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Opuntiol: An Active Principle of *Opuntia elatior* as an Eco-Friendly Inhibitor of Corrosion of Mild Steel in Acid Medium

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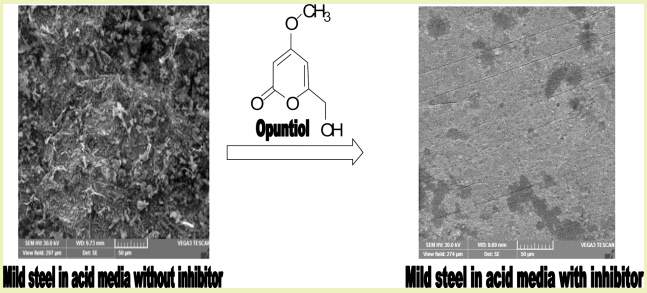
Supporting Information

ABSTRACT: The anticorrosion ability of *Opuntia elatior* fruit extract was tested on mild steel (MS) in 1 M HCl and H_2SO_4 media by a weight loss method at various temperatures, electrochemical experiments such as potentiodynamic polarization (PDS) and electrochemical impedance spectroscopy (EIS), and surface characterization techniques using scanning electron microscope (SEM) and X-ray diffraction (XRD) studies. The major phytoconsituent, opuntiol, was isolated chromatographically and characterized by infra-red (IR) and nuclear magnetic resonance (NMR) spectroscopic studies. Further, its corrosion inhibitive effect was investigated by PDS, EIS, SEM, and XRD studies. The results of the weight loss studies indicated that inhibition efficiencies were enhanced with an increase in concentration of extract and decreased with a rise in temperature. Adsorption of the extract on a mild steel surface obeyed the Temkin isotherm. Results of PDS revealed the mixed mode inhibitive action, and results of EIS studies confirmed the adsorption of the extract at the metal−solution interface. Further, SEM and XRD studies clearly revealed the film-forming ability of opuntiol on the surface of mild steel. Thus, the anticorrosion activity of *O. elatior* can be related to the presence of opuntiol.



KEYWORDS: *Opuntia elatior, Opuntiol, Temkin isotherm, Corrosion inhibition, Physisorption*

INTRODUCTION

Mild steel (MS) finds use in extensive industrial applications such as handling of acids, alkalis, and salt solutions. The aggressiveness of these substances causes severe corrosion in the engineering structures of mild steel, which leads to huge financial and material losses. Hence, the study of mild steel corrosion and the inhibition of corrosion of mild steel have invited the attention of scientists and technocrats to device ways to control the corrosion. Among the various corrosion control measures, the use of corrosion inhibitors is a familiar method. It is known that corrosion inhibitors act by adsorbing on the metal surface. They retard the corrosion of metal by reducing the cathodic and/or anodic processes, reducing the rate of diffusion of metal ions, and increasing the metal surface resistance.[1] Many heterocyclic organic molecules having N, S, and O atoms[2,3] have been shown to function as potential corrosion inhibitors. But many of them are highly toxic and raise environmental concerns. The use of plant-based corrosion inhibitors is nowadays very common because they are cost-effective and eco-friendly.[4−9] In our laboratory, we have carried out an anticorrosion study of several plant extracts, namely, *Solanum nigrum*,[10] *Rauvolfia serpentina*,[11] *Piper nigrum*,[12] *Strychnos nux-vomica*,[13] *Datura stramonium*,[14] *Solanum tuberosum*,[15] *Datura metel*,[16,17] *Spirulina platensis*,[18] *Hydroclathrus clathratus*,[19] *Caulerpa racemosa*,[20] *Ervatamia coronaria*,[21] and *Kappaphycus alvarezii*,[22] which have shown excellent corrosion inhibitive effects. Though there are numerous reports available on the natural green inhibitors, only a few reports are available wherein the corrosion protection activity is correlated to the presence of phytoconstituents. Piperine from *Piper nigrum*,[12] brucine from *Strychnous nux-vomica*,[13] and atropine obtained from *Datura stramonium*[14] have been reported to possess anticorrosion effects. Further corrosion inhibitive properties of reserpine obtained from *Rauvolfia serpentina*[11] and caulerpin, a bis-indole alkaloid isolated from a marine alga *Caulerpa racemosa*,[20] have been reported from our laboratory. In continuation of our studies to determine the active principle responsible for the anticorrosion effect of green inhibitors,[10,21] we report here the anticorrosive efficiency of *Opuntia elatior* and correlate its corrosion inhibiting effect to the presence of opuntiol (Figure 1), the major phytoconstituent of *Opuntia elatior* (present in 1% of its dry weight).[23] The other phytoconstituents of *O. elatior* are proline, linolenic acid, campesterol, and betacyanin.[24] The plant showed excellent antibacterial, antiviral, and antioxidant properties.

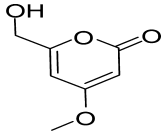


Figure 1. Structure of opuntiol.

