

Research Article

A Study of N,N-Diethylammonium O,O'-Di(*p*-methoxyphenyl)dithiophosphate as New Corrosion Inhibitor for Carbon Steel in Hydrochloric Acid Solution

Chuan Lai,^{1,2,3} Bin Xie,^{2,3} Changlu Liu,¹ Wan Gou,¹
Lvshan Zhou,¹ Xiulan Su,¹ and Like Zou^{2,3}

¹School of Chemistry and Chemical Engineering, Sichuan University of Arts and Science, Dazhou 635000, China

²Institute of Functional Materials, Sichuan University of Science and Engineering, Zigong 643000, China

³Material Corrosion and Protection Key Laboratory of Sichuan Province, Sichuan University of Science and Engineering, Zigong 643000, China

Correspondence should be addressed to Bin Xie; xiebinsuse@163.com

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N,N-Diethylammonium O,O'-di(*p*-methoxyphenyl)dithiophosphate (EAPP) as a new corrosion inhibitor was synthesized in the present work. The corrosion inhibition of EAPP in hydrochloric acid for carbon steel was evaluated by potentiodynamic polarization measurements, electrochemical impedance spectroscopy, weight loss measurements, and scanning electron microscopy. The results indicate that the EAPP is mixed type inhibitor, and the adsorption of EAPP on carbon steel surface obeys Langmuir isotherm. In addition, the inhibition efficiency increases with increasing the concentration of inhibitor and decreases with increasing the hydrochloric acid concentration, temperature, and storage time.

1. Introduction

Acid solutions are often used in industry for cleaning, chemical decaling, and pickling of metals [1, 2]. During those processes, as the existence of undesirable and extensive attack for metals as an important metal in different acid solutions, the application of corrosion inhibitors for preventing corrosion phenomena seems to be more and more important [3, 4]. Using inhibitor is one of the most practical methods for protection against corrosion especially in acid solution to prevent metals dissolution [5, 6]. Compared with other existing corrosion protection technologies, the method of using corrosion inhibitor has some advantages, such as no special equipment required, low cost, and easy operation.

During the past decade, many inhibitors have been studied in different media. Most of the effective corrosion inhibitors are organic compounds containing electronegative atoms (such as nitrogen atoms, phosphorus atoms, sulfur

atoms, and oxygen atoms), the unsaturated bonds (such as double bonds or triple bonds), and the plane conjugated systems including all kinds of aromatic cycles [7–9]. In fact, the O,O'-diaryldithiophosphates contain nitrogen, phosphorus, sulfur, and oxygen atoms in their structure, which could be potential inhibitors.

As a result, in order to develop the new effective corrosion inhibitor, the aim of the present work is to investigate the corrosion inhibition of N,N-diethylammonium O,O'-di(*p*-methoxyphenyl)dithiophosphate (EAPP) for carbon steel in hydrochloric acid (HCl) solution. Firstly, the organic compound of EAPP was synthesized. Meanwhile, EAPP acting as the novel corrosion inhibitor for carbon steel in HCl solution were studied using potentiodynamic polarization measurements, electrochemical impedance spectroscopy, weight loss measurements, and scanning electron microscopy. In addition, the effects of inhibitor concentration, HCl concentration, temperature, and storage time on inhibition action were fully investigated.

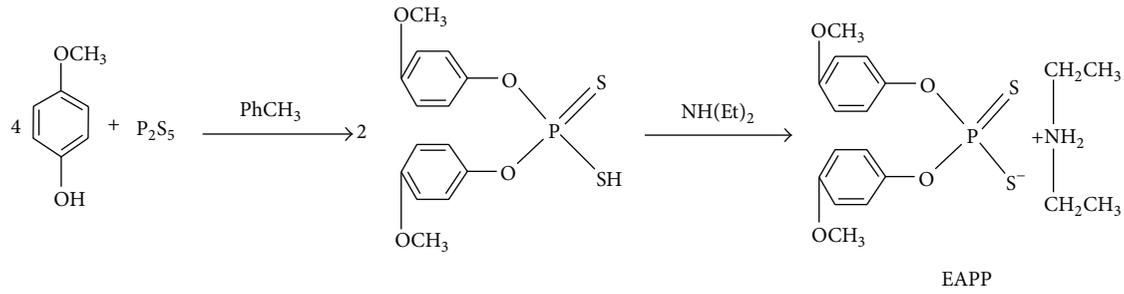


FIGURE 1: Synthesis and molecular structure of the investigated inhibitor.

