of dimensions 4×1×0.2 cm were used for weight loss study. Carbon steel encapsulated in teflon was polished to a mirror finish and degreased with trichloroethylene. The surface area of the exposed metal surface was 1 cm². This specimen was used in electrochemical studies.

The experiment was carried out at room temperature (37°C). Three carbon steel specimens were immersed in 100 ml of the solution containing 120 ppm of Cl⁻ and sulphate ion and various concentrations of the inhibitor in the absence and presence of Zn²⁺ (ZnSO₄·7H₂O) for a period of 5 days. The weight of the specimen before and after immersion was determined using Shimadzu balance AY62. Inhibition efficiency (IE) was calculated from the relationship $IE = (1 - W_2/W_1) \times 100$, where W_1 = corrosion rate in the absence of inhibitor, and W_2 = corrosion rate in the presence of the inhibitor.

The carbon steel specimens were immersed in various test solution for a period of 5 days. After 5 days, the specimens were taken out and dried. The film formed on the surface of the metal specimens, was analysed by surface analysis technique.

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IR spectra were recorded with a Perkin Elmer-1600 spectrophotometer. The FTIR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr and making the pellet.

Polarization study was carried out in an H and CH electrochemical work station Impedance Analyzer Model CHI 660A, provided with *iR* compensation facility, using a three electrode cell assembly. Carbon steel was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. After having done *iR* compensation, polarization study was carried out. The corrosion parameters such as linear polarization resistance (LPR), corrosion potential E_{corr} , corrosion current I_{corr} and Tafel's slopes (b_a and b_c) were measured.

AC impedance spectra were recorded in the same instrument used for polarization study, using the same type of three electrode cell assembly. The real part (Z') and imaginary part (Z'') of the cell impedance were measured in Ohms for various frequencies. The charge transfer resistance (R_t) and double layer capacitance (C_{dl}) values were calculated.

UV-visible absorption spectra of solutions were recorded using a Hitachi Model U-3400 spectrophotometer. The same instrument was used for recording UV-visible reflectance spectra of the film formed on the metal surface.

RESULTS AND DISCUSSION

Corrosion rates of carbon steel, immersed in an aqueous solution containing 120 ppm of Cl⁻, in the absence and presence of EPA and Zn²⁺ and sulphate ion, obtained by weight loss method are given in Table 1. The inhibition efficiencies are also given in this table.

Table 1. Corrosion rates (CR) of carbon steel, immersed in an aqueous solution containing 120 ppm of Cl⁻ ion in the absence and presence of inhibitor and the inhibition efficiencies (IE) obtained by weight loss method (when Zn²⁺ = 25 ppm, sulphate ion = 36.79 ppm; when Zn²⁺ = 50 ppm, sulphate ion = 73.58 ppm). Inhibitor EPA + Zn²⁺; Immersion period 5 days.

| Cl ⁻ ppm | EPA, ppm | Zn ²⁺ , ppm | | | | | |
|------------------------|-------------|------------------------|----------|------------|----------|------------|----------|
| | | 0 | | 25 | | 50 | |
| | | CR, mdd | IE, % | CR, mdd | IE, % | CR, mdd | IE, % |
| 120 | 0 | 18.36 | - | 21.66 | 18 | 19.09 | 4 |
| 120 | 50 | 17.44 | 5 | 13.22 | 28 | 8.44 | 54 |
| 120 | 100 | 16.16 | 12 | 12.48 | 32 | 6.43 | 65 |
| 120 | 150 | 15.05 | 18 | 10.10 | 45 | 5.14 | 72 |
| 120 | 200 | 14.69 | 20 | 9.55 | 48 | 3.67 | 80 |
| 120 | 250 | 13.95 | 24 | 8.81 | 52 | 2.94 | 84 |

It is observed that when carbon steel is immersed

in 120 ppm of Cl⁻, the corrosion rate is 18.36 mdd. Upon addition of various concentrations of EPA, the corrosion rate slowly decreases. The inhibition efficiency gradually increases from 5% to 24% (250 ppm of EPA has 24% IE). That is the formulation consisting of 250 ppm of EPA in 120 ppm of Cl⁻ ion environment, has 24% IE.