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EXPERIMENTAL SECTION

Specimen Composition and Dimensions. Mild steel specimens containing 0.07% of C, 0.34% of Mn, 0.08% of P, and rest iron with a dimension 1.5 cm × 5.0 cm × 0.2 cm were utilized for weight loss measurements. For electrochemical, SEM, and XRD studies, coupons with 1 cm^2 exposed surface area were employed. Prior to use, the specimens were well cleaned, polished, degreased with acetone, and desiccated for further studies.

Inhibitor Preparation. A total of 200 g of dry powdered *O. elatior* fruit was refluxed with 500 mL of ethanol for 3 h to obtain the fruit extract. The extract was evaporated to obtain the solid residue that was further dried and powdered. A known quantity of powdered sample was dissolved in 1 M HCl and H_2SO_4 separately, and solutions of different concentrations were prepared.

Isolation and Characterization of Phytoconstituent. Perusal of the literature showed that opuntiol is the major constituent present in *O. elatior*.[23] The concentrate (20 g) of the fruit extract of *O. elatior* was taken in a small amount of ethanol, adsorbed over a silica gel (60−120 mesh) column, and subjected to gradient elution by varying proportions of petroleum ether and chloroform. Chlorophyll and waxes were removed from the extract by repeated elution with petroleum ether. Elution with chloroform yielded a white solid (0.5 g) that was characterized using IR and NMR spectroscopic techniques.[23]

Weight Loss Method. The pre-weighed MS coupons were placed in 1 M HCl and H_2SO_4 solutions separately for 2 h duration without and with different concentrations of plant extracts at various temperatures, namely, 303, 313, and 323 K. Then, the coupons were properly washed, dried, and reweighed. Using weight loss data, the inhibition efficiency (% IE) and values of surface coverage (θ) were calculated by following equation

$$\mathrm{IE}(\%) = \frac{W_0 - W_i}{W_0} \times 100 \quad (1)$$

where, W_0 is the weight loss value in the absence of inhibitor, and W_i is the weight loss values in the presence of inhibitor.

The corrosion rate (mg m^{-2} s^{-1}) values were obtained from the following equation

$$\mathrm{CR} = \frac{\text{weight loss of mild steel specimen}}{\text{immersion time} \times \text{surface area}} \quad (2)$$

Electrochemical Studies. The PDS and EIS curves were recorded using a CHI electrochemical analyzer (Model No. 760D) with a built-in software. A three-electrode cell setup containing a reference electrode (calomel electrode), auxiliary electrode (Pt electrode), and working electrode (mild steel embedded in a Teflon holder with exposed surface area in a corrosive environment of 0.785 cm^2) was used in the present study. For each experiment, the working electrode was abraded with 1/0, 2/0, 3/0, and 4/0 grade sand papers and washed with distilled water prior to use. Electrochemical studies were carried out for the fruit extract of *O. elatior* as well as for opuntiol. Prior to each PDS and EIS experiment, the working electrode was allowed to stand freely for about 30 min in acid solutions, and its OCP was recorded as a function of time. The steady state OCP corresponding to the corrosion potential of the working electrode was obtained after 30 min of immersion. The Tafel curves were recorded in both cathodic and anodic directions with respect to corrosion potential of the working electrode (OCP ± 300 mV) with 0.5 mV s^{-1} potential sweep. EIS curves were recorded at OCP values of 0.1−10000 Hz frequency range at 2 mV amplitude. The impedance diagrams are presented in Nyquist plots. EC Lab V10.02 software was used to fit and analyze the measured impedance curves. The % IE values were calculated using the following relation

$$\mathrm{IE}(\%) = \frac{R_{ct(i)} - R_{ct(b)}}{R_{ct(i)}} \times 100 \quad (3)$$

where, $R_{ct(i)}$ is the charge transfer values in the presence of inhibitor, and $R_{ct(b)}$ is the charge transfer value in the absence of inhibitor. Figure 2 is the Randle's equivalent circuit used in the present study in which

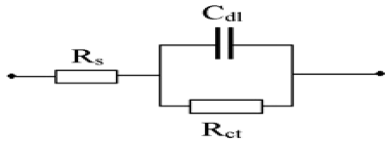

Figure 2. Electrical equivalent circuit.

R_s represents solution resistance, R_{ct} represents charge transfer resistance, and C_{dl} represents double layer capacitance.

Surface Analysis. *SEM Analysis.* Perusal of Tables 1 and 2 show that the maximum IE values were obtained at 500 ppm of *O. elatior* extract and 50 ppm of opuntiol in 1 M H_2SO_4. Hence, it was decided to record SEM micrographs in a H_2SO_4 medium with 500 ppm of *O. elatior* extract and with 50 ppm of opuntiol separately for 2 h as a control study. Then, the MS coupons were taken out and washed with double distilled water. The SEM images were taken using a HITACHI scanning electron microscope (Model S-3000 H).

XRD Measurements. The XRD pattern of MS coupons after the 24 h immersion in an acid medium in the absence and presence of inhibitor was recorded using an inel XRD instrument. The coupons were taken out, rinsed with double distilled water, and properly dried for recording the XRD pattern.

