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Acalypha torta Leaf Extract as Green Corrosion Inhibitor for Mild Steel in Hydrochloric Acid Solution

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ABSTRACT: The inhibitive action of ethanol extract of *Acalypha torta* leaves (EAL) on corrosion of mild steel in 1 M HCl solution was investigated by weight loss, potentiodynamic polarization, electrochemical impedance spectroscopy, chronoamperometric measurements, and scanning electron microscopic observations. The adsorption of EAL on mild steel follows a Langmuir adsorption isotherm, and the activation parameters governing the adsorption process were calculated and discussed. Polarization measurements reveal that the EAL acts as a mixed-type inhibitor. The inhibition efficiencies obtained from weight loss measurements and electrochemical tests were in good agreement.

INTRODUCTION

Mild steel is one of the important alloys of iron which has numerous industrial and engineering applications because of its excellent mechanical properties. Its important applications are in acid pickling, industrial cleaning, acid descaling, oil well acidizing, and petrochemical processes.[1] Even though mild steel has wide applications, it is very much susceptible to corrosion due to its thermodynamic instability especially in acid medium and hence the study of mild steel corrosion phenomena has become important particularly in acidic media. Among the acids, hydrochloric acid (HCl) is one of the most widely used agents in several industries for the removal of undesirable oxide films and corrosion products of the surface of steel. The most common, effective, and economic methods to prevent metal dissolution in an acidic environment is the use of corrosion inhibitors.

The heterocyclic organic compounds containing O, S, and N atoms have good inhibition properties. However, they are toxic, expensive, and not environmentally safe. Hence, investigations are focused toward the development of naturally occurring substances as ecofriendly corrosion inhibitors. Extracts of some plants have been reported as inhibitors for different metals in acid solution.[2−8]

In 1 M HCl solution, the plant extracts of *Henna*,[9] *Oxandra asbecki*,[10] *Argemone mexicana*,[11] *Isertia cocccinea*[12] and *Palicourea guianensis*[13] have been tested as corrosion inhibitors for steel. These plant extracts are low cost, nontoxic, readily available, and ecofriendly substances. Therefore finding naturally occurring substances as corrosion inhibitors is a subject of great practical significance.[9−14]

The literature study reveals that medicinal plant extracts were generally rich in flavanoids, alkaloids, polyphenols, and other natural organic compounds which possess heteroatoms like N, O, and S in their molecular structure. *Acalypha torta* belongs to the family Euphorbiaceae, possesses diverse medicinal properties, and is used for the treatment of malaria, rheumatism, upset stomach, dermatitis, and infantile eczema.[15−17] These medicinal properties are due to the presence of natural organic compounds containing heteroatoms. It is well-known that the acid corrosion of metals can be inhibited by compounds containing heteroatoms. As a result in the present work, the ethanol extract of *Acalypha torta* leaves (EAL) has been selected to examine its potential to control corrosion of mild steel in 1 M HCl medium. In this study the corrosion inhibitive effect of EAL has been confirmed by weight loss, polarization, electrochemical impedance spectroscopy (EIS), chronoamperometry, and scanning electron microscopic (SEM) techniques.

2. METHODS AND MATERIALS

2.1. Preparation of Plant Extract. Leaves of *Acalypha torta* were washed thoroughly and dried. A 400 g portion of dried and pulverized leaves of *Acalypha torta* were soaked in 2 L of chloroform−methanol (2:1) at room temperature for 72 h. The solvent was changed three times in 72 h at 24 h intervals. The extract was filtered through cheesecloth and Whatmann no.1 filter paper. The residue was then dried and re-extracted three times in 2 L of ethanol at room temperature and at 24h intervals. After filtration, the filtrate was evaporated and finally thick brown slurry was obtained and refrigerated.[17] The corrosion test solutions were prepared by dissolving specified amount of EAL in 1 M HCl solution and used for corrosion study.

