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Studies on corrosion inhibitory effect and adsorption behavior of waste materials on mild steel in acidic medium

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ABSTRACT

Corrosion inhibition behavior of orange peel waste extracts in 1 M hydrochloric acid medium on mild steel surface was studied using weight loss studies, electrochemical impedance spectroscopy (EIS), potentiodynamic polarization and surface phenomena using scanning electron microscopy (SEM) analysis. Better inhibition efficiency for orange extracts was achieved by weight loss measurements. The maximum inhibition efficiency of 99.19% was achieved for 0.25% of waste extract at room temperature. EIS analysis revealed that increase in concentration of orange peel extract (OPE) increases the charge transfer resistance with the continuous decrease in capacitance which confirms the development of dense protective layer on the metal surface at the electrolyte interface. Both the electrochemical investigation such as potentiodynamic polarization and impedance analysis confirmed the improved inhibitory efficiency of OPE by following Langmuir isotherm model. SEM analysis confirms that the corrosion inhibition of OPEs was proceeded by forming an organic protective layer over the mild steel surface by means of the adsorption process by physical contact. These results confirm that the ethanolic extract of orange waste can be used as a potential corrosion inhibitor in industrial applications.



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KEYWORDS Inhibition; EIS; adsorption isotherm



1. Introduction

Corrosion of mild steel due to acidic attack is one of the universal problems with financial implications due to the necessity of mild steel in several industries, for example, oil and gas transportation, chemical industries, fuel refineries, etc. Corrosion may be caused by chemical cleaning, pickling and rescaling of the metals in these industries [1]. As a result, prevention against corrosion in acid atmosphere is desired. Utilization of chemical inhibitors is one of the most successful ways to avoid deterioration of materials. Inhibitors are mostly employed to trim down the attack of corrosive ions present in the solution which is in contact with the metal substrates. The organic molecules consisting of heteroatoms with unsaturated bonds were commonly employed as inhibitors for mild steel. The usefulness of these synthesized organic inhibitors is allied to the occurrence of functional groups with sulfur, oxygen and nitrogen heteroatoms in their structure [2–5]. On the other hand, nearly every organic compound is not only costly but also noxious to mankind and the surroundings. To triumph over this problem, a detailed analysis of new commercially cheap, non-hazardous and ecological corrosion inhibitors is vital. This biologically mediated innovative type of corrosion inhibitors can result from the available plant sources. Extracts of plant sources are considered as an extremely wealthy source of various chemical compounds like alkaloids, flavonoids and phenolic compounds that can be derived by easy methods [6]. Currently, several plant sources

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© 2018 The Author(s). Published by Informa UK Limited, trading as Taylor & Francis Group on behalf of The Korean Ceramic Society and The Ceramic Society of Japan This is an Open Access article distributed under the terms of the Creative Commons Attribution License (http://creativecommons.org/licenses/by/4.0/), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited. have been studied as efficient corrosion inhibitors for mild steel in acidic environments. Various parts of the plant and fruit extracts like *Justicia gendarussa*[7], *Mansoa alliacea*[8], *Chenopodium Ambrosioids*[9], *Pimenta dioica*[10], *Grape Pomace*[11], *Dodonaea viscose*[12], *Aniba rosaeodora*[13], *Geissospermum laeve* [14], *Aloe*[15], *Psidium Guajava*[16], *Pterolobium hexapetalum*[17], *Azadirachta indica*[18], *Celosia argentea* [17], *peltophorum ptetocarpum*[19] and *Phyllanthus amarus* [20] were used as corrosion inhibitors.

This study aims to explore the corrosion inhibition of ethanolic extract of orange peel on mild steel specimen in hydrochloric acid medium. The corrosion resistance behavior of the extract was examined by weight loss studies, electrochemical techniques such as impedance spectroscopy and potentiodynamic polarization studies. The surface of mild steel covered by different concentrations of inhibitors was confirmed by SEM analysis. In addition, adsorption isotherm plots were analyzed to fit with the isotherm model.

2. Experimental procedure

2.1. Materials

2.1.1. Inhibitor preparation

Commercially available orange fruits were purchased from local market. Orange peel was taken off, sliced into small fragments, cleaned and dried for further process. Dried samples were made into fine powder. About 50 g of powdered peels were refluxed at 60°C for 8 h using 150 mL of ethanol and then filtered. The extract obtained was dried under reduced pressure using rotary evaporator and stored in refrigerator (4°C). The high gummy liquid extracted was used for corrosion inhibition studies in acidic medium at various concentrations.