TABLE 1: Chemical composition of experimental carbon steel (in wt.%).

Element	C	Si	Mn	P	S	Fe
Composition	0.16	0.27	0.42	<0.014	<0.011	Bal.

2. Materials and Methods

2.1. Materials. In this work, phosphorus pentasulphide (P_2S_5), *p*-methoxyphenol (*p*- CH_3OPhOH), diethylamine ($NH(Et)_2$), toluene ($PhCH_3$), hydrochloric acid (37%, HCl), and acetone (CH_3COCH_3) were purchased from Sinopharm Chemical Reagent Co., Ltd. All the reagents were commercially available and used without further purification. The standard samples (50 mm × 20 mm × 5 mm) and working electrode were prepared by carbon steel, whose chemical composition is given in Table 1. Meanwhile, CHI 660D electrochemical workstation (China) was used for electrochemical measurements.

The test solutions of HCl solution were prepared by analytical grade hydrochloric acid and distilled water. During the electrochemical and weight loss measurements, the temperature of test solution was controlled by a water thermostat, and all experiments were open to the air and carried out under static conditions.

2.2. Synthesis of Inhibitor. *N,N*-Diethylammonium *O,O'*-di(*p*-methoxyphenyl)dithiophosphate (EAPP) acting as the new corrosion inhibitor was synthesized according to the synthetic procedures shown in Figure 1, which is prepared by reaction of phosphorus pentasulphide, *p*-methoxyphenol with diethylamine in toluene as the solvent based on the method described in our previous works [10–12].

2.3. Electrochemical Measurements. The electrochemical measurements were done by conventional three-electrode system consisting of carbon steel working electrode with an exposed area of 0.785 cm^2 , a graphite electrode as counter electrode, and saturated calomel electrode (SCE) as reference. All potentials in this work were referred to the SCE. The working electrode was immersed in test solution at open circuit potential (OCP) for 30 min to be sufficient to attain a stable state before measurement.

The potential sweep rate for potentiodynamic polarization (Tafel) curves was 0.166 mV s^{-1} . Corrosion current density (i_{corr}) was determined from the intercept of extrapolated cathodic and anodic Tafel lines at the corrosion potential (E_{corr}). The inhibition efficiency (IE_{Tafel}) was calculated from the following equation [13, 14]:

$$IE_{Tafel} (\%) = \frac{i_{corr} - i_{corr(inh)}}{i_{corr}} \times 100, \quad (1)$$

where i_{corr} and $i_{corr(inh)}$ are the corrosion current density values of carbon steel in HCl solution without and with EAPP, respectively.

The electrochemical impedance spectroscopy (EIS) measurements were performed in frequency range of 100 kHz to 10 m Hz using a sinusoidal AC perturbation with amplitude of 10 mV. EIS parameters were fitted by using ZSimpWin software. Charge transfer resistance (R_{ct}) was obtained from the diameter of the semicircle of the Nyquist plot. The inhibition efficiency (IE_{EIS}) derived from EIS was calculated using the following equation:

$$IE_{EIS} (\%) = \frac{R_{ct} - R_{ct}^0}{R_{ct}} \times 100, \quad (2)$$

where R_{ct} and R_{ct}^0 are the values of charge transfer resistance observed in the presence and absence of inhibitor.

In this paper, the storage time was defined as the time interval from the time of addition EAPP in HCl solution to the time of using the test solution for electrochemical measurements.