RESULTS AND DISCUSSION

Structure of Phytoconstituent: Opuntiol. The recorded IR spectrum (Figure 3a) of the compound showed a band around 3405 cm^{-1} indicating a monomeric hydroxyl group, and a strong absorption band around at 1690 cm^{-1} revealed the presence of a carbonyl group in the isolated compound. Further, the absorption bands at 2926, 1457, and 1384 cm^{-1} correspond to C−H stretching vibrations. The band at 1099 cm^{-1} is indicative of the presence of a C−O bond of alcoholic group. The band at 2369 cm^{-1} is due to the vibrating frequencies of a O−C═O group. In the ^{1}H NMR spectrum (Figure 3b), signals at δ 6.172 and δ 5.476 ppm showed the presence of a pyrone ring, and a signal at δ 3.835 ppm accounted for the presence of methoxy protons. On the basis of IR and ^{1}H NMR, the isolated compound was characterized as 6-hydroxymethyl-4-methoxy-2H-pyran-2-one (opuntiol). The spectral data correlated well with previous reports.[23,24]

Weight Loss Method. The IE values calculated from weight loss measurements for mild steel in the acid media having various concentrations of *O. elatior* extract at various temperatures such as 303, 313, and 323 K are shown in Figure 4a and b. It is shown in the figure that the plant extract reduced the corrosion of mild steel in acid media markedly. The IE increased with a rise in concentration of the green inhibitor and reached maximum IE at 500 ppm concentration of extract. The increase in IE with the increase in concentration is suggestive of the increase in the extent of protection efficiency of *O. elatior*.

Effect of Temperature. A comparison of the inhibition efficiency of extract on MS in acid solutions (Figure 4a and b) in the absence and presence of various concentrations of extract at various temperatures (303, 313, and 323 K) indicated that IE enhanced with an increase in inhibitor concentrations and decreased with an increase in temperature.

The decrease in IE with a rise in temperature is suggestive of physisorption,[25,26] which could be attributed to the gradual desorption of the extract from the surface of the metal.

Adsorption Isotherm. In order to understand the adsorption mode, various standard adsorption isotherms were fitted using the results of the weight loss study (Table S2, Supporting Information). The best fits were obtained when θ was plotted against ln C that revealed that the green inhibitor obeys the

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Table 1. Effect of Opuntiol on MS in 1M HCl (Electrochemical Studies)

sample no.	conc. (ppm)	b_a mV dec^{-1}	b_c mV dec^{-1}	$-E_{corr}$ mV	I_{corr} μA cm^{-2}	R_{ct} Ω cm^2	C_{dl} μF cm^{-2}	% of IE
1	Blank	95	113	470	446	30	74.15	–
2	10	110	115	465	162	52	68.31	37.03
3	20	99	126	461	141	70	56.60	43.33
4	30	94	122	472	125	76	49.45	48.48
5	40	92	133	481	110	101	44.20	66.66
6	50	90	138	474	97	107	40.89	71.96

Table 2. Effect of Opuntiol on MS in 1M H_2SO_4 (Electrochemical Studies)

sample no.	conc. (ppm)	b_a mV dec^{-1}	b_c mV dec^{-1}	$-E_{corr}$ mV	I_{corr} μA cm^{-2}	R_{ct} Ω cm^2	C_{dl} μF cm^{-2}	% of IE
1	Blank	116	113	452	615	17	106.42	–
2	10	103	118	423	438	27	67.56	42.31
3	20	102	102	431	393	30	57.77	57.14
4	30	82	109	432	354	33	50.33	60.52
5	40	94	110	459	322	51	50.19	70.29
6	50	92	117	439	308	62	36.91	72.58

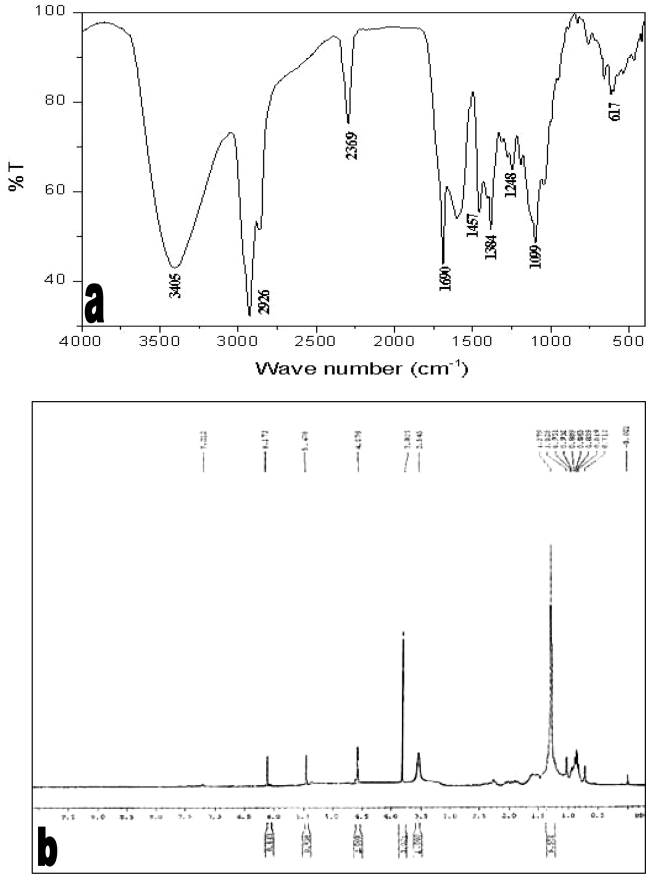

Figure 3. (a) IR spectrum of opuntiol. (b) ^{1}H NMR spectrum of opuntiol.

