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Research Article

Evaluation of Corrosion Inhibition of Mild Steel in 1 M Hydrochloric Acid Solution by *Mollugo cerviana*

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The inhibiting effect of methanolic extract of *Mollugo cerviana* plant on the corrosion of mild steel in 1 M HCl solution has been investigated by different techniques like potentiodynamic polarization, electrochemical impedance spectroscopy, and weight loss methods for five different concentrations of plant extract ranging from 25 to 1000 mg/L. The results indicated that the corrosion inhibition efficiency increased on increasing plant extract concentration till 500 mg/L and decreased on further increasing concentration. The extract was a mixed type inhibitor with the optimum inhibition concentration of 500 mg/L in potentiodynamic polarization. The adsorption of the plant extract on the mild steel surface was found to obey Langmuir adsorption isotherm. Surface analysis was also carried out to find out the surface morphology of the mild steel in the presence and in the absence of the inhibitor to find out its efficiency. The obtained results showed that the *Mollugo cerviana* extract acts as a good inhibitor for the corrosion of mild steel in 1 M HCl solution.

1. Introduction

Corrosion inhibitors have been widely studied in many industries to reduce the corrosion rate of metal surfaces in contact with aggressive medium [1–3]. Acidic solutions are commonly used in various industries and applications include acid pickling, industrial cleaning, acid rescaling, oil-well acidification, and petrochemical processes [4]. Among the acid solutions, hydrochloric acid is one of the most widely used in the pickling processes of metals.

Use of inhibitors is one of the most practical methods for protection against corrosion especially in acid solutions to prevent metal dissolution and acid consumption [5]. Mild steel in acid solution is widely used in various industrial processes and corrosion of mild steel known to occur in this environment. One of the effective methods to prevent corrosion is the use of organic inhibitors [6]. A great number of scientific studies have been devoted to the subject of corrosion inhibitors for mild steel in acidic media [2, 7–14].

Plant extracts are viewed as environmentally friendly and ecologically acceptable inhibitors. Plant products are low-cost, readily available, and renewable sources of materials. The extracts from their leaves, barks, seeds, fruits, and roots comprise of mixtures of organic compounds containing nitrogen, sulphur, and oxygen atoms and some [15–20] have been reported to function as effective inhibitors of metal corrosion in different aggressive environments. Generally, the inhibitive effect of plant extract is attributed to the adsorption of organic substances on the metal surface, blocking active sites or even forming a protective barrier.

In recent years, natural compounds such as herbal plants are employed as inhibitors in order to develop new cleaning chemicals for green environment. Several studies have been reported in the use of natural products as corrosion inhibitors in different media [21–30].

M. cerviana (L.) Ser. (Molluginaceae) has been widely used as a pot herb, enhances eyesight, reduces body odour, acts as a good antiseptic, and is used in the treatment of cough. The aerial plant of *Mollugo cerviana* was collected near Sathankulam, Tuticorin district, Tamil Nadu, India, in July 2010. The plant was identified and authenticated by Dr. V. Chelladurai, Research Officer (Rted), Botanist, and the specimen was deposited at Department of Botany, Auxilium College, Vellore.

The objective of this study is to evaluate the inhibitive effect of methanolic extract of *M. cerviana* as a green corrosion inhibitor on the mild steel in 1 M HCl solution. The corrosion performance was studied using different techniques like potentiodynamic polarization, electrochemical impedance spectroscopy (EIS), and weight loss methods. The scanning electron microcopy (SEM) was used for its morphological studies.

2. Materials and Methods

2.1. Preparation of the Plant Extract. The fresh aerial parts of *M. cerviana* (500 g) were washed with water, air dried, powdered, and extracted with methanol (Merck) for 24 hrs. The methanolic solution was filtered and refluxed further to get a concentrated solution. The clear dark brown methanolic concentrated solution was dried under vacuum to get a semisolid liquid and it was stored in air tight container and preserved in the refrigerator for the further studies.