2.4. Weight Loss Measurements. Before weight loss measurements, all of the standard carbon steel samples were mechanically abraded with emery paper up to 1200 grit, then rinsed with distilled water, degreased in acetone, and dried at room temperature. The finely polished and dried carbon steel samples were weighed on a digital balance with 1 mg sensitivity and immersed in HCl solution in the absence and presence of EAPP at various temperatures for 12 h. After immersing, the tests samples were rinsed with distilled water, cleaned with acetone, dried, and reweighed. The weight loss was calculated as the difference in weight of the sample before and after immersion in test solutions.

In all the above measurements, at least three closer results were considered, and their average values have been

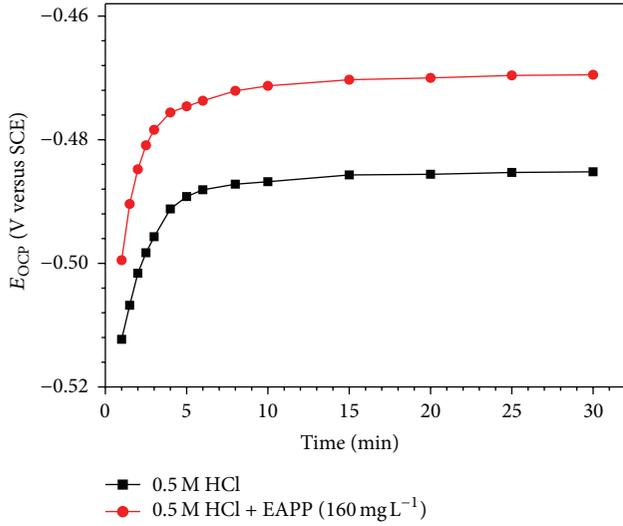


FIGURE 2: Open circuit potential-time curves for carbon steel in 0.5 M HCl in the absence and presence of 160 mg L⁻¹ EAPP at 300 K.

reported. The corrosion rate (v , g m⁻² h⁻¹) and inhibition efficiency ($IE_{\text{Weight loss}}$) were calculated from (3) and (4) [15, 16], respectively,

$$v = \frac{m_i - m_{ii}}{St} = \frac{\Delta m}{St}, \quad (3)$$

$$IE_{\text{Weight loss}} (\%) = \frac{v_0 - v}{v_0} \times 100, \quad (4)$$

where m_i and m_{ii} are the mass of the sample before and after corrosion, Δm is the weight loss of the sample before and after corrosion, S is the total surface area of the sample, t is the immersion time, and v_0 and v are corrosion rate of the carbon steel sample in HCl solution without and with different concentration of EAPP.

2.5. Scanning Electron Microscopy. The surface morphologies of carbon steel samples before and after immersion in 0.5 M HCl at 300 K in the absence and presence of 160 mg L⁻¹ EAPP for 2 h were examined by scanning electron microscopy (SEM, Tescan Vega III).

3. Results and Discussion

3.1. Electrochemical Measurements

3.1.1. Open Circuit Potential Curves. Figure 2 shows the variation of open circuit potential (E_{OCP}) of carbon steel working electrode with immersion time (t) in 0.5 M HCl solution in absence and presence of 160 mg L⁻¹ EAPP at 300 K. The initial potential is moved positive value with time, and gradually remains steady value. Meanwhile, it can be observed that, after 20 min immersion, only negligible changes in the E_{OCP} are measured. Therefore, the steady-state was achieved after 30 min for electrochemical tests. The E_{OCP} value at 30 min in blank solution (0.5 M HCl without

TABLE 2: The polarization parameters and corresponding inhibition efficiency for carbon steel in 0.5 M HCl in the absence and presence of different concentrations of EAPP at 300 K.

C (mg L ⁻¹)	E_{corr} (mV)	i_{corr} ($\mu\text{A cm}^{-2}$)	IE_{Tafel} (%)
0	-524	308.54	—
80	-521	70.22	77.24
100	-504	38.17	87.63
120	-493	32.46	89.48
160	-490	20.82	93.25