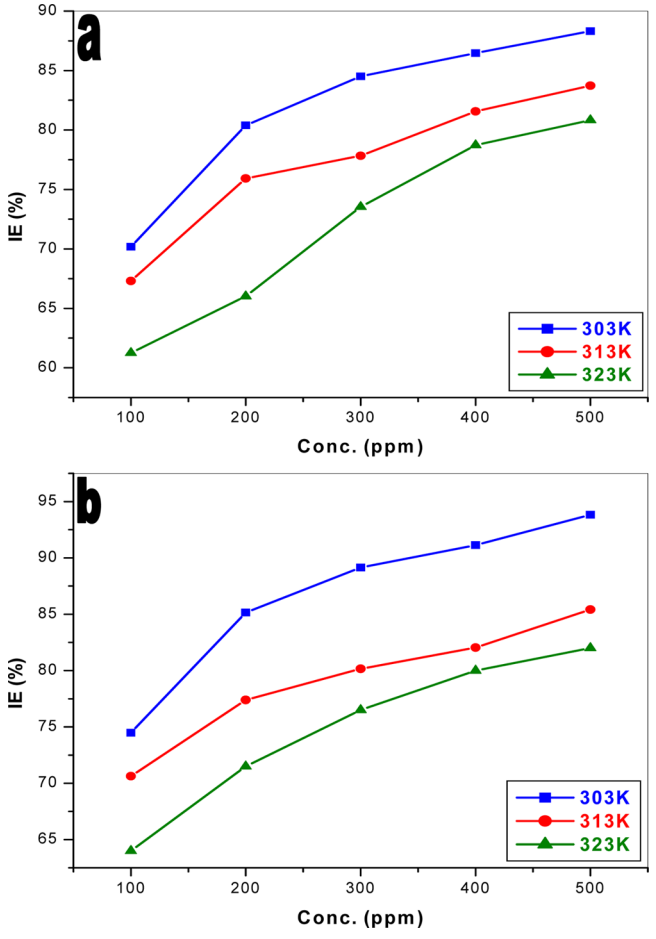

Figure 4. (a) Concentration of *Opuntia elatior* vs % of IE on MS in 1 M HCl. (b) Concentration of *Opuntia elatior* vs % of IE on MS in 1 M H_2SO_4.

Temkin adsorption isotherm (Figure S1, Supporting Information). The values of surface coverage were calculated from the weight loss data using the following relation

$$\theta = \frac{W_0 - W_i}{W_0} \quad (4)$$

where, W_0 is the weight loss value in the absence of inhibitor, and W_i is the weight loss values in the presence of inhibitor.

From the slopes of the isotherm, values of K_{ads} can be obtained using eq 5

$$\exp^{(-2a\theta)} = KC \quad (5)$$

where C is the inhibitor concentration, θ is the degree of surface coverage, and K_{ads} is the equilibrium constant of adsorption process.

The calculated K_{ads} values obtained from the slopes of the isotherm are listed in Table 1. The attraction between the

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adsorbate (inhibitor molecules) and adsorbent (metal) is related to K_{ads} values. The higher values of K_{ads} are an indication of efficient adsorption of inhibitor molecules and high inhibition efficiency.[27] Because the molecular mass of the extract components is not known, the thermodynamic parameters such as the standard free energy of adsorption value cannot be calculated.[19]

Physisorption may occur in the following possible ways: (1) by decreasing the active sites on metal by simple blocking and (2) by increasing the energy barrier of the cathodic and/or anodic processes occurring in acid solutions without inhibitor.[19]

Activation Parameter. Activation energy (E_a) values are essential to know the kinetics of the corrosion process. From the weight loss data obtained for three various temperatures (303, 313, and 323 K), Arrhenius plots of ln CR versus $1000/T$ were drawn (Figure S3, Supporting Information). The E_a values were computed from the slope values of the Arrhenius plots using the following expression

$$\ln \mathrm{CR} = \ln A - \frac{E_a}{RT} \tag{7}$$

where CR = corrosion rate, E_a = activation energy of the corrosion process, R = universal gas constant, T = absolute temperature, and A = Arrhenius pre-exponential factor.