2.2. Preparation of Metal Samples. The mild steel specimens having composition 0.04% C, 0.35% Mn, 0.022% P, 0.036% S, and remainder Fe were used for weight loss as well as electrochemical studies. The mild steel coupons of dimension 4 cm × 2 cm × 0.1 cm were used for weight loss measurements and of 1 cm ×1 cm (exposed area) with a 5 cm long stem (isolated with Araldite resin) were used for electrochemical experiments. Prior to each experiment, the mild steel samples were mechanically abraded with different grades of emery

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Table 1. Effect of Temperature on the Corrosion Rate of Mild Steel in 1 M HCl at Different Concentrations of EAL

	temperature							
	303 K		313 K		323 K		333 K	
C (ppm)	υ_{corr} (mg cm^{-2} h^{-1})	η_w (%)	υ_{corr} (mg cm^{-2} h^{-1})	η_w (%)	υ_{corr} (mg cm^{-2} h^{-1})	η_w (%)	υ_{corr} (mg cm^{-2} h^{-1})	η_w (%)
blank	4.3		12.16		19.70		31.27	
250	0.99	77	5.47	55	9.46	52	18.76	40
500	0.73	83	4.26	65	7.49	62	15.32	51
750	0.60	86	3.04	75	5.52	72	12.20	61
1000	0.47	89	1.82	85	3.74	81	8.76	72

papers (grade nos. 220, 660, and 1200), washed in double distilled water and acetone, dried in room temperature, and stored in moisture free desiccator.

2.3. Weight Loss Measurements. The weight loss of precleaned and dried mild steel specimens were determined by weighing the metal samples before and after immersing in 100 cm^3 of 1 M HCl in the absence and presence of various concentration of EAL at 303, 313, 323 and 333 K. The experiments were performed in triplicate and the mean value is reported. All experiments were carried out in aerated and static conditions. The corrosion rate (υ_{corr}) of mild steel was determined using the relation

$$\upsilon_{corr} = \frac{\Delta m}{St} \tag{1}$$

where υ_{corr} is the corrosion rate of mild steel (g cm^{-2} h^{-1}), Δm is the corrosion weight loss of mild steel (g), S is the surface area of mild steel specimen (cm^2), and t is the time of exposure.

The percentage inhibition efficiency η_w (%) was calculated using the relationship

$$\eta_w(\%) = \frac{\nu^{\circ}_{corr} - \nu_{corr}}{\nu^{\circ}_{corr}} \times 100 \tag{2}$$

where υ°_{corr} and υ_{corr} are the corrosion rates of mild steel in the absence and presence of EAL, respectively.

2.4. Electrochemical Measurements. The electrochemical measurements were conducted in a conventional glass cell using CHI 660C electrochemical analyzer (CH instruments, 3700 Tennison Hill Drive, Austin, TX 78738). A mild steel specimen (of 1 cm^2 area), a platinum electrode, and a saturated calomel electrode were used as working, auxiliary, and reference electrodes. In the case of polarization and electrochemical impedance spectroscopic measurements (EIS) prior to each measurement, a stabilization period of 30 min was allowed to establish a steady state open circuit potential (OCP). Each experiment was carried out in triplicate, and the average values of corrosion parameters are reported.

The potentiodynamic polarization measurements were carried out over a potential automatically from +200 to −200 mV at OCP with a scan rate of 0.5 mV s^{-1}. All the potentials reported were with reference to SCE. The corrosion kinetic parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), and anodic (β_a)/cathodic (β_c) Tafel slopes were generated from the software installed in the instrument. The percentage inhibition efficiency η_T (%) was computed from I_{corr} values using the relationship

$$\eta_T(\%) = \frac{I^{\circ}_{corr} - I_{corr}}{I^{\circ}_{corr}} \times 100 \tag{3}$$

where I°_{corr} and I_{corr} are the corrosion current densities without and with inhibitor, respectively.