About 1 μ L of the peel extract was diluted in the ethanol and was analyzed by gas chromatography-mass spectrometry (GC-MS) using Clarus 680 GC with a fused silica column. The column is packed with Elite-5MS (5% biphenyl and 95% dimethylpolysiloxane, having the sample size of 30 m × 0.25 mm ID × 250 μ m df). Finally, the components were removed using He as carrier gas followed at a constant flow of 1 mL/min. The temperature of oven was adjusted as 60°C (2 min), followed by 300°C at the rate of 10°C min⁻¹, injector temperature of 260°C and detection temperature of 240°C. The GC-MS spectrum obtained for the orange peel extract (OPE) was compared with the database spectrum stored in the GC-MS NIST (2008) library.

The OPEs were also screened using the fourier transform-infrared (FT-IR) spectroscopy (Schimadi-IR affinity). The IR graph was obtained in the range of $4000-400 \text{ cm}^{-1}$ and analyzed for characteristic functional groups responsible for corrosion inhibition.

2.1.2 Substrate preparation

Mild steels of the composition Fe-99 146%, P 0.039%, Si 0.06%, Mn 0.55%, C 0.205% were used for the current study. Prior to experiments, each sample was successively polished with silicon carbide sheets in the range from 120, 200, 400, 600, 800, 1000 and 1200 and finally degreased with acetone. Further, the samples were washed with double-distilled water followed by drying in oven.

2.1.3 Preparation of electrolyte

The suitable concentration of 1 M hydrochloric acid was prepared using deionized H_2O and used for corrosion studies. The OPE of concentration ranging from 0.03%, 0.07%, 0.1% to 0.25% were prepared using 1 M HCl.

2.2 Methods

2.2.1 Weight loss studies

Mild steel coupons of dimension 3 cm \times 2.5 cm \times 0.5 cm were weighed initially and immersed in 80 mL of 1 M HCl solution for 12 h with and without the addition of different concentrations of OPE. The experiment was conducted in triplicates to avoid the error during analysis. At the end of exposure period, specimens were washed and their final weight recorded. The inhibition efficiency (IE%) was calculated in percentage according to Equation 1:

$$IE(\%) = (W_1 - W_i) / W_1 \times 100 \tag{1}$$

where W_1 = change in weight without inhibitor in milligram, W_i = change in weight with inhibitor at i concentration in milligrams.

The corrosion rate was determined using the following formula:

$$CR = \Delta W / A t (mg cm^{-2}h^{-1})$$
 (2)

where ΔW = change in weight (mg), A = area of the sample exposed (cm²) and t = exposed time (h).

2.2.2 Electrochemical measurements

The electrochemical behavior for the mild steel specimens in the presence and absence of OPEs was conducted using Biologic SP-150 Potentiostat, France. A three-electrode set-up was used for the study having mild steel specimen of dimension 1×1 cm² as the working electrode, thin platinum wire as auxiliary electrode and a silver/silver chloride electrode (Ag/AgCl) as reference electrode. All the experiments were done at room temperature by immersing in 80 mL of 1 M HCl in static condition. Prior to each experiment, the substrate was permitted to corrode generously and its open circuit potential (OCP) was recorded as a function of time up to 30 min to obtain a equilibrium between the electrode and the electrolyte. Potentiodynamic polarization curves were acquired by sweeping the electrode

potential from -300 mV to +300 mV with respect to OCP at a scanning rate of 1 mV s⁻¹. The corrosion potential and current densities were derived by potentio-dynamic polarization extrapolation method using software. The above process was repetitive for each concentration of OPE. The electrochemical impedance spectroscopy (EIS) studies were conducted with a frequency range from 100,000 Hz to 0.01 Hz by applying AC signal with a small amplitude of 10 mV at a scan rate of 10 points per decade.