The influence of a divalent metal ion, Zn²⁺, on the efficiency of ethyl phosphonic acid, in controlling corrosion of carbon steel, is given in Tables 2 and 3. It is observed that in the presence of 25 ppm of Zn²⁺, (and 36.79 ppm of sulphate ion), the IE of EPA slightly improves. The divalent Zn²⁺ ion forms a complex with EPA, diffuses towards the carbon steel surface, forms Fe²⁺-EPA complex on metal surface and Zn²⁺ is released. In the presence of 50 ppm of Zn²⁺, (73.58 ppm of sulphate ion), the inhibition efficiency of EPA further increases.

A synergistic effect exists between Zn²⁺ and EPA. For example 50 ppm of Zn²⁺ (73.58 ppm of sulphate ion) has 4% IE. (The negative IE indicates the acceleration of corrosion rate in presence of Zn²⁺ alone). 250 ppm of EPA has 24% IE. However, their combination has 84% IE. This is due to the synergistic effect existing between EPA and Zn²⁺.

Synergism parameters were calculated using the relation:

$$S_1 = (1 - I_{1+2}) / (1 - I_1 - I_2)$$

where $I_{1+2} = (I_1 + I_2) - (I_1 \times I_2)$; I_1 = inhibition efficiency of substance 1; I_2 = inhibition efficiency of substance 2; I_{1+2} = combined inhibition efficiency of substance 1 and 2.

Synergism parameters are indications of synergistic effect existing between two inhibitors [16, 17]. The values of synergism parameters (Table 2) are greater than one, indicating synergistic effect existing between Zn²⁺ and various concentrations of EPA.

Table 2. Synergism parameters derived from inhibition efficiencies of EPA – Zn²⁺ system (when Zn²⁺ = 25 ppm, sulphate ion = 36.79 ppm; when Zn²⁺ = 50 ppm, sulphate ion = 73.58 ppm).

| EPA, ppm | Zn ²⁺ , ppm | | S_1 | Zn ²⁺ , ppm | | S_1 |
|-------------|------------------------|----|-------|------------------------|----|-------|
| | 0 | 25 | | 0 | 50 | |
| | 0 | - | | 18 | - | |
| 50 | 5 | 28 | 2.81 | 5 | 54 | 0.38 |
| 100 | 12 | 32 | 6.74 | 12 | 65 | 0.86 |
| 150 | 18 | 45 | 7.34 | 18 | 72 | 1.20 |
| 200 | 20 | 48 | 7.68 | 20 | 80 | 1.20 |
| 250 | 24 | 52 | 8.57 | 24 | 84 | 1.38 |

To investigate whether the influence of Zn²⁺ on the inhibition efficiencies of EPA is statistically significant, F-test was carried out [18]. The results

are given in Tables 3 and 4. In Table 3, the influence of 25 ppm of Zn²⁺ on the inhibition efficiencies of 50, 100, 150, 200 and 250 ppm of EPA is investigated. The obtained F-value 154.1 is statistically significant, since it is greater than the critical F-value of 5.32 for 1, 8 degrees of freedom at 0.05 level of significance. Therefore, it is concluded that the influence of 25 ppm Zn²⁺ on the inhibition efficiencies of various concentrations of EPA is statistically significant.