The EIS measurements were carried at OCP in the frequency range 1 mHz to 100 kHz with 5 mV sine wave as the excitation signal. Impedance data were analyzed using ZSimp-Win 3.21 software. The inhibition efficiency η_Z (%) was evaluated from charge transfer resistance (R_{ct}) values using the following equation:

$$\eta_Z(\%) = \frac{R_{ct} - R^{\circ}_{ct}}{R_{ct}} \times 100 \tag{4}$$

where R°_{ct} and R_{ct} are the charge transfer resistances without and with EAL, respectively.

The chronoamperometric experiments were performed by polarizing the working electrode anodically at −0.41 V (SCE) for 600 s.

The surface morphology of the mild steel samples after immersion in 1 M HCl in the absence and presence of 1000 ppm EAL was analyzed using scanning electron microscopy (JEOL, JSM 6400, JEOL Datum Shanghai Co. Ltd., Building A, No. 51, Rijing Road, Waigaoqiao F.T.Z., Shanghai, 200120)

3. RESULTS AND DISCUSSION

3.1. Weight Loss Measurements. Weight loss of mild steel surface in 1 M HCl was determined at 303, 313, 323, and 333 K in the absence and presence of different concentration of EAL. The obtained corrosion parameters are depicted in Table 1.

It is clear from the table that the percentage inhibition efficiency increases with concentration (C) of the extract and decreases with temperature. The decrease in corrosion rate (υ_{corr}) with increase in concentration of EAL is due to the fact that the surface coverage of metal increases by the adsorption of inhibitor molecules.[18] The increase in corrosion rate with increase in temperature may be probably due to increased rate of desorption of inhibitor molecules of EAL from the mild steel surface at higher temperature.[19]

3.1.1. Effect of Temperature. Temperature has more pronounced effect on the rate of electrochemical corrosion of metal. In acid medium, υ_{corr} increases exponentially with increase in temperature. Also, the decrease of hydrogen overpotential observed at higher temperature.[20] Usually corrosion reactions are regarded as Arrhenius processes and the υ_{corr} can be expressed by the relation[9]

$$\ln \nu_{corr} = \ln - A\frac{E_a}{RT} \tag{5}$$

Where υ_{corr} is the corrosion rate, E_a is the apparent activation energy of the corrosion process, R is the universal gas constant (8.314 J K^{-1} mol^{-1}), T is the absolute temperature, and A is the Arrhenius pre-exponential factor. Arrhenius plots of ln υ_{corr} versus $1/T$ for mild steel corrosion in 1 M HCl in the absence and presence of different concentration of EAL are presented in Figure 1, and the calculated parameters are given in Table 2.

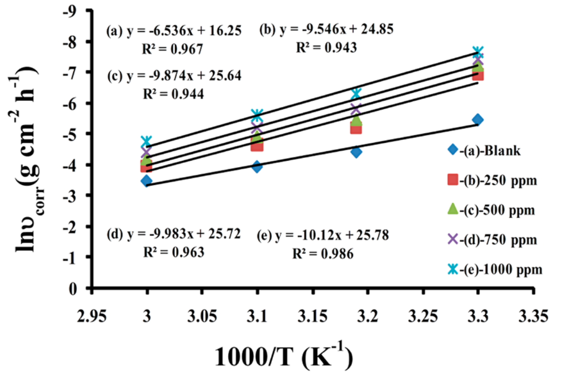

Figure 1. Arrhenius plot for mild steel in 1 M HCl in the absence and presence of different concentrations of EAL.

Table 2. Activation Parameters of Mild Steel in 1 M HCl in the Absence and Presence of Different Concentrations of EAL

C_{inh} (ppm)	A (g cm^{-2} h^{-1})	E_a (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J mol^{-1} K^{-1})
blank	11.41×10^6	54.34	51.70	−118.57
250	6.198×10^{10}	79.37	76.72	−47.14
500	1.366×10^{11}	82.09	79.46	−40.57
750	1.479×10^{11}	82.99	80.29	−40.16
1000	1.571×10^{11}	84.14	81.42	−39.66