2.2. Preparation of Specimen. Mild steel specimens having nominal composition of 0.023% P; 0.04% Si; 0.017% Ni; 0.37% Mn; 0.078% C; 0.02% S; 0.002% Mo; Fe balance were used. Specimens of dimension 3 × 3 × 0.1 cm and 1 × 1 × 0.1 cm were used for weight loss and electrochemical studies. The specimens were embedded in epoxy resin leaving a working area of 1 cm^2. The surface preparation of the mechanically abraded specimens was carried out using different grades of silicon carbide emery paper (up to 1200 grit) and subsequent cleaning with acetone and rinsing with double-distilled water were done before each experiment.

2.3. Electrolyte. The electrolyte of 1 M HCl solution was prepared by diluting 37% HCl (Merck) using double-distilled water which is used as a corrosive solution. The five concentrations of *M. cerviana* extract varied from 25 to 1000 mg/L.

2.4. Electrochemical Measurements. Electrochemical measurements, including potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS), were performed in a conventional three electrode cell using a computer-controlled potentiostat/galvanostat (Autolab PGSTAT 302N potentiostat from Eco-Chemie, Netherlands). Platinum electrode was used as the counter electrode, Ag/AgCl, and 3 M KCl as the reference electrode, and the mild steel specimen was used as a working electrode (WE). The area of the WE exposed to the solution was approximately 1 cm^2.

Before each potentiodynamic polarization (Tafel) and electrochemical impedance spectroscopy (EIS) studies, the electrode was allowed to corrode freely and its open circuit potential (OCP) was recorded as a function of time up to 20 min, which was sufficient to attain a stable state. After this, a steady-state of OCP corresponding to the corrosion potential (E_{corr}) of the working electrode was obtained. The potentiodynamic measurements were started from cathodic to the anodic direction ($E = E_{corr} \pm 250$ mV) at a scan rate of 10 mVs^{-1}. Electrochemical impedance spectroscopy measurements were carried out using AC signals at the amplitude of 10 mV and measurement frequency from 100 kHz–10 mHz at the stable OCP. Fresh solution and fresh mild steel samples were used after each sweep.

2.5. Weight Loss Method. Triplicate steel specimens were immersed in 100 mL of electrolyte with and without the *M. cerviana* extract for 1, 24, and 72 hrs at room temperature. The cleaned specimens were weighed before and after immersion into the corrosive solution. The specimens were removed, rinsed with water and acetone, and dried. The loss of weight of the steel samples was determined using an analytical balance with the precision of 0.0001 ± 0.1 mg. The weight loss measurements were performed according to ASTM standard G 31-72. The percentage inhibition efficiency (IE) was obtained from the following equation:

$$\text{IE} = \frac{W_0 - W_1}{W_0} \times 100, \quad (1)$$

where W_0 and W_1 are the weight of the mild steel in the absence and in the presence of inhibitor.

2.6. Surface Analysis. The surface morphology of mild steel specimens immersed in 1 M HCl in the absence and presence of 500 mg/L of *M. cerviana* extract at room temperature for 2 hrs was studied using Leica S440i, a scanning electron microscope (SEM) operated at a voltage of 20 kV with probe current 100 pA.

3. Results and Discussion

3.1. Potentiodynamic Polarization. The potentiodynamic anodic and cathodic polarization plots for mild steel specimens in 1 M HCl solution with and without the plant extract *M. cerviana* at five different concentrations are given in Figure 1. The respective electrochemical parameters including the values of corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (b_c), anodic Tafel slope (b_a), inhibition efficiency (IE), and surface coverage (θ) are given in Table 1. As the inhibitor concentration increased, the corrosion current densities of mild steel decreased till 500 mg/L of plant extract [25, 26]. This behavior is due to the adsorption of the inhibitor present in the plant extract on mild steel/solution interfaces. In this study, the maximum corrosion potential of mild steel was shifted to 20 mV cathodically with respect to the blank. From the potentiodynamic polarization curves (Figure 1), it is evident that both the anodic and the cathodic reactions are inhibited. So, the *M. cerviana* extracts act as a mixed type inhibitor with predominant cathodic effect.

The polarization resistance increased noticeably with the increase in *M. cerviana* concentration and the percentage of IE values also increased with inhibitor concentrations. The percentage of inhibition efficiency (%IE) was calculated from

TABLE 1: The electrochemical polarization parameters for mild steel in 1 M HCl solution in the absence and in the presence of *M. cerviana* extract at different concentrations.