Table 3. Distribution of F-value between the inhibition efficiencies of various concentrations of EPA (0 ppm Zn²⁺) and the inhibition efficiencies of EPA in the presence of 25 ppm Zn²⁺ (when Zn²⁺ = 25 ppm, sulphate ion = 36.79 ppm).

| Source of variance | Sum of squares | Degrees of freedom | Mean square | F | Level of significance of F |
|--------------------|----------------|--------------------|-------------|-------|----------------------------|
| Between | 1587.6 | 1 | 1587.6 | 154.1 | P > 0.05 |
| Within | 82.1 | 8 | 10.3 | | |

In Table 4, the influence of 50 ppm of Zn²⁺ on the inhibition efficiencies of 50, 100, 150, 200 and 250 ppm of EPA is represented. The obtained F-value is 609.4 statistically significant, since it is greater than the critical F-value of 5.32 for 1, 8 degrees of freedom at 0.05 level of significance. Therefore, it is concluded that the influence of 50 ppm Zn²⁺ on the inhibition efficiencies of various concentrations of EPA is statistically significant.

Table 4. Distribution of F-value between the inhibition efficiencies of various concentrations of EPA (0 ppm Zn²⁺) and the inhibition efficiencies of EPA in the presence of 50 ppm Zn²⁺ (when Zn²⁺ = 50 ppm, sulphate ion = 73.58 ppm).

| Source of variance | Sum of squares | Degrees of freedom | Mean square | F | Level of significance of F |
|--------------------|----------------|--------------------|-------------|-------|----------------------------|
| Between | 7617.6 | 1 | 7617.6 | 609.4 | P > 0.05 |
| Within | 99.6 | 8 | 12.5 | | |

The potentiodynamic polarization curves of carbon steel immersed in an aqueous solution containing 120 ppm of Cl⁻ are shown in Fig. 1. The corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel's slopes (b_a ,

b_c) and linear polarization resistance (LPR) are given in Table 5. When carbon steel is immersed in an aqueous solution containing 120 ppm of Cl⁻ ion, the corrosion potential is -490 V vs SCE. When the inhibitors are added (250 ppm of EPA, 50 ppm of Zn²⁺ and 73.58 ppm of sulphate ion), the corrosion potential shifts to the anodic side (-431 V vs SCE). Further, the LPR value increases from 7.31×10^2 Ohm·cm² to 96×10^2 ohm·cm² and the corrosion current decreases from 8.21×10^{-5} A/cm² to 0.494×10^{-5} A/cm². These results suggest that a protective film (probably, Fe²⁺-EPA complex) is formed on the metal surface. This protects the metal from corrosion.

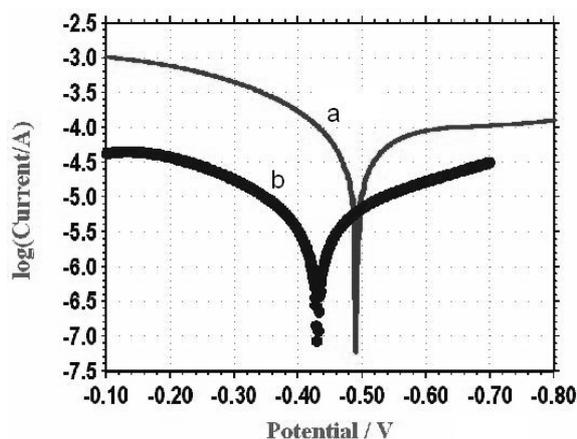


Fig. 1. Polarization curves of carbon steel, immersed in various test solutions 120 ppm Cl⁻ 120 ppm Cl⁻ + 250 ppm EPA + 50 ppm Zn²⁺ + 73.58 ppm of sulphate ion.

The AC impedance spectra of carbon steel, immersed in various test solutions are shown in Fig. 2. The AC impedance parameters such as charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in Table 5. The equivalent circuit diagram is shown in Scheme 1. In the presence of inhibitors (250 ppm of EPA, 50 ppm of Zn²⁺ and 73.58 ppm of sulphate ion), the R_t value increases and C_{dl} value decreases. This indicates that a protective film is formed on the metal surface. The corresponding Bode's plots are shown in Fig. 3. It is observed that in the absence of inhibitors the real impedance value ($\log Z$) is 2.28 Ohm. In the presence of inhibitors, this value increases to 2.62.