Table S2 of the Supporting Information lists the calculated E_a values. It is apparent from the table that, the calculated E_a values are increased with a rise in inhibitor concentration for both acid media. The changes in E_a values could be due to the fact that on addition of the extract to the acid solution the energy barrier of corrosion process is enhanced, which lead to the inhibition of corrosion. It is evident that the entire process is governed by the surface reaction.[19] Thus, the observed results revealed the inhibitive effect of *O. elatior*.

Electrochemical Studies. *Potentiodynamic Polarization Measurements.* The polarization behavior of MS in acid solutions without and with different concentrations of extracts of *O. elatior* are shown in Figure S3 of the Supporting Information. The electrochemical parameters such as I_{corr}, E_{corr}, and Tafel slopes (b_a and b_c) were calculated by extrapolating the Tafel lines to the corresponding corrosion potential, and they are listed in Tables S4 and S5 of the Supporting Information. From Figure S3 of the Supporting Information, it is clear that both anodic (dissolution of metal) and cathodic (hydrogen evolution) reactions were retarded after the introduction of inhibitor to the acid media. The inhibition of corrosion enhanced with an increase in inhibitor concentration. In the presence of different fruit extract concentrations, the E_{corr} of MS shifted around ±27 mV as compared to the blank in HCl and H_2SO_4. Inhibitors are said to be cathodic or anodic, only if the E_{corr} values are displaced more than ±85 mV as compared to the blank.[28] This indicates that *O. elatior* inhibited the corrosion of MS in mixed mode. The cathodic domain curves appeared as parallel lines indicating that the introduction of extract to the acidic media did not alter the hydrogen evolution mechanism, and the reduction of H^+ ions at the surface of metal takes place mainly through a charge transfer mechanism.[29,30] The addition of plant extracts shifts the anodic Tafel curves thereby affecting the metal dissolution also. The I_{corr} values decreased with an increase in *O. elatior* concentration thus decreasing the corrosion reaction. The randomly changed Tafel slopes indicate that the added extract simply blocks the reactive sites in the metal surface suggesting that the fruit extract acts in a mixed mode way.[31] Similar results have also been observed for MS in acid solutions without and with various concentrations of opuntiol. The observed polarization behavior of opuntiol is depicted in Figure 5, and the calculated

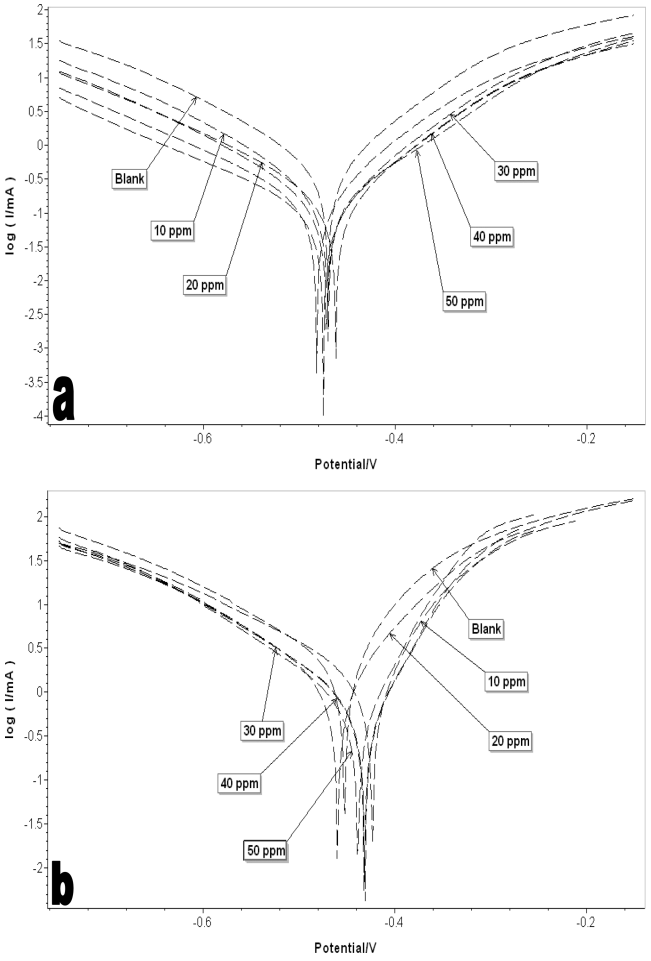

Figure 5. (a) Tafel plots of opuntiol on MS in 1 M HCl. (b) Tafel plots of opuntiol MS in 1 M H_2SO_4.

parameters are listed in Tables 1 and 2. The comparison of Tafel curves of *O. elatior* and opuntiol on MS in acid solutions is given in Figure 6. The inhibition of corrosion of MS is more in the presence of *O. elatior* as compared to the presence of opuntiol. This is due to the presence of other phytoconstituents in addition to opuntiol in *O. elatior* extract.