Concentration of the inhibitor (mg/L)	b_a (mV/dec)	b_c (mV/dec)	E_{corr} (mV)	I_{corr} (mA/cm^2)	% IE	θ
Blank (HCl)	77.98	43.26	−456.93	351.23	—	—
25	68.27	51.43	−439.01	0.07	77.97	0.78
100	77.21	57.12	−447.21	0.05	83.07	0.83
250	92.34	60.40	−441.16	0.04	83.09	0.83
500	125.14	84.56	−436.82	0.03	89.03	0.89
1000	95.6	65.53	−441.13	0.04	88.09	0.88

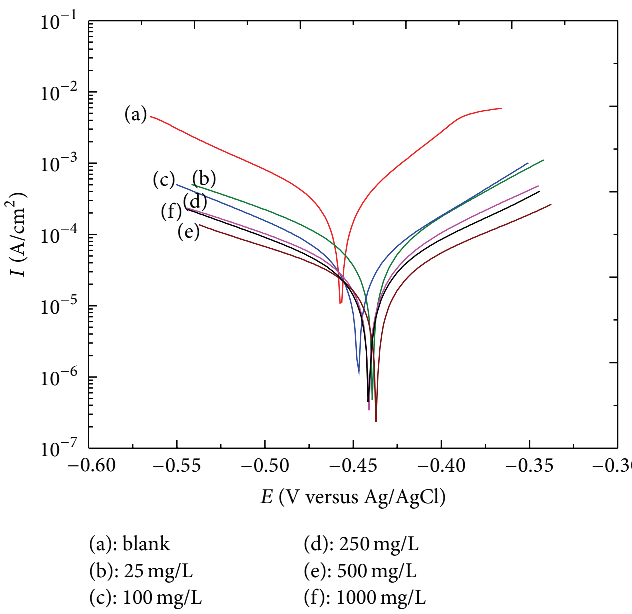

FIGURE 1: Tafel plots of mild steel in 1 M HCl in the absence and in the presence of *M. cerviana* at different concentrations.

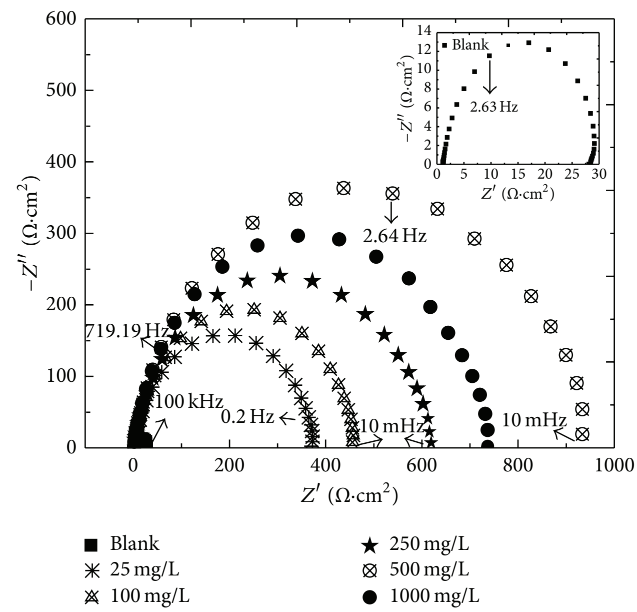

FIGURE 2: Nyquist plots for mild steel in 1 M HCl in the absence and presence of *Mollugo cerviana* at different concentrations.

potentiodynamic polarisation curves based on the following equation:

$$\%\text{IE} = \frac{i_{\text{corr},0} - i_{\text{corr}}}{i_{\text{corr}}} \times 100, \quad (2)$$

where $i_{\text{corr},0}$ is the corrosion current density in the absence of the inhibitor and i_{corr} is the corrosion current density in the presence of the inhibitor obtained from Tafel plots. The chosen plant extract showed the maximum IE of 89% at 500 mg/L. The high inhibition efficiency was due to the bonding of the adsorbed film of the inhibitor on the steel surface [31]. There is no significant change in corrosion potential with the addition of the extracts at different concentrations. The surface coverage is approximately 0.84 which is good to act as a corrosive inhibitor.