In 1 M HCl solution, the activation energy increases to a greater extent in the presence of EAL compared to uninhibited solution. The increase in the E_a may be interpreted as physical adsorption.[21,22] It can also be concluded that the whole process is controlled by surface reaction since the energy of activation of the corrosion process in the absence and presence of EAL are greater than 20 kJ mol^{-1}.[23,24] Further both the E_a and frequency factor values for inhibited solutions increase with inhibitor concentration which indicates that the addition of EAL decrease the metal dissolution in 1 M HCl by increasing the energy barrier for the corrosion reaction by the adsorption of active organic molecules at the metal surface.[9,25,26]

The change in enthalpy (ΔH^*) and entropy (ΔS^*) of activation were calculated by the transition-state equation given below.[9]

$$\ln \frac{\upsilon_{corr}}{T} = \left[\ln \frac{R}{Nh} + \frac{\Delta S^*}{R}\right] - \frac{\Delta H^*}{RT} \qquad (6)$$

where N is the Avogadro's number and h is the Planck's constant. The plots of $\ln \upsilon_{corr}/T$ versus $1/T$ are depicted in Figure 2. Straight lines were obtained with a slope of $-\Delta H^*$ and ΔS^* values were calculated from the intercepts of $\ln \upsilon_{corr}/T$ axis and are given in Table 2.

It is evident from the table that the activation parameters (ΔH^* and ΔS^*) of mild steel dissolution reaction in 1 M HCl in the presence of EAL are higher than in the absence of extract. The positive sign of enthalpies reflects the endothermic nature of mild steel dissolution process.[27] Whereas the large and negative values of entropy of activation in the absence and presence of EAL implies that the rate-determining step for the activated complex is an association step rather than a dissociation step, meaning that during adsorption process the decrease in disordering takes place on moving from reactants to the activated complex.[28] Thus orderliness increases as reactants are converted to activated complex.

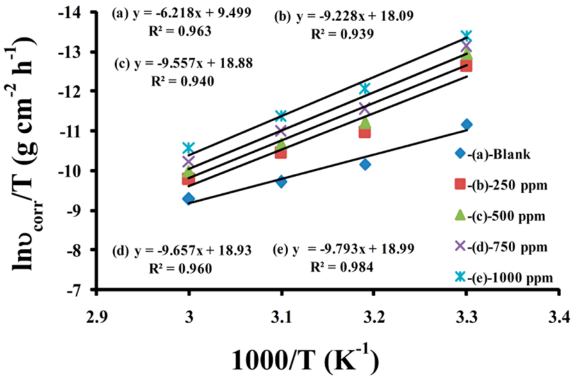

Figure 2. Transition state plot for mild steel in 1 M HCl in the absence and presence of different concentrations of EAL.

3.1.2. Adsorption Isotherm. Adsorption isotherms are very important in determining the mechanism of organo-electrochemical reaction.[29] Hence in order to gain more information about the mode of adsorption of EAL on mild steel surface in 1 M HCl at 303, 313, 323, and 333 K, attempts were made to fit experimental data with several adsorption isotherms like Langmuir, Temkin, Freundlich, Bockris−Swinkles, and Flory−Huggins isotherms. However the best fit was obtained with Langmuir isotherm which is in good agreement with the equation:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (7)$$

where C is the inhibitor concentration, θ is the degree of surface coverage defined as $\eta_w(\%)/100$ at different concentration of inhibitor evaluated from weight loss measurement, and K_{ads} is the equilibrium constant of adsorption process. The plots of C/θ versus C were straight lines with almost unit slopes and are given in Figure 3. It is found that all the regression coefficients are very close to one which indicates that adsorption of active organic molecules of EAL on the mild steel surface obeys Langmuir adsorption isotherm.