Electrochemical Impedance Spectroscopy. Nyquist plots for mild steel in 1 M HCl and H_2SO_4 in the absence and presence of fruit extract are depicted in Figure S4 of the Supporting Information. R_{ct} and C_{dl} values obtained from the plots are given in Tables S4 and S5 of the Supporting Information. The electrochemical impedance spectra given as Nyquist diagrams are characterized by one depressed capacitative loop. The deviation from semi-circular shape is due to the dispersion of angular frequency that resulted from the surface roughness and inhomogeneities of the solid surface.[32] The impedance of MS in both acid media markedly increased after the addition of *O. elatior* extract and opuntiol. The R_{ct} values of the inhibited solutions increased with a concentration in inhibitor. This is due to the protective film formed at the mild steel–acid interface. The C_{dl} values were decreased with an increase in concentration of extract. The

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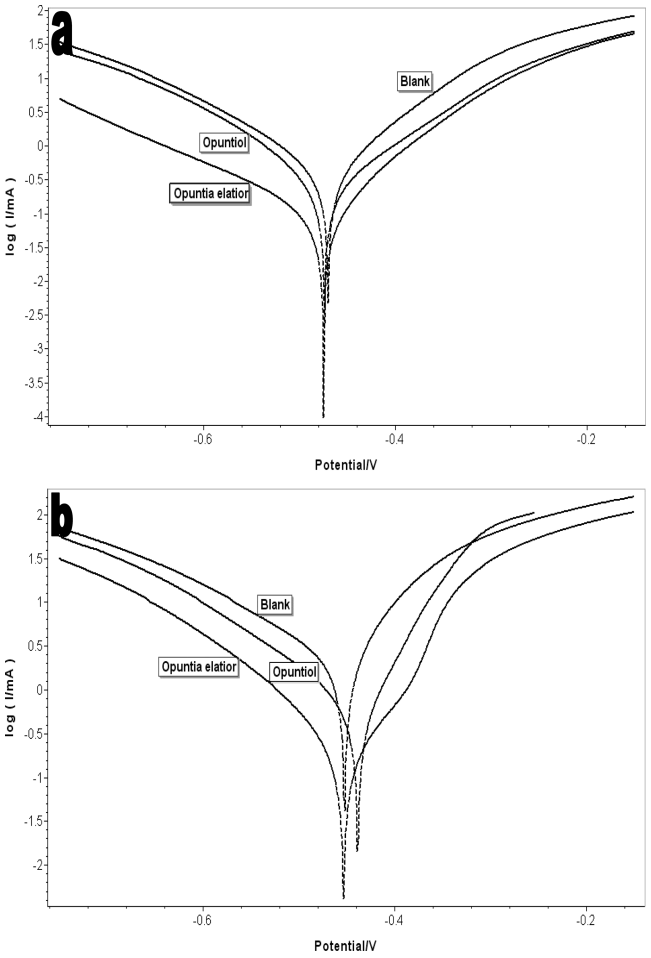

Figure 6. Comparison of Tafel plots of *O. elatior* and opuntiol on MS in (a) 1 M HCl and (b) 1 M H_2SO_4.

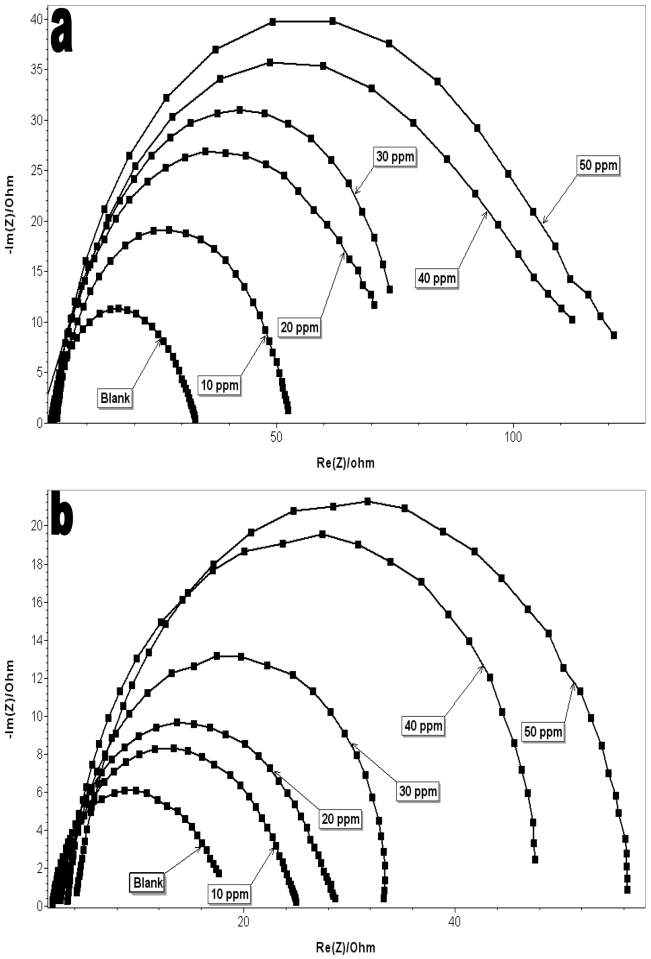

Figure 7. (a) Nyquist plots of opuntiol on MS in 1 M HCl. (b) Nyquist plots of opuntiol on MS in 1 M H_2SO_4.

protective layer thickness (d) is associated with C_{dl} according to eq 8.[25]

$$C_{dl} = \frac{\varepsilon^0 \varepsilon}{d} S \tag{8}$$

where d = thickness of the protective film, S = electrode surface area, ε° = permittivity of the air, and ε = local dielectric constant. Similar results have also been observed for MS in both acid media in the absence and presence of various concentrations of opuntiol. The observed impedance behavior of opuntiol is depicted in Figure 7, and the impedance parameters are listed in Tables 1 and 2.