3.2. Electrochemical Impedance Spectroscopy. The Nyquist plots of mild steel in 1 M HCl solution in the absence and in the presence of *M. cerviana* extract at different concentration as inhibitor during an immersion time of 20 min at room temperature are given in Figure 2. The depression in the figure is the characteristic of the inhomogeneities of the metal surface during corrosion [32]. The diameter of the capacitive loop stands for the resistance of the corrosion and it can be seen that the resistance decreases significantly with the decrease in diameter. Considering the impedance diagrams, the size of the capacitive loop increased by increasing the concentration of inhibitor. The Nyquist impedance plots were analyzed by fitting the experimental data to a simple equivalent circuit model given in Figure 3.

In this equivalent circuit (Figure 3), R_s is the solution resistance, R_{ct} is the charge transfer resistance, and CPE is a constant phase element, which is placed in parallel to charge transfer resistance element. Thus, in these situations, pure double layer capacitors are better described by a transfer function with constant phase elements (CPE) to give a more accurate fit [33] and its impedance is given by

$$Z = A^{-1}(i\omega)^{-n}, \quad (3)$$

where A is proportionality coefficient, ω is the angular frequency, i is the imaginary number, and n is an exponent related to the phase shift and can be used as a measure of surface irregularity.

TABLE 2: The electrochemical impedance spectroscopy parameters for mild steel in 1 M HCl solution in the absence and presence of *M. cerviana* extract at different concentrations.

Concentration of the inhibitor (mg/L)	CPE (μF/cm^2)	R_s (Ω·cm^2)	R_p (Ω·cm^2)	R_{ct} (Ω·cm^2)	% IE	θ
Blank	0.09	1.06	050.11	028.67	—	—
25	0.06	1.35	289.78	374.86	92.37	0.92
100	0.04	0.78	381.87	462.20	93.81	0.94
250	0.05	1.17	533.59	621.13	95.36	0.95
500	0.05	1.08	839.67	937.93	96.95	0.97
1000	0.04	0.89	628.67	742.61	96.95	0.97

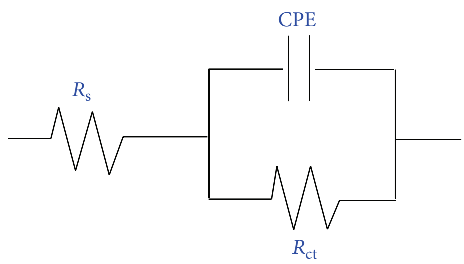

FIGURE 3: Equivalent circuit model used to fit the metal/solution interface.

TABLE 3: Mild steel weight loss data in 1 M HCl solution in the absence and in the presence of *M. cerviana* extract at different concentrations at different time at room temperature.

Concentration of the inhibitor (mg/L)	Inhibition efficiency (%)			θ
	1 h	24 h	72 h	
Blank	—	—	—	—
25	25.64	38.83	75.14	0.75
100	44.09	60.29	86.09	0.86
250	62.89	70.02	92.11	0.92
500	65.19	88.76	92.63	0.92
1000	64.5	80.25	92.30	0.92

For ideal electrodes, when $n = 1$, CPE can be considered as a real capacitor. The charge transfer resistance (R_{ct}) values are calculated based on the difference in impedance at lower and higher frequencies. According to the Helmholtz, the decrease in CPE can be attributed to increase in the thickness of the electrical double layer [34]. In EIS studies, the inhibition efficiency was calculated using (4)

$$\%\text{IE} = \frac{R_{ct} - R_{ct0}}{R_{ct}} \times 100, \quad (4)$$

where R_{ct} and R_{ct0} are charge transfer resistance in presence and absence of inhibitor obtained from the electrochemical impedance diagrams. The quantitative results of impedance measurements are given in Table 2. From the table result, it is clear that by increasing the inhibitor concentration, the CPE values tend to decrease and the R_{ct} values increase. Polarization resistance increases from 50.1 to 839.7 Ω cm^2 and double layer capacitance decreases from 0.092 to 0.046 μF/cm^2 with the increase of *M. cerviana* extract concentration. The solution resistance is approximately constant. The decrease in this capacitor values can be attributed to the formation of a protective layer on the electrode surface [35]. The increase in the polarization resistance leads to an increase in inhibition efficiency. The results also indicate that the increase of charge transfer resistance is related to a decrease in the double layer capacitance up to 500 mg/L. The effect is reversed with further increase in the concentration of the extract as seen in the result. As the concentration of the plant extract increased above 500 mg/L, the corrosion resistance decreased. In fact, the highest inhibition efficiency was reached at 500 mg/L of the plant extract which is considered to be an optimum inhibitor concentration. The surface coverage is approximately 0.95 which is found to be good to act as an anticorrosive agent. The results obtained from EIS method is in good agreement with the linear polarization and weight loss measurements.