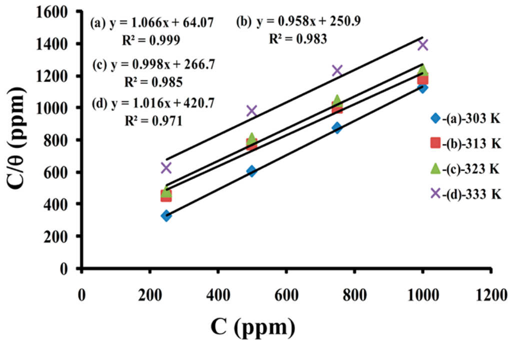

Figure 3. Langmuir adsorption plots of mild steel in 1 M HCl solution containing different concentrations of EAL at different temperature.

The Langmuir adsorption isotherm is based on the assumption that all adsorption sites are equivalent and the binding of particles occurs independently from nearby sites being occupied or not.[30] This indicates that the adsorbing species of EAL occupies typical adsorption site at the metal/solution interface. Moreover it is not possible to discuss the adsorption behavior of plant extracts as inhibitors in terms of thermodynamic parameters, such as the standard free energy of adsorption value because the molecular mass of the extract components is not known. The same limitation has been noted by some authors.[13,31,32]

3.2. Electrochemical Measurements. *3.2.1. Potentiodynamic Polarization Measurements.* In order to know the kinetics of anodic and cathodic reactions, polarization experiments were carried out potentiodynamically in unstirred 1 M HCl solution in the absence and presence of different concentration of EAL and the obtained polarization curves are shown in Figure 4. The corrosion kinetic parameters derived from these curves are presented in Table 3.

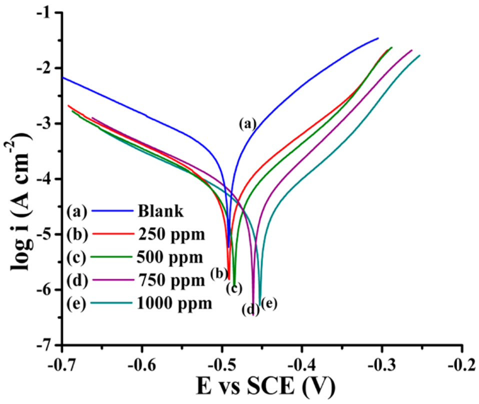

Figure 4. Polarization curves for mild steel in 1 M HCl in the absence and presence of different concentrations of EAL.

Table 3. Polarization Parameters for Mild Steel in 1 M HCl in the Absence and Presence of Different Concentrations of EAL

C (ppm)	$-E_{corr}$ (mV)	β_c (mV dec^{-1})	β_a (mV dec^{-1})	I_{corr} (μA cm^{-2})	η_T (%)
blank	492	146.39	86.11	362.9	
250	491	142.67	91.34	77.89	79
500	485	147.7	82.62	54.79	85
750	461	143.8	70.58	44.11	87
1000	453	146.2	74.43	29.19	91

The close examination of Figure 4 reveals that the addition of EAL to 1 M HCl affects both the anodic and cathodic parts of the curve. This indicates that the addition of extract to acid solution reduces the anodic dissolution of metal and also impedes the cathodic hydrogen evolution reaction.[12] It can be seen from the Table 3 that the corrosion current density I_{corr} decreased noticeably with increase in EAL concentration and also the corrosion potential E_{corr} of mild steel shifts toward less negative direction which suggests that EAL behaves as a very good corrosion inhibitor for mild steel in 1 M HCl solution. An inhibitor can be classified as an anodic—or cathodic—type when the change in E_{corr} value is larger than 85 mV.[33] But in the present study, the largest displacement exhibited by the extract was 39 mV, from which it can be concluded that EAL acts as a mixed type inhibitor. On the other hand, the anodic and cathodic slope values of inhibited solution have changed with respect to uninhibited solution which also reiterates that the extract is mixed type inhibitor. The obtained protection efficiency values are in good correlation with the values of weight loss measurements.

3.2.2. Electrochemical Impedance Spectroscopic Measurements. Electrochemical impedance spectroscopy (EIS) has been widely used in investigating corrosion inhibition processes since it provides more information on both the resistive and capacitive behavior at metal/solution interface. The corrosion behavior of mild steel in 1 M HCl solution in the absence and presence of the extract was investigated using this technique. The impedance data are presented as Nyquist and Bode plots in Figures 5 and 6, respectively.