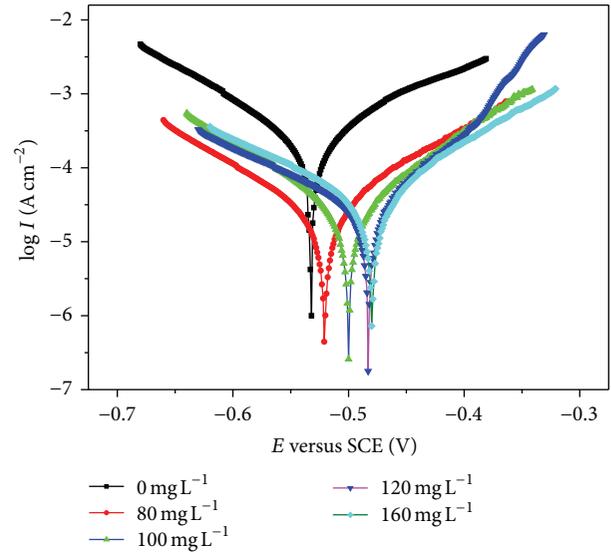


FIGURE 3: Potentiodynamic polarization curves at 300 K for carbon steel in 0.5 M HCl in the absence and presence of different concentrations of EAPP.

inhibitor) is 0.4852 V (versus SCE). The steady-state potential moves positive after adding inhibitor to 0.5 M HCl solution, which indicates that the corrosion of carbon steel is retarded by addition of EAPP in HCl solution.

3.1.2. Potentiodynamic Polarization Measurements

(1) Effect of EAPP Concentration. At 300 K, the potentiodynamic polarization curves of carbon steel in 0.5 M HCl in the absence and presence of different concentrations of EAPP are shown in Figure 3. Meanwhile, the corrosion potential E_{corr} (mV versus SCE), corrosion current density i_{corr} ($\mu\text{A cm}^{-2}$), and inhibition efficiency (IE_{Tafel}) are given in Table 2. The inhibition efficiency was calculated by (1).

From Figure 3 and Table 2, both the anodic and cathodic curves shift to lower current densities, which indicate that the addition of EAPP can reduce the carbon steel anodic dissolution and also retard the hydrogen ions reduction. The inhibition effect enhances with the increase in EAPP concentration, resulting from the adsorption of EAPP on the carbon steel electrode surface. One possible mechanism is the adsorption of EAPP on carbon steel surface through the electron pair of heteroatoms (sulfur and oxygen atoms), the π electron of benzene rings in the molecular structure of

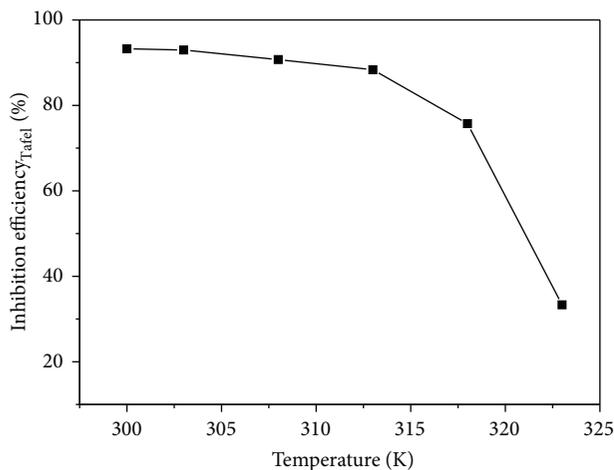


FIGURE 4: Relationships between inhibition efficiency and temperature from potentiodynamic polarization measurements for carbon steel in 0.5 M HCl with 160 mg L⁻¹ EAPP.

EAPP, which blocks the carbon steel surface and reduces the corrosive attraction of carbon steel in HCl media.

Clearly, the corrosion current density decreases considerably in the presence of EAPP comparing with that in the absence of EAPP and decreases with the EAPP concentration increasing. Correspondingly, the inhibition efficiency increases with the EAPP concentration, due to the increase in the blocked fraction of the carbon steel electrode surface by adsorption. The inhibition efficiency of 160 mg L⁻¹ EAPP reaches up to 93.25%, which presents that the EAPP is a good inhibitor for carbon steel in 0.5 M HCl.