2.2.3 Surface analysis

The variation in the surface of the metal was analyzed by the Scanning Electron Microscope (Carl Zeiss Supra-55 SEM). Steel specimen of size 1 cm \times 1 cm \times 0.5 cm was immersed in electrolyte for 12 h in normal condition in absence of inhibitor as considered as control and the specimen exposed at optimum concentrations of inhibitor possess higher corrosion rate by weight loss method was considered for SEM analysis. Further, the specimens were removed, cleaned with deionized water, dried and analyzed by SEM.

3. Results and discussion

3.1 Characterization of orange peel extract

IR spectra recorded for OPE are depicted in Figure 1. An absorption peak at 3325 cm⁻¹ is assigned for linked OH⁻ groups. The peaks at 2971 and 2880 cm⁻¹ correspond to aliphatic stretching and aromatic stretching of C–H groups. The peaks at 1733, 1650 and 1043 cm⁻¹ are attributed to the stretching vibration of C=O groups and C=C groups. The stretching and bending mode of C–C group was confirmed by the peaks present at the wavenumbers 1372 and 1088 cm⁻¹. Peaks at 878 and 629 cm⁻¹ are due to the stretching mode of C–H groups. This confirms that the OPE consists of mixture of compounds like poly phenolic, flavonoids and

carbonyl crowd-containing compounds with unsaturated bonds, oils and pectin, which was also supported by pervious reported literature [21–23]. For further confirmation, the GC–MS analysis was done and the chromatogram was matched with GC–MS NIST (2008) library. The presence of four major compounds in orange peel extract were summarized in Table 1. The corresponding chromatograph is shown in Figure 2 containing unsaturated group, phenolic groups and heteroatoms which provide active sites for the adsorption of the inhibitors on the surface.

3.2. Weight loss studies

Rate of corrosion (mg cm⁻²h⁻¹) and inhibition efficiency (%) of mild steel exposed to 1 M HCl at different concentrations of OPE inhibitors are shown in Figure 3. It is experiential that corrosion rate for the steel specimen was found to be decreased with the consecutive enhancement in inhibition effectiveness on increasing OPE concentration from 0.03% to 0.25%. This performance is may be due to the increase in the adsorption of OPE at the metal/electrolyte boundary on increasing its concentration. This inference established that OPE acts as an efficient corrosion inhibitor.

3.3. Electrochemical studies

3.3.1 Potentiodynamic polarization studies

The potentiodynamic polarization plots for mild steel substrate in acidic medium with and without various concentrations of OPEs at room temperature are shown in Figure 4. Kinetic factors such as cathodic slope constant (b_c), anodic slope constant (b_a), corrosion potential (E_{corr}) and current density (I_{corr}) were deliberate from the extrapolation of anodic and cathodic curves and given in Table 2. The anodic b_a and cathodic b_c slope values were mutually changed with addition of inhibitor molecule to the electrolyte. The cathodic slope



Figure 1. IR spectra for orange peel ethanolic extract.

Table 1. Chemical components identified in ethanolic extract of orange peel by GC-MS.



value b_c was decreased by increasing the concentration of inhibitor, which shows that the adsorption of the inhibitor molecule was on the cathodic site and acts as a barrier from the attack of aggressive ions. In general, an inhibitor can be categorized as anodic, cathodic and mixed form, which was determined by the shift of E_{corr} value toward the desired direction in comparison to the E_{corr} value obtained in the absence of inhibitor [24,25]. The shift in the corrosion potential value (Ecorr) of more than 85 mV towards anodic and cathodic direction proved that the OPE acts either as anodic or as cathodic inhibitor. However, if difference exists within 85 mV, it can be assumed to be as mixed type of inhibitor. It was found that the values of E_{corr} are shifted toward the negative direction by increasing the concentrations of inhibitors. Even though there was a shift in E_{corr} the maximum shift in the potential value with addition of inhibitor was 72 mV; this behavior shows that the orange peel is a mixed-type inhibitor. It is also evident from Figure 4 that the presence of



Figure 2. GC–MS chromatograph of orange peel extract.



Figure 3. Variation of corrosion rate and inhibition efficiency as the function of concentration.

extract peels alters mutually both cathodic and anodic slopes with increasing concentration of the inhibitors. The OPEs influence the potentiodynamic polarization curves signifying that the extracts control the dissolution ions from the metal and the hydrogen evolution, implying that the peel extract acted as a mixed form of inhibitor. The corrosion current density (I_{corr}) values was found to be decreased with increase in inhibitor concentration, governing the increased inhibition effectiveness with respect to concentration. The inhibition phenomena of orange peel extract proceeds by adsorbing on the metal substrate and acts as a barricade that prevents the mild steel surface from the attack of the destructive species.