A decrease in C_{dl} occurs when the opuntiol with low dielectric constant replaces the high dielectric constant water molecules on the surface of MS. Moreover, the double-layer capacitance and thickness of the double layer are inversely related to one another. Thus, a decrease in the values of C_{dl} could be due to the adsorption of opuntia extract and opuntiol on the metal surface.[33] From this observation, it is confirmed that the opuntia extract and opuntiol are adsorbed at the mild steel−acid interface.[2]

The adsorption of inhibitor molecules over the metal surface can be effected in one or more of the following ways: (1) van der Waals (electrostatic) interaction between charged species and the charged metal, (2) interaction of metal with the unshared electron pairs of inhibitor molecules, (3) interaction of metal with π electrons of inhibitor molecules, and (4) and all the above interactions.[34]

Figure 8 shows the comparison of impedance curves of MS in acid media with *O. elatior* extract and opuntiol. It is shown in the figure that the diameter of the capacitive loop of MS is higher in the presence of *O. elatior* extract than opuntiol. This could be due to the synergistic influence of other phytoconstituents such as sterols, aminoacids, and betacyanin in addition to opuntiol in the *O. elatior* fruit extract.

The inhibition efficiency of *O. elatior* and opuntiol is higher in 1 M H_2SO_4 than in 1 M HCl, which could be attributed to the greater availability of active sites on the surface of MS for adsorption in a H_2SO_4 solution due to the lesser adsorptive strength of the sulfate ions on MS.[31]

Comparison of the IE of opuntiol with that of *O. elatior* (Table 3) very clearly reveals that the active principle responsible for the anticorrosion effect of *O. elatior* could be opuntiol. At 500 ppm concentration, the *O. elatior* produced nearly 83% inhibition, while opuntiol even at 50 ppm concentration could produce 72% inhibition. Thus, the anticorrosion effect of the extract could be correlated to the presence of phytoconstituents such as opuntiol. It is the synergistic influence of the various phytoconstituents that makes the extract a highly potent anticorrosion agent.

Surface Analysis. *SEM Analysis.* Figure S5a−c of the Supporting Information show the SEM images corresponding

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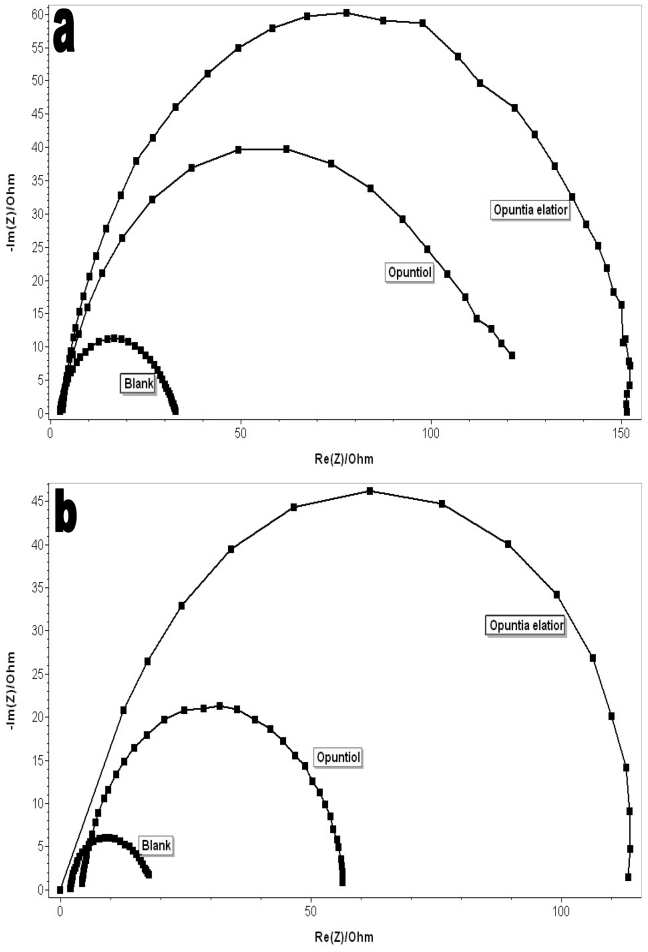

Figure 8. Comparison of Nyquist plots of *O. elatior* and opuntiol on MS in (a) 1 M HCl and (b) 1 M H_2SO_4.