In addition, it can classify an inhibitor as cathodic or anodic type if the displacement in corrosion potential is more than 85 mV with respect to corrosion potential of the blank [17, 18]. From Table 2, it can be found that the corrosion potentials shift slightly in the positive or negative direction. All corrosion potentials of carbon steel in 0.5 M HCl at 300 K shift less than 85 mV (versus SCE), and it can be interpreted that EAPP act as mixed type inhibitor acting on both the hydrogen evolution reaction and carbon steel dissolution.

(2) *Effect of Temperature.* Temperature is an important factor that influences the corrosion rate of carbon steel in HCl solution and modifies the adsorption of inhibitor on carbon steel surface. In order to study the effect of temperature on the inhibition characteristics, experiments were conducted at 300 K to 323 K.

The effect of temperature on inhibition efficiency by potentiodynamic polarization measurements is shown in Figure 4. Clearly, inhibition efficiencies decrease with the experimental temperature, which can be attributed to the fact that the higher temperatures might cause desorption of inhibitor molecule from carbon steel surface. With increase in the temperature from 300 K to 323 K, the inhibition efficiency has dropped from 93.25% to 33.32%.

(3) *Effect of HCl Concentration.* The effect of HCl concentration on the inhibition efficiency of EAPP with the

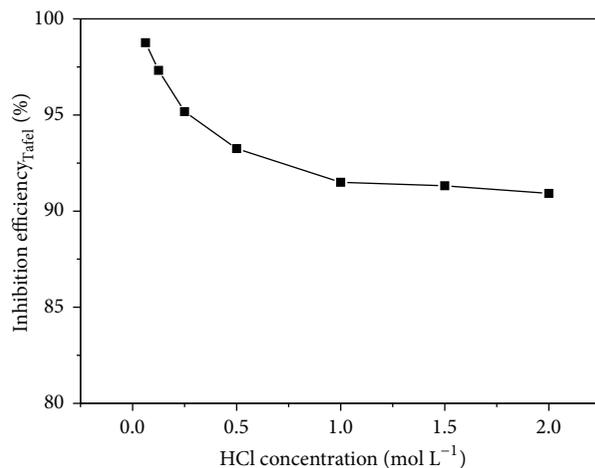


FIGURE 5: Relationships between inhibition efficiency and concentration of HCl with 160 mg L⁻¹ EAPP from potentiodynamic polarization measurements for carbon steel at 300 K.

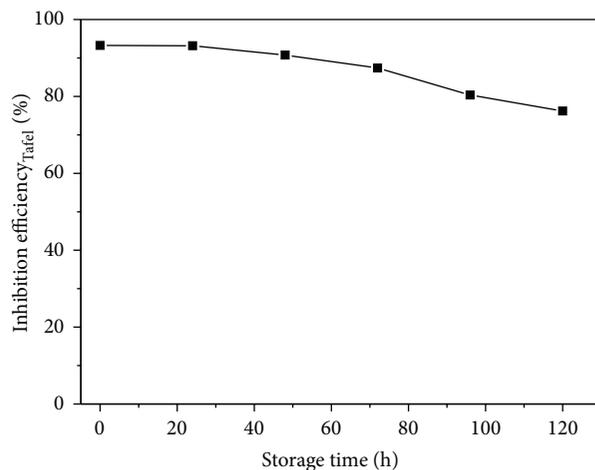


FIGURE 6: Relationships between inhibition efficiency and storage time from potentiodynamic polarization measurements for carbon steel in 0.5 M HCl with 160 mg L⁻¹ EAPP.

concentration of 160 mg L⁻¹ for carbon steel at 300 K is shown in Figure 5. It can be found that the increase of acid concentration resulted in decrease of inhibition efficiency gradually, and the minimum inhibition efficiency in 2.0 M HCl is 90.92%. A similar result has been reported in the literature [19].

(4) *Effect of Storage Time.* The effect of storage time on inhibition efficiency of EAPP with the concentration of 160 mg L⁻¹ for carbon steel in 0.5 M HCl at 300 K is shown in Figure 6. As can be seen from Figure 6, the inhibition efficiency decreases with storage time increasing, but, with addition of EAPP in 0.5 M HCl at 48 hours later, the inhibition efficiency is 90.92%, which shows that EAPP still exhibit the excellent corrosion inhibition for carbon steel in 0.5 M HCl.