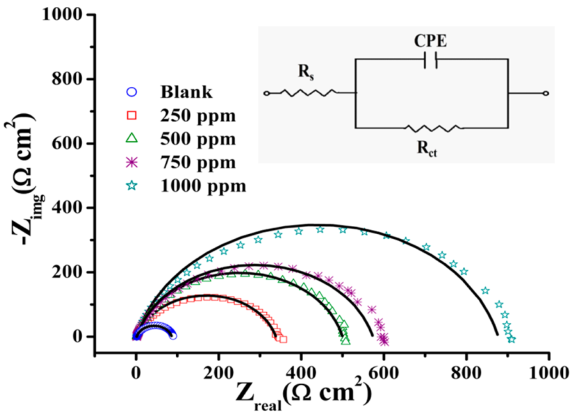

Figure 5. Nyquist plots (solid line shows fitted results) for mild steel in 1 M HCl in the absence and presence of different concentrations of EAL.

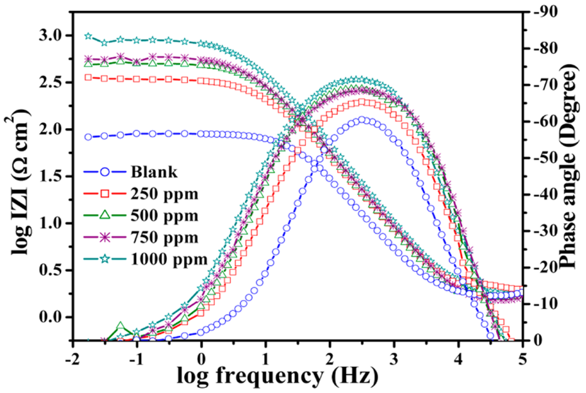

Figure 6. Bode plots for mild steel in 1 M HCl in the absence and presence of different concentrations of EAL.

The electrochemical equivalent circuit employed to analyze impedance spectra is shown in the inset of Figure 5.[34] In the equivalent circuit, R_s represents the solution resistance, R_{ct} represents the charge transfer resistance, and CPE represents the constant phase element describing the interface double layer. The impedance (Z) of CPE can be represented as follows:[35]

$$Z_{CPE} = Q^{-1}(j\omega)^{-n} \tag{8}$$

where Q is the CPE constant, ω is the angular frequency, $j^2 = -1$ is the imaginary number, and n is the CPE exponent which gives details about the degree of surface inhomogeneity resulting from surface roughness, inhibitor adsorption, porous layer formation, etc.[36] The impedance parameters deduced from the analysis of impedance spectra (Figure 5) are tabulated in Table 4.

Table 4. Electrochemical Impedance Parameters for the Corrosion of Mild Steel in 1 M HCl Containing Different Concentrations of EAL

C_{inh} (ppm)	R_{ct} (Ω cm^2)	Q ($\mu\Omega^{-1}$ S^n cm^{-2})	n	C_{dl} (μF cm^{-2})	η_z (%)
blank	84.7	115.2	0.864	55.59	
250	336.6	92.3	0.829	45.03	74.8
500	498.2	73.2	0.856	41.94	82.9
750	571.0	75.5	0.842	41.85	85.1
1000	874.7	58.8	0.855	35.55	90.3