Table 5. Corrosion parameters of carbon steel, immersed in an aqueous solution containing 120 ppm of Cl⁻ ion obtained by polarization study and AC impedance spectra. Inhibitor EPA + Zn²⁺.

| System | E_{corr} mV vs SCE | b_c mV/dec | b_a mV/dec | LPR Ohm·cm ² | I_{corr} A/cm ² | R_t ohm·cm ² | C_{dl} F/cm ² | Impedance logZ, Ohm |
|--|-------------------------|-----------------|-----------------|----------------------------|---------------------------------|------------------------------|-------------------------------|------------------------|
| 120 ppm of Cl ⁻ ion | 490 | 506 | 189 | 7.31×10^2 | 8.21×10^{-5} | 154 | 5.88×10^{-8} | 2.28 |
| 120 ppm of Cl ⁻ + 73.58 ppm of sulphate ion + 50 ppm of Zn ²⁺ + 250 ppm of EPA | 431 | 245 | 190 | 96×10^2 | 0.494×10^{-5} | 455 | 1.98×10^{-8} | 2.62 |

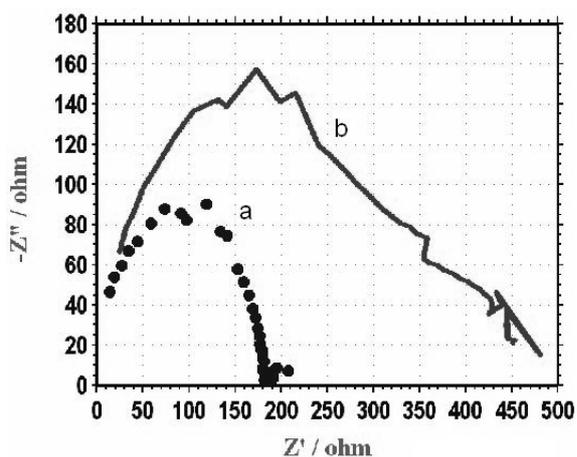
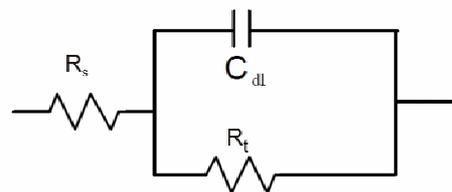


Fig. 2. AC impedance spectra of carbon steel, immersed in various test solutions (Nyquist's plots).

The FTIR spectrum of pure EPA (KBr) is shown in Fig. 4a. The various bands are assigned as follows [19]. The absorption bands due to the bending of O–P–O appear at 474.1, 502.9 and 598.1 cm⁻¹. The P–O stretching frequency occurs at 1071.7 cm⁻¹. The band at 1275.6 cm⁻¹ represents P=O stretching. The band at 1415.8 cm⁻¹ results from P–CH₂–CH₃ absorption. The P(O)OH group causes absorption at 2335, 2356.8 and 2955.6 cm⁻¹. Thus, based on the assignment of groups at various frequencies, ethyl phosphonic acid is characterized by the FTIR spectrum.

The FTIR spectrum of the film scratched from the surface of the metal, immersed in the environment consisting of 120 ppm Cl⁻, 250 ppm EPA, 50 ppm Zn²⁺ and 73.58 ppm of sulphate ion is given in Fig. 4b. It is seen from the spectrum that the P–O stretching frequency decreases from 1071.7 cm⁻¹

to 990 cm⁻¹. This also suggests that the O atom of the phosphonic acid is coordinated to Fe²⁺. This confirms the formation of Fe²⁺–EPA complex on the metal surface. Further, the band at 1340 cm⁻¹ is due to Zn(OH)₂ [20–22].



R_s = Solution resistance
 R_t = Charge transfer resistance
 C_{dl} = Double layer capacitance
 Scheme 1. Equivalent circuit diagram.