TABLE 3: The electrochemical parameters of impedance and corresponding corrosion inhibition efficiencies or carbon steel in 0.5 M HCl in the absence and presence of different concentrations of EAPP at 300 K.

C (mg L^{-1})	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	IE_{EIS} (%)
0	23.37	46.93	—
80	113.23	14.17	77.24
100	212.07	9.39	87.63
120	252.92	6.64	89.48
160	426.46	4.38	93.25

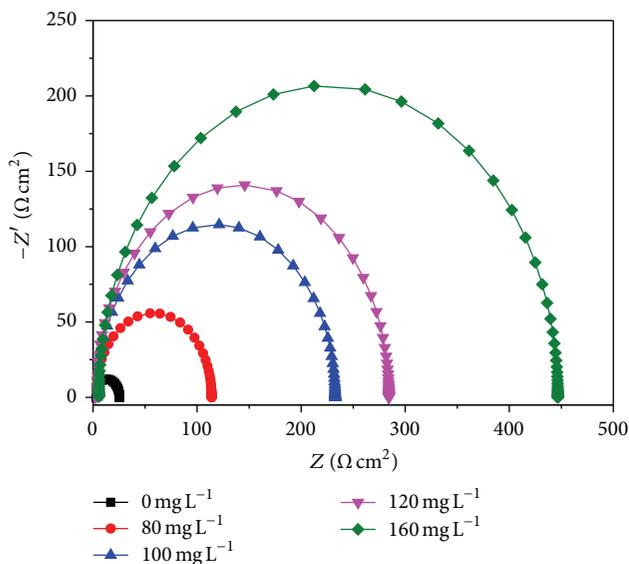


FIGURE 7: Nyquist plots for carbon steel in 0.5 M HCl in the absence and presence of different concentrations of EAPP at 300 K.

3.1.3. *Electrochemical Impedance Spectroscopy.* Figure 7 shows the Nyquist diagrams for carbon steel in 0.5 M HCl with different concentrations of EAPP at 300 K. The double layer capacitance (C_{dl}), charge transfer resistance (R_{ct}), and inhibition efficiency (IE_{EIS}) obtained from EIS measurements are presented in Table 3. It is found that all Nyquist plots show a single capacitive loop, in both uninhibited and inhibited solutions, which is attributed to the charge transfer of corrosion process. The impedance spectra show that the single semicircle and the diameter of semicircle increase with the concentration of the inhibitors. From Table 3, it is revealed that the R_{ct} increased and the C_{dl} values decreased with the concentration of inhibitor increasing. The increase of R_{ct} can be attributed to the formation of protective film on the carbon steel/solution interface. The decrease of C_{dl} may be due to the decrease of the local dielectric constant or the increase of the thickness of the electrical double layer, indicating that the inhibitors adsorbed on the carbon steel surface. The best corrosion inhibition efficiency recorded by EIS is 93.25% for carbon steel in 0.5 M HCl with 160 mg L^{-1} EAPP, which confirms that EAPP can act as good inhibitors for carbon steel in 0.5 M HCl. The result obtained from

TABLE 4: The corrosion rate and inhibitor efficiency for carbon steel in 0.5 M HCl in the absence and presence of different concentrations of EAPP at 300 K from weight loss measurements.

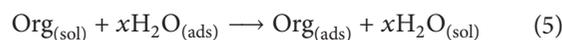
C (mg L^{-1})	v ($\text{g m}^{-2} \text{ h}^{-1}$)	$IE_{\text{Weight loss}}$ (%)
0	4.598	—
40	2.028	55.90
80	1.086	76.38
100	0.729	84.15
120	0.446	90.30
160	0.233	94.93

EIS is in good agreement with the results obtained from potentiodynamic polarization measurements.