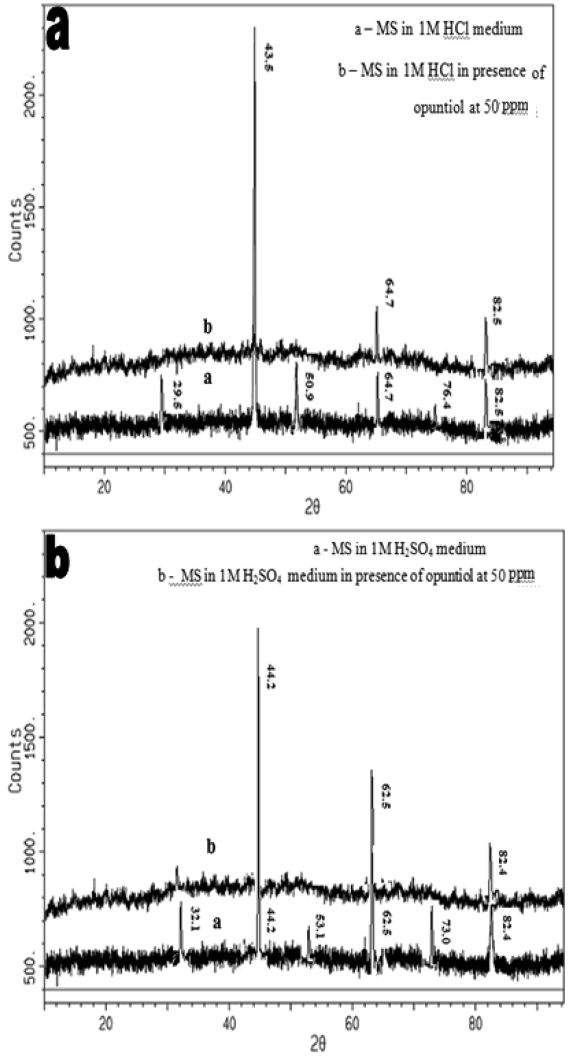

Figure 9. (a) XRD pattern of the film formed on mild steel in 1 M HCl. (b) XRD pattern of the film formed on mild steel in 1 M H_2SO_4.

to the brightly polished mild steel specimen, MS in 1 M H_2SO_4, in the absence and presence of 50 ppm of opuntiol.

Figure S5b of the Supporting Information shows the damaged MS surface caused by the aggressive attack of acid solution. The surface damage of MS was diminished markedly after the addition of 50 ppm of opuntiol as evident from Figure S5c of the Supporting Information. Therefore, it could be concluded that adsorption of opuntiol formed the protective layer over the surface of MS. The observation supported the anticorrosion potential of opuntiol against MS corrosion in acid solutions.

XRD Analysis. Figure 9a and b shows the recorded XRD patterns of the MS surface without and with opuntiol in 1 M HCl and 1 M H_2SO_4. The peaks due to Fe appeared for 2θ values at 43.5°, 64.7°, and 82.5° and that of iron oxide appeared at 29.5°, 50.9°, and 76.4°. The results revealed that the mild steel undergoes corrosion with the formation of iron oxide (corrosion product) in acid medium. However, the absence of the iron oxide peaks in the presence of opuntiol clearly suggests that added opuntiol protects the mild steel surface by forming film over it thereby hindering the corrosion process.

CONCLUSIONS

The results of the present study revealed that *O. elatior* functioned as a good corrosion inhibitor for MS in 1 M HCl and H_2SO_4 solutions in a concentration-dependent mode. IE of *O. elatior* extract decreases with a rise in temperature, which is suggestive of physisorption. *O. elatior* is adsorbed over a MS

Table 3. Comparison of Inhibition Efficiency of Opuntiol with *O. elatior* Extract

	opuntiol			*O. elatior*		
sample no.	conc. (ppm)	HCl	H_2SO_4	conc. (ppm)	HCl	H_2SO_4
1	10	37.03	42.31	100	56.41	62.02
2	20	43.33	57.14	200	72.22	73.84
3	30	48.48	60.52	300	78.41	79.26
4	40	66.66	70.29	400	78.87	82.11
5	50	71.96	72.58	500	79.72	84.95

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surface obeying the Temkin isotherm. From electrochemical studies, it is apparent that both *O. elatior* and opuntiol function as mixed mode inhibitors in both acid media. SEM and XRD measurements supported the formation of a protective layer by opuntiol on the MS surface. The anticorrosion study of opuntiol clearly revealed its role in the protection of mild steel in acid media. Opuntiol with other phytoconstituents such as proline, linolenic acid, campesterol, and betacyanin inhibit the acid corrosion of mild steel.

■ ASSOCIATED CONTENT

Supporting Information

Results of Temkin adsorption isotherm and Arrhenius plots, electrochemical experiments (PDS and EIS) for MS in 1 M HCl and 1 M H_2SO_4 in the absence and presence of various concentrations of *O. elatior* fruit extract along with the SEM images of MS in 1 M H_2SO_4, and MS in 1 M H_2SO_4 with 500 ppm of *O. elatior* fruit extract and MS in 1 M H_2SO_4 in the presence of 50 ppm of opuntiol. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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