3.3. Weight Loss Method. Weight loss data of mild steel in 1 M HCl in the absence and in the presence of various concentrations of inhibitor were obtained. This physical measurement is a direct answer to find out corrosive environments and the average corrosion rate. The variation of inhibition efficiency at different immersion times like 1, 24, and 72 hrs at room temperature in 1 M HCl is shown in Table 3. It has been observed that 500 mg/L of *M. cerviana* extract is considered to be an optimum concentration that exhibits higher efficiency against corrosion of mild steel. The results from the Table 3 also show that the corrosion rate decreases with increase in the concentration of the inhibitor in HCl and the maximum surface coverage is found to be 0.926 at 500 mg/L. There is considerable decrease in inhibition efficiency above 500 mg/L. The inefficiency above 500 mg/L may be due to weakening of metal-inhibitor interactions, resulting in the replacement of inhibitor by water or chlorine ions with decrease in inhibition efficiency [36]. The increasing inhibition efficiency and decreasing corrosion rate were attributed to the adsorption of inhibitor on the metal surface. From this result, it is clear that the extract is adsorbed at the corrosion sites of mild steel surface responsible for anticorrosion activity. The mild steel gets blocked and the adsorbed film of inhibitor acts as a physical barrier between mild steel and corrosion medium.

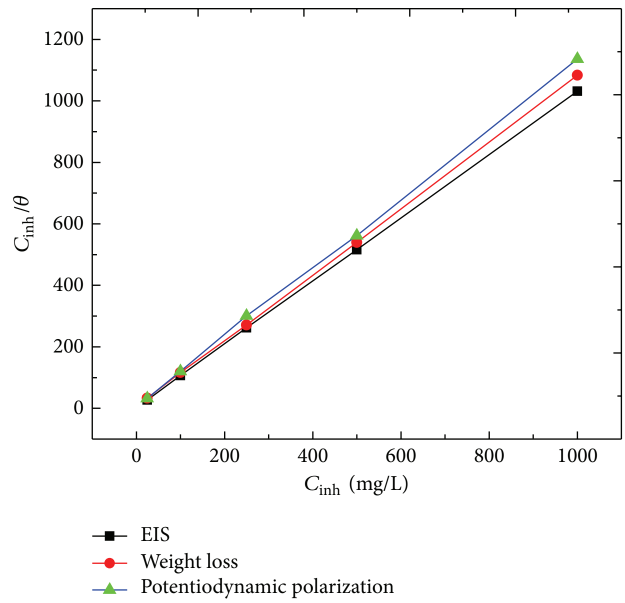

FIGURE 4: Langmuir adsorption isotherms for mild steel in 1 M HCl solution of *M. cerviana* at different concentrations by EIS, potentiodynamic polarization, and weight loss methods.