3.2. Weight Loss Measurements

3.2.1. *Effect of EAPP Concentration.* The inhibition efficiency ($IE_{\text{Weight loss}}$) of different concentrations of EAPP for carbon steel in 0.5 M HCl at 300 K is listed in Table 4. Clearly, inhibition efficiency increases with increase in concentration of EAPP. It should be noted that when the concentration of EAPP is 160 mg L^{-1} , the maximum inhibition efficiency is 94.93%, which is higher than the inhibition efficiency of N,N-diethylammonium O,O'-diphenyldithiophosphate for carbon steel in 5% HCl (85.24%) [20], and confirms that the EAPP can act as a good corrosion inhibitor for carbon steel in 0.5 M HCl.

3.2.2. *Adsorption Isotherm.* The adsorption isotherm can give important information about the interaction of the inhibitor and metal surface. Two main types of interaction can be used to describe the adsorption behavior of the inhibitor, which are physisorption and chemisorption [21–23]. These processes depend on the chemical structure of the inhibitor molecule, the temperature during the experiments, the electrochemical potential, the charge, and nature of the metal. The adsorption of organic inhibitor molecules from the aqueous solution can be considered as a quasi-substitution process between the organic compounds in the aqueous phase [$\text{Org}_{(\text{sol})}$] and water molecules associated with the metallic surface [$\text{H}_2\text{O}_{(\text{ads})}$] as represented by the following equilibrium [24, 25]:



where x is the number of water molecules replaced by one organic molecule. In this situation, the adsorption of EAPP was accompanied by desorption of water molecules from the carbon steel surface.

Fundamental information on the adsorption of inhibitor on metal surface can be provided by adsorption isotherm. In order to find the most suitable adsorption isotherm for adsorption of EAPP on carbon steel surface, various isotherms including Langmuir, Temkin, Frumkin, and Flory-Huggins adsorption isotherms [26, 27] are employed to fit the experimental data from weight loss measurements. It is found

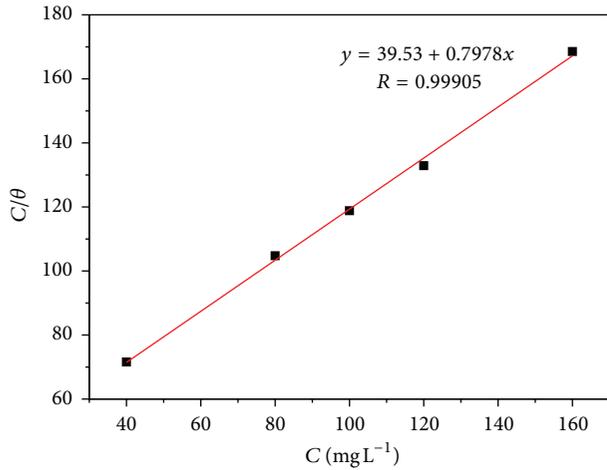


FIGURE 8: Langmuir adsorption isotherm of EAPP on carbon steel in 0.5 M HCl at 300 K from weight loss measurements.

that the adsorption of EAPP on steel surface obeys Langmuir adsorption isotherm as shown in the following [6, 26, 27]:

$$\frac{C}{\theta} = \frac{1}{K_{\text{ads}}} + C, \quad (6)$$

where C is the concentration of EAPP, K_{ads} is the adsorption equilibrium constant, and θ is the surface coverage. The surface coverage (θ) for different concentrations of EAPP in 0.5 M HCl is obtained according to the following equation by weight loss measurements [9, 11, 16]:

$$\theta = \frac{v_0 - v}{v_0}, \quad (7)$$

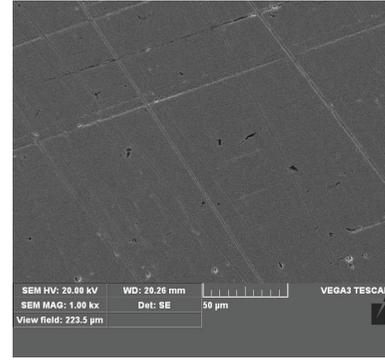
where v_0 and v are corrosion rate of the samples in 0.5 M HCl solution without and with EAPP, respectively.