The UV-visible absorption spectra of various test solutions are shown in Fig. 5. Fig. 5a is the absorption spectrum of a solution containing 250 ppm of EPA. Fig. 5b is the spectrum of the solution containing 100 ppm of Fe²⁺. When 250 ppm of EPA and 100 ppm of Fe²⁺ are mixed the spectrum Fig. 5c is obtained. There is increase in intensity. A peak appears at 230 nm. This peak corresponds to Fe²⁺–EPA complex formed in the solution.

The UV-visible reflectance spectrum of the film formed on the metal surface after immersion in the solution containing 120 ppm of Cl⁻, 250 ppm of EPA, 50 ppm of Zn²⁺ and 73.58 ppm of sulphate ion is shown in Fig. 6. A peak appears at 230 nm. This peak corresponds to Fe²⁺–EPA complex formed on the metal surface. This is in agreement with the UV-visible absorption spectrum, obtained in the case of aqueous solution.

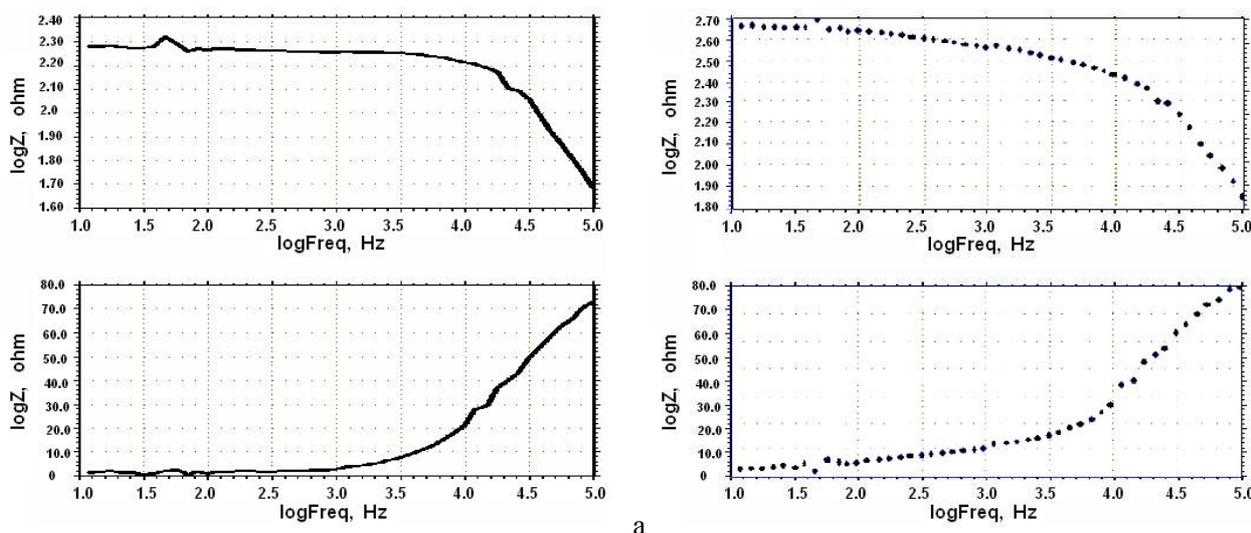


Fig. 3. AC impedance spectra of carbon steel, immersed in an aqueous solution (Bode plots); a. containing 120 ppm Cl⁻; b. containing 120 ppm Cl⁻ + 250 ppm EPA + 73.58 ppm of sulphate ion + 50 ppm Zn²⁺.