3.3.2 Electrochemical impedance studies

The electro kinetic behavior and pathway for mild steel in the presence of OPEss was inspected by EIS. The EIS study for mild steel substrate was done after 60 min of immersion in electrolyte in the absence and presence of inhibitors at different concentrations and the experiment was executed at the frequency range between 1,00,000 Hz and 0.1 Hz and is shown in Figure 5. The derived impedance spectra were fitted using simple equivalent circuit model consisting of solution resistance (R_s) , charge transfer resistance (R_{ct}) and constant phase elements (CPE) and are mentioned in Figure 6. The constant phase elements (Y_o) and n demonstrate the homogeneity of the electrode surface in the absence and presence of inhibitor in corrosive media. These electrochemical parameters were derived from this study using the Z fit software and summarized in Table 3. The value of double layer capacitance (C_{dl}) is as follows:

$$C_{dl} = (Y_o . R_{ct}^{1-n})^{1/n}$$
(3)

It is observed from Figure 5a. Nyquist plots show depressed semicircles demonstrating that the corrosion of mild steel was principally controlled through the charge transfer process. In verity, the R_{ct} of the steel



Figure 4. Potentiodynamic polarization slopes of mild steel in 1 M HCl at different concentrations of OPE.

Table 2. Values of the polarization data obtained for MS in 1 M HCl solution with and without inhibitors.

Concentration of OE (%)	E _{corr} (mV vs. Ag/AgCl)	<i>b_c</i> (mV)	<i>b_a</i> (mV)	l _{corr} (μA cm ²)	Surface coverage (θ)	Inhibition efficiency (%)
Control	-451	167	156	1908.67	-	-
0.03	-500	158	156	471.82	0.7528	75.28
0.07	-529	202	200	200	0.8952	89.52
0.1	-517	136	213	147	0.9229	92.29
0.25	-523	116	233	88.41	0.9536	95.36



Figure 5. EIS studies of mild steel in 1 M HCl with different concentrations of OPE: (a) Nyquist plots, (b) Bode plot, (c) phase angle plots.

electrode was found to be enhanced in the presence of OPE inhibitor. There was an increase in the diameter of the capacitive loop with increased inhibitor level. Table 3 is evident that the increase in inhibitor concentration leads to increase in the charge transfer resistance. This increased R_{ct} favors the adsorption of the inhibitor compound on the metal/electrolyte interface. Additionally,

the *n* value talks about the homogeneity and capacitive behavior of the metal substrate. The lower *n* value corresponds to the non-homogeneity and surface roughness of the mild steel due to damage caused by corrosive media in the absence of inhibitor. Table 3 clearly shows an increment in the *n* value from 0.810 to 0.845 upon increasing the inhibitor concentration that clearly infers



Figure 6. The simple equivalent circuit used to fit the EIS data.

Table 3. Values of the EIS data obtained by fitting for MS in 1 M HCI medium at different concentrations of inhibitors.

Concentrations of OE (%)	R_{ct} (Ω cm ²)	$C_{dl} ~(\mu F ~ cm^{-2})$	п	Surface coverage (θ)	Inhibition efficiency (%)
Control	18.64	395	0.810	-	-
0.03	137.3	206	0.829	0.8642	86.42
0.07	216.6	167	0.846	0.9139	91.39
0.1	367.3	68	0.845	0.9492	94.92
0.25	380	64	0.841	0.9510	95.10

the decrease in non-homogeneity on the metal surface due to formation of protective film by adsorption of inhibitor molecule. The values of electrical double layer were decreased with increase in percentage of inhibitor. This is attributed to the increase in thickness of the protective layer formed on the metal surface with the upsurge in the inhibitor concentration. The decrease in the C_{dl} value is due to the replacement of the adsorbed water molecules and aggressive ions with the inhibitor molecules at the metal/electrolyte junction. From the above discussion, it was confirmed that the inhibition of corrosion took place by adsorption on metal surface.