The plots of C/θ versus C yield the straight lines as shown in Figure 8. The strong correlation ($R > 0.999$) suggests that the adsorption of EAPP on carbon steel surface in HCl solution obeys the Langmuir adsorption isotherm. Meanwhile, the K_{ads} value can be determined from the intercepts of the straight lines and K_{ads} is also related to the standard free energy of adsorption (ΔG_{ads}^0), using the following expression [25, 27, 28]:

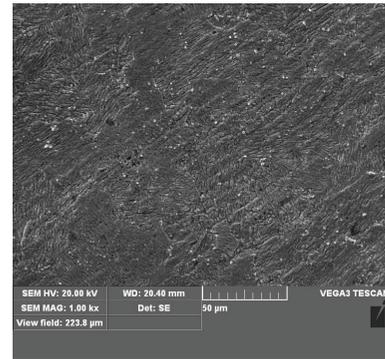
$$K_{\text{ads}} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{\text{ads}}^0}{RT}\right), \quad (8)$$

where R is gas constant ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$), T is absolute temperature (K), and 55.5 is the molar concentration of water in the solution expressed in molarity units (mol L^{-1}).

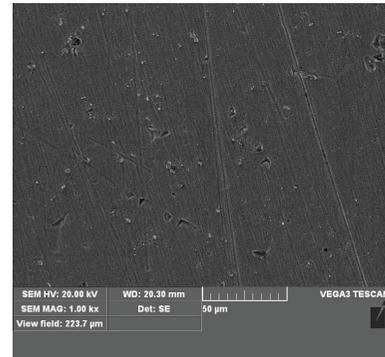
The large values of free energy of adsorption (ΔG_{ads}^0) and its negative sign are usually characteristic of a strong interaction and a high efficient adsorption. In general, the values of ΔG_{ads}^0 around -20 kJ mol^{-1} or less negative are associated with an electrostatic interaction between charged inhibitor molecules and charged metal surface (physisorption) and those of -40 kJ mol^{-1} or more negative involve



(a)



(b)



(c)

FIGURE 9: SEM micrographs for the carbon steel surface: before corrosion (a), after immersion in 0.5 M HCl without EAPP (b), and with 160 mg L^{-1} EAPP (c) at 300 K for 2 h.

charge sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a coordinate type bond (chemisorption) [21–23]. Based on (8), the value of ΔG_{ads}^0 for EAPP on carbon steel in HCl solution at different temperatures is $33.11 \text{ kJ mol}^{-1}$. It indicates that the adsorption of EAPP on carbon steel surface in 0.5 M HCl is chemical adsorption.

3.3. Scanning Electron Microscopy. Figure 9 shows the scanning electron microscopy (SEM) images of carbon steel before and after being immersed in 0.5 M HCl without and with 160 mg L^{-1} EAPP at 300 K for 2 h, which is recorded

in order to see the changes occurring during corrosion process in the absence and presence of the inhibitor of EAPP. Figure 9(a) shows the surface morphology of the sample before immersion in 0.5 M HCl, which mostly seems smooth with only some nicks. However, as can be seen from Figure 9(b), the sample treated with uninhibited solution (0.5 M HCl without EAPP) is strongly corroded, and the surface becomes rough and porous, which reveal that the carbon steel surface is highly corroded and damaged in blank solution (0.5 M HCl without EAPP). In contrast, it can be seen from Figure 9(c) that, in the presence of 160 mg L⁻¹ EAPP, the steel surface is much less damaged, which further confirms the inhibition action and adsorption of EAPP on carbon steel surface. This SEM result is in good agreement with that obtained from weight loss and potentiodynamic polarization measurements.

4. Conclusions

- (1) The inhibition efficiency of N,N-diethylammonium O,O'-di(p-methoxyphenyl)dithiophosphate (EAPP) for carbon steel in HCl solution increases with increasing concentration of EAPP and decreases with an increase of temperature, HCl concentration, and storage time.
- (2) The corrosion current density significantly decreases and corrosion potential slightly changes with the addition of EAPP in HCl solution, and the synthesized inhibitor of EAPP is mixed type inhibitor.
- (3) The adsorption of EAPP on the carbon steel surface is chemical adsorption and obeys Langmuir isotherm.

Conflict of Interests

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

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