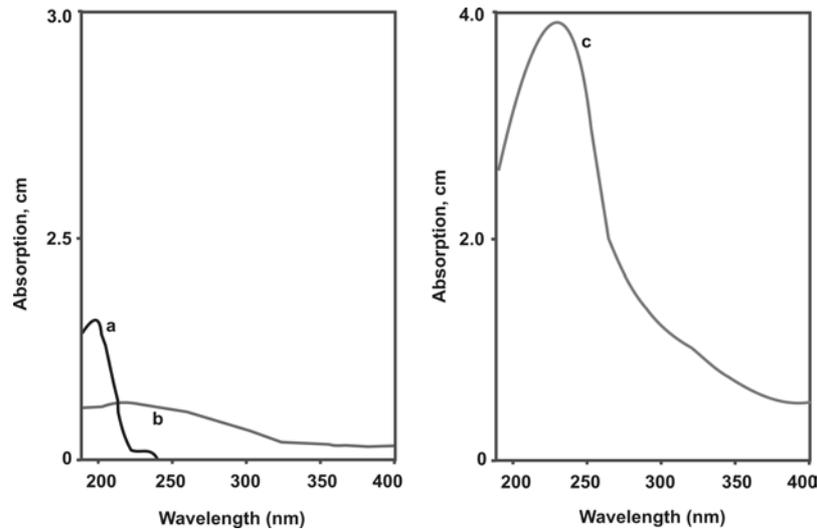


Fig. 5. UV-visible absorption spectra of solutions EPA 250 ppm, Fe²⁺ 100 ppm, EPA 250 ppm + Fe²⁺ 100 ppm.

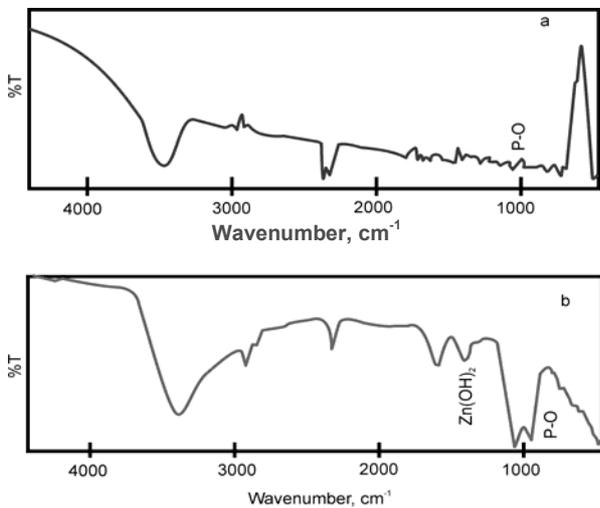


Fig. 4. FTIR spectra of pure EPA (KBr) film formed on carbon steel surface after immersion in the solution containing 120 ppm Cl⁻ + 250 ppm EPA + 73.58 ppm of sulphate ion + 50 ppm Zn²⁺.

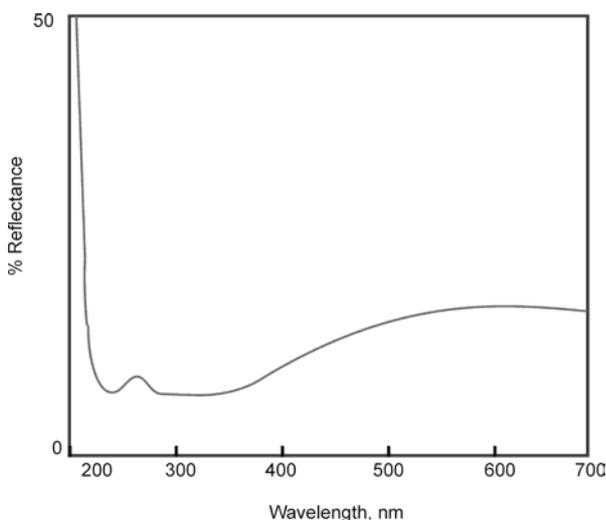


Fig. 6. UV-visible reflectance spectra of mild steel surface immersed in the environment 120 ppm Cl⁻ + EPA 250 ppm + 73.58 ppm of sulphate ion + 50 ppm Zn²⁺.

Mechanism of corrosion inhibition

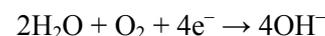
Results of the weight-loss method reveal that the formulation consisting of 250 ppm ethyl phosphonic acid (EPA) and 50 ppm Zn²⁺ offers an inhibition efficiency of 84%. Results of the polarization study show that this formulation acts as an anodic inhibitor. The FTIR spectra show that Fe²⁺–EPA complex and Zn(OH)₂ are present on the inhibited metal surface. The UV-visible reflectance spectrum shows the presence of Fe²⁺–EPA complex on the metal surface.