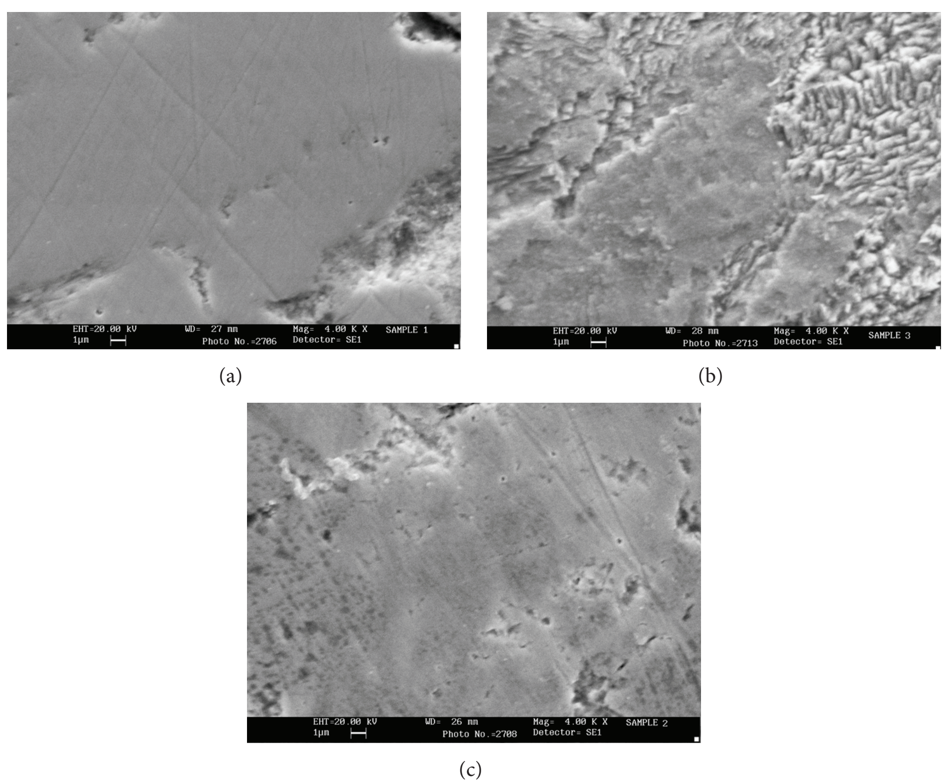

FIGURE 5: SEM images of (a) mild steel, (b) mild steel without inhibitor, (and c) mild steel with inhibitor at 500 mg/L with the magnification of 1500x.

The inhibition increased with an increase in the immersion time and stabilized after 72 hrs with the inhibition rate of 92.6%. The results obtained from the weight loss measurement are in good agreement with those obtained from the other electrochemical methods.

3.4. Adsorption Isotherm. The interaction between an inhibitor and the mild steel surface can be described by the adsorption isotherm. To obtain the adsorption isotherm, the degree of surface coverage is calculated from the equation $\theta = \%IE/100$. It was determined at different inhibitor concentration using Langmuir adsorption isotherm and it can be expressed as

$$\frac{C_{\mathrm{inh}}}{\theta} = \frac{1}{K} + C_{\mathrm{inh}}, \tag{5}$$

where K is equilibrium constant of adsorption and C_{inh} is the inhibition concentration. Many researchers have explained the Langmuir adsorption isotherm with an interaction of adsorbed species on the metallic surfaces [37, 38]. The Langmuir adsorption isotherm was drawn by plotting C_{inh}/θ versus C_{inh}, considering the θ values from weight loss method at 72 hrs, potentiodynamic polarization, and EIS measurements at room temperature for mild steel in 1 M HCl at different concentration of inhibitors, and the graph is shown in Figure 4. The straight line obtained in the graph clearly shows that the chosen inhibitor obeys the Langmuir adsorption isotherm. From this, it can be concluded that the *M. cerviana* extract can act as a good inhibitor against corrosive materials.

3.5. Surface Analysis. The surface analysis was carried out using SEM for the mild steel surface immersed in 1 M HCl solution in the absence and in the presence of 500 mg/L of inhibitor. The SEM images of the mild steel, 1 M HCl, and with the inhibitor are given in Figure 5. A significant surface damage was observed on the mild steel, which was immersed in 1 M HCl solution (Figure 5(b)) indicating that the surface is highly corroded. The surface of the corroded area of the mild steel (5B) was protected with the addition of the inhibitor as is evident from the surface morphology (Figure 5(c)) and the presence of the inhibitor at the optimum concentration exhibited a smooth surface indicating restricted corrosion unlike 5B. The SEM images clearly indicate that the mild steel surface was protected from corrosion in the presence of plant extract which is acting as a corrosion inhibitor. In other words, the *M. cerviana* extract inhibits corrosion of mild steel in 1 M HCl solution. These results are in good agreement with the other methods like electrochemical and weight loss measurements.