The Bode and phase angle plots obtained at different concentrations of OPE are shown in Figures 5b & 5c, respectively. Bode plots provide the impedance value with respect to frequencies. Bode curves show higher impedance values at lower frequencies for higher inhibitor concentration respectively. This confirms the higher shielding effect of inhibitors molecules on the mild steel surface, which is correlated to the adsorption of the OPE compounds over metal surface. According to the phase angle plots (Figure 5c), increase in the concentration of inhibitors in the electrolyte medium results in rise of the phase angles closer to 90°, which signifies more inhibitive performance due to adsorption of the inhibitor molecules on the surface of the mild steel at higher concentrations. Inhibition efficiency was derived using the following formula:

$$IE(\%) = (R_{ct(i)} - R_{ct(0)}) / R_{ct(i)} \times 100$$
(4)

where

 $R_{ct(i)}$ = with inhibitor at ith concentration, where R_{ct} refers to charge transfer resistance, $R_{ct(0)}$ = charge transfer resistance without inhibitor.

The obtained results confirm that there was an improvement in the IE (%) and degree of surface

coverage with increase in level of inhibitor concentration. This increase in the surface coverage with respect to increase in the inhibitor concentration leads to better protection efficiency on the MS surface from the attack of acidic medium. The maximum inhibition efficiency of 99.19% was achieved at 0.25%.

3.4 Adsorption isotherm studies

Adsorption isotherms were employed to understand the interaction between inhibitor and the surface of MS substrates. The degree of surface coverage (Θ) obtained from the various methods such as EIS, potentiodynamic polarization and weight loss studies was used to evaluate the best isotherm that fits into the information obtained. Langmuir, Freundlich and Temkin were employed to establish the isotherms most appropriate to experimental data. The linear correlation coefficient with R^2 values which is nearer to unity was taken to define the type of adsorption process. Langmuir isotherm model which shows good fit with the values was considered for the current study. Langmuir adsorption process speaks only about the interaction between the inhibitor molecules and the metal substrates and not between the inhibitor molecules.

$$C/\theta = 1/K_{ads} + C \tag{5}$$

where C = concentration of OPE in g L⁻¹, $\Theta =$ surface coverage and $K_{ads} =$ adsorption equilibrium constant. The plot of C/Θ versus C yielded linear graph that is shown in Figure 7. This explains that the adsorption of the inhibitor molecules on the mild steel surface is reliable on Langmuir adsorption isotherm model and the correlation coefficient (R^2) obtained are near to unity.



Figure 7. Langmuir isotherm plot of MS in 1 M HCl at different concentration s of inhibitors.

Table 4. Values of activation parameters obtained for MS in 1M HCl solution at various concentration of inhibitor.

Method	Slope	Y-intercept	R ²	$\ln K_{ads}$ (L g ⁻¹)	ΔG_{ads} (kJ mol $^{-1}$)
Weight loss	1.00589	0.00698	0.9999	143.2	-29.61
EIS	1.03738	0.03185	0.9998	31.3	-25.81
TAFEL	1.01470	0.07983	0.9997	12.52	-23.53

The K_{ads} is reported to be related with the standard free energy of the adsorption process. The value of K_{ads} obtained can be used to calculate the standard free energy (ΔG_{ads}) via the following equation:

where ΔG_{ads} = standard free energy of adsorption process, K_{ads} = adsorption equilibrium constant, R = gas constant (8.314 J K⁻¹ mol⁻¹), T = temperature (300 K), A = water density (1000 g L⁻¹). Calculated values of K_{ads} and ΔG_{ads} are listed in Table 4.





Figure 8. SEM images of MS in 1 M HCl solution immersed for 12 h. (a) Polished specimen, (b) without inhibitor, (c) with 0.25% OPE.

The calculated values of standard free energy of adsorption process were negative for all methods used and range from -23.53 to -29.61 kJ mol⁻¹ for OPE. The negative value of ΔG_{ads} indicates that the adsorption of inhibitor on the MS substrates occurs spontaneously. Usually, values of standard free energy values lesser or closer to -20 kJ mol⁻¹ are reliable with electric charges between molecules in inhibitor and surface charged steel substrate (physisorptions) while those more negative than -40 kJ mol⁻¹ involves sharing of electrons from the inhibitor molecules to the metal surface to form a chemical bonding (chemisorptions) [26-28]. In the case of OPE, ΔG_{ads} value of the inhibitor was found to be from -23.53 to -29.61 kJ mol⁻¹, this indicates the phenomena of adsorption of inhibitor on the metal surface by physical adsorption process.