In order to explain all the observations in a holistic way, a suitable mechanism of corrosion inhibition is proposed as follows:

When carbon steel specimen is immersed in the neutral aqueous environment, the anodic reaction is:



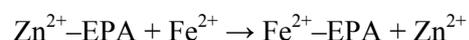
and the cathodic reaction is:



When the environment, consisting of 120 ppm + 250 ppm EPA + 50 ppm Zn²⁺ + 73.58 ppm of sulphate ion is prepared, there is formation of Zn²⁺–EPA complex in solution.

Now, when the metal (carbon steel) is immersed in this environment, the Zn²⁺–EPA complex diffuses from the bulk of the solution to the surface of the metal.

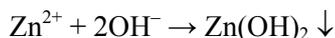
On the surface of the metal, Zn²⁺–EPA complex is converted into Fe²⁺–EPA complex in the local anodic regions as the latter is more stable than the former [23].



This reaction takes place on the surface of the metal in the local anodic regions. Also, formation of Fe³⁺–

EPA complex to some extent cannot be ruled out.

Now, the released Zn²⁺ ions on the surface will form Zn(OH)₂ precipitate in the local cathodic regions.



Thus, the protective film consists of Fe²⁺–EPA complex and Zn(OH)₂.

CONCLUSION

The inhibition efficiency of EPA–Zn²⁺ system in controlling corrosion of carbon steel in an aqueous solution containing 120 ppm of Cl[–], has been evaluated by weight loss method. The present study leads to the following conclusion.

- Weight loss study reveals that the formulation consisting of 250 ppm EPA, 50 ppm of Zn²⁺ and 73.58 ppm of sulphate ion had 84% inhibition efficiency in controlling corrosion of carbon steel immersed in an aqueous solution containing 120 ppm of Cl[–] ion.
- Synergism parameters suggest that a synergistic effect exists between EPA and Zn²⁺.
- Polarization study reveals that this system functions as anodic inhibitor.
- AC impedance spectra reveal that a protective film is formed on the metal surface.
- FTIR spectra reveal that the protective film consists of Fe²⁺–EPA complex as well as of Zn(OH)₂, hydroxyl chloride and hydroxyl sulphate.
- The UV-visible absorption spectra reveal the formation of Fe²⁺–EPA complex, in solution.
- The UV-visible reflectance spectra reveal the presence of Fe²⁺–EPA complex on the metal surface.

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СИНЕРГИЧЕН ЕФЕКТ В СИСТЕМАТА ЕТИЛФОСФОРНА КИСЕЛИНА-Zn²⁺ ЗА КОНТРОЛ НА КОРОЗИЯТА НА ВЪГЛЕРОДНА СТОМАНА В СРЕДА СЪДЪРЖАЩА ХЛОРИДИ

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(Резюме)

Чрез метода на тегловните загуби в отсъствие и присъствие на Zn²⁺ е определена инхибиторната ефективност на етилфосфорна киселина (ЕФК) за контролиране на корозията на въглеродна стомана потопена във водни разтвори съдържащи 120 ppm хлорни и сулфатни йони. Изследването на тегловните загуби показва, че разтвор съдържащ 250 ppm ЕФК, 50 ppm Zn²⁺ и 73.58 ppm сулфатен йон има 84% инхибиторна ефективност за контролиране на корозията на въглеродна стомана потопена във воден разтвор съдържащ 120 ppm хлорни йони. Параметрите сочат за присъствие на синергичен ефект между ЕФК и цинковите йони. Поляризационно изследване показва, че тази система работи като аноден инхибитор. АС импедансни спекти показаха образуване на защитен филм на повърхността на метала. ИЧ спектри показаха, че защитния филм се състои от комплекс на Fe²⁺ и ЕФК. Резултатът е потвърден и с отражателни спектри в УВ-видимата област.