4. Conclusion

M. cerviana extracts acted as an effective corrosion inhibitor for mild steel in 1 M HCl solution. The polarization studies showed that *M. cerviana* extract was a mixed type inhibitor and its corrosion efficiency increased with the inhibitor concentration. From the EIS plots of mild steel, it is clear that the charge transfer resistance increased with increase in *M. cerviana* extract. Inhibition efficiency obtained from weight loss measurements was in good agreement with polarization and EIS methods. The adsorption of the *M. cerviana* extracts of mild steel in 1 M HCl solution obeys the Langmuir adsorption isotherm and the SEM studies showed the formation of inhibitor film on mild steel surface.

Conflict of Interests

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

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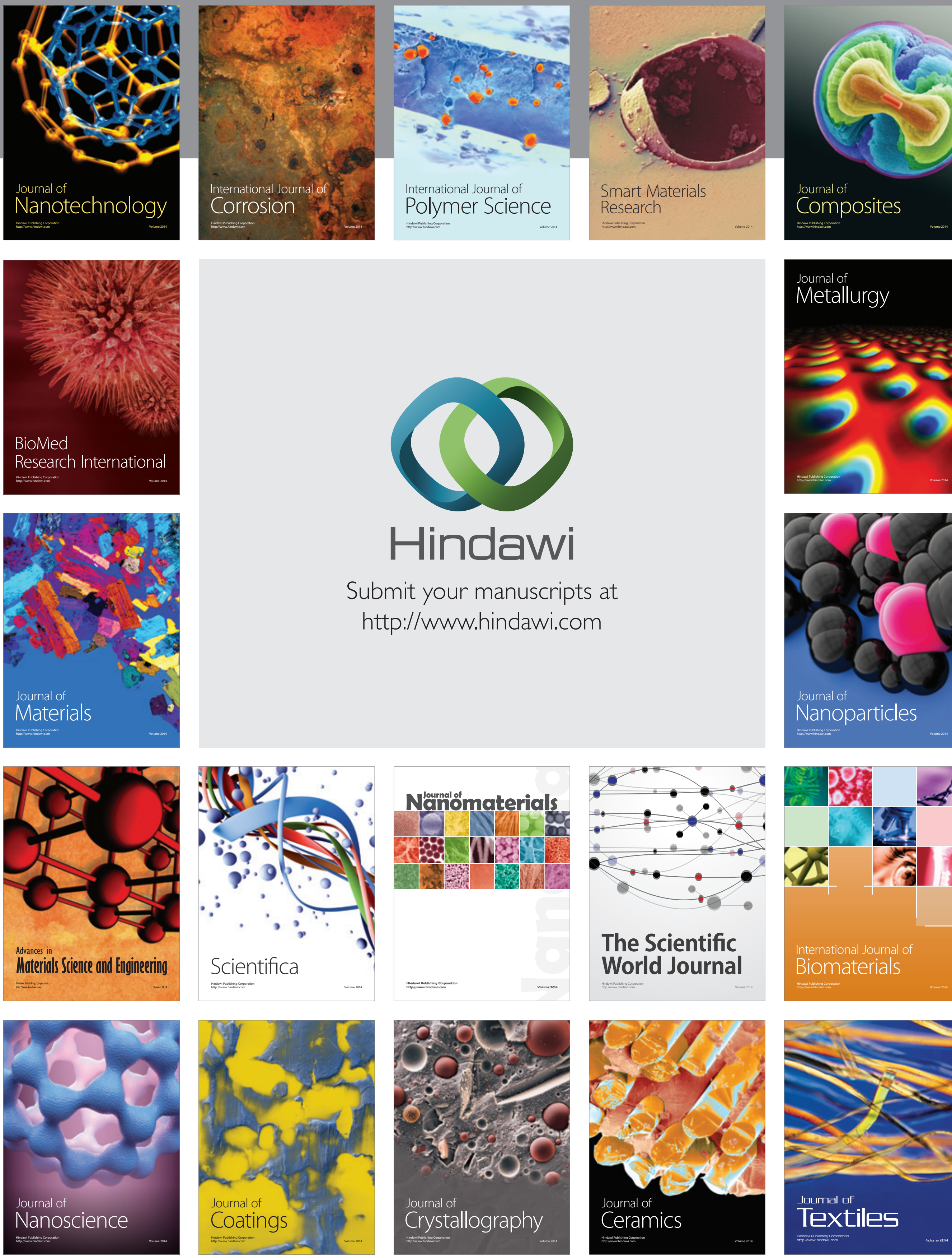
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