3.5 Surface analysis

The changes that take place on the surface of the mild steel specimen were studied in 1 M HCl solutions in the absence and in the presence of OPE after 12 h immersion time and are given in Figure 8a, 8b & 8c. The mild steel surface immersed in the acidic media in the absence of the inhibitor observes clear corrosion pits on the surface due to generalized corrosion damage caused by the aggressive ions. These corrosion damages are observed to be missing from the face of the steel substrates in the presence of OPEs in the acid solution (Figure 8c). The surface morphology of the specimens exposed in acidic solutions with the peel extracts (Figure 8c) is observed to be very much similar to that of the polished specimens (Figure 8a). This signifies that the development of shielding layer by the inhibitor on the metal surface acts as a blockade of corrosion attack by the acid solutions.

3.6 Mechanism of inhibition

The peel extract used in our work consists of mixtures of chemical compounds in nature. The photochemical constituent of the ethanolic extract of orange peel is given in Table 1. The corrosion of mild steel in 1 M HCl solution containing OPEs can be inhibited due to the adsorption of various chemicals present in peel extracts through their lone pair of electrons and pi-electrons interacting with the vacant d-orbital present in iron. The heteroatom "O" and pi-electrons of the organic compounds act as the reaction sites in adsorption process. The presence of four major organic compounds in the orange peel extract confirmed by GC-MS analysis is shown in Table 1. It is motivating that all the recognized compounds from peel extracts contained oxygen and pi-electrons in their molecular structure. This is evident that the corrosion inhibition of steel substrates mediated through peel extract might be attributed to the adsorption of the naturally available organic structures containing O or pi-electrons in their molecules. Thus, this inhibition phenomenon is

highly complex due to the presence of mixture of chemical constituents. According to the previous reported literature, and also form GC–MS library search, OPE of ethanol contains limonene as major compound which is an alicyclic alkene. As the calculated ΔG_{ads} free energy change ranges between –23 and –30 kJ mol⁻¹, it is clear that the interactions of OPE with metal surface are neither ionic nor chemical interaction. Hence, it might be attributed to the interaction between pi-electron of alkene on the mild steel surface through physical process.

$$\leftrightarrow Inhibitor_{(Sol)} + nH_2O_{(ads)}Inhibitor_{(ads)} + nH_2O_{(Sol)}$$
(7)

As seen above, the inhibition of mild steel is mainly endorsed due to the presence of heteroatom containing organic compounds in the OPE. The formation of protective film was observed due to the adsorption of inhibitor compounds on the metal surface. This suppresses the ionic diffusion toward or from the mild steel surface. The exchanges of the inhibitor molecules with adsorbed H2O molecules present on the metal surface protecting it from further attack of the aggressive electrolytes. This barrier effect reduces the metal atoms participating in electrochemical process and consequently increases the corrosion effectiveness.

4. Conclusions

The corrosion inhibition and adsorption studies of OPE on MS in acidic media was examined. The outcome shows the following:

- The presence of phenolic groups -OH, carbonyl groups C=O and unsaturated groups C=C, C=H containing compounds with heteroatoms were identified in the ethanolic OPE. These functional groups act as an active site for adsorption process on the metal substrates.
- (2) The corrosion rate and inhibition efficiency of the mild steel in acidic media increases with the increase in concentration of OPE. The maximum efficiency of 99.19% was achieved for 0.25% of peel extract.
- (3) The potentiodynamic polarization studies conclude that OPE performed as a mixed form of inhibitor.
- (4) According to EIS, there was an increase in polarization resistance (R_{ct}) and decrease in double layer capacitance (C_{dl}) with respect to increase in concentration of OPE. This behavior was found to be due to the formation of dense protective layer on the metal/electrolyte interface.
- (5) The adsorption process followed the Langmuir isotherm model. The negative value of ΔG_{ads} concludes that the adsorption process was spontaneous and proceeds through physisorptions.

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 - (6) The surface morphology studies of the mild steel substrates by scanning electron microscope also conclude the formation of protective layer.

Thus, the overall results conclude that the OPE can be effectively used as a green corrosion inhibitor.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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