The double layer capacitance (C_{dl}) can be calculated by the following relation[19]

$$C_{dl} = (QR_{ct}^{\,1-n})^{1/n} \tag{9}$$

The calculated C_{dl} values are listed in Table 4. It can be concluded from the Figure 5 that a single semicircle has been observed at high frequency. This can be attributed to charge transfer of the corrosion process, and also, the diameter of the semicircle increases in increasing EAL concentration. It is apparent from the Table 4 that the presence of EAL in acid media leads to decrease in C_{dl} and Q values. The decrease in Q values can be attributed to the decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer.[37] Meanwhile the increase in R_{ct} values indicates that the extent of adsorption increase with increase in extract concentration and also the adsorbed extract forms a protective film on the mild steel surface which becomes a barrier to hinder the mass and charge transfer processes. This results in an increase in the protection efficiency. On the other hand, one time constant was observed in the Bode diagram (Figure 6). The maximum phase angle for corroding system represented by a simple RC parallel combination should be 90° when $R_s = 0$. However, depressed semicircles are usually obtained for an electrode/solution interface, which has been known to be associated with a rough electrode surface.[38] The corrosion of mild steel in acid media increases the roughness of the electrode surface and therefore reduces the phase angle. But in Figure 6, the phase angle increase with increase in extract concentration which indicates the decrease in surface inhomogeneity. Due to all these factors as the concentration of EAL increases, the protection efficiency also increases.

3.2.3. Chronoamperometric Measurements. In order to verify the ability of EAL to inhibit anodic processes of mild steel, the chronoamperometric experiments were carried out by polarizing anodically the electrode potential at −0.41 V (vs SCE) for 600 s. the current density values obtained during the electrooxidation of mild steel were recorded in 1 M HCl in the absence and presence of 1000 ppm EAL. The chronoamperometric curves are depicted in Figure 7.

By considering the current density values in the absence and presence of 1000 ppm EAL, the protection efficiency was calculated around 90%.[39] Hence, it can be concluded that the extract act as an efficient corrosion inhibitor for mild steel in 1 M HCl.

3.3. Surface Morphological Studies. The SEM images (Figure 8) of mild steel immersed in 1 M HCl solution in the absence and presence of 1000 ppm EAL were recorded to establish the interaction of extract with the metal surface. It could be observed from the Figure 8A that the specimen surface was strongly damaged in the absence of the extract and the surface is rough and highly porous. Whereas in the presence of EAL (Figure 8B), the specimen is in better condition having smooth surface compared with that of the surface immersed in acid medium alone which is attributed to the formation of a protective layer by the constituents of EAL. This indicates that EAL hinders the dissolution of iron and there by reduces the rate of corrosion of mild steel in 1 M HCl solution.

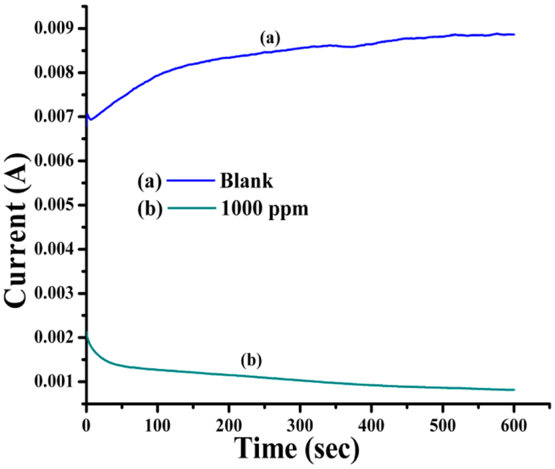

Figure 7. Chronoamperometric curves for mild steel in 1 M HCl in the absence (a) and in the presence (b) of 1000 ppm of EAL.

3.4. Mechanism of Inhibition. The weight loss and electrochemical experiments reveals that the corrosion of mild steel is retarded in the presence of different concentration of EAL. The inhibitor molecules present in the extract blocks the surface of mild steel via adsorption mechanism. The *Acalypha* species contain high concentrations of polyphenols, terpenoids, and plant sterols, and these organic compounds contains fused benzene rings and O heteroatoms in the rings.[40] The values of thermodynamic parameters indicated that the adsorption of EAL on mild steel surface involves a physisorption. The macromolecules present in the extract get electrostatically adsorbed on the mild steel surface by donor−acceptor interactions between the π electrons of fused benzene rings and the vacant d-orbital of iron atoms.

Generally corrosion takes place through two reactions:

(1) Anodic reaction which leads to metal dissolution.

$$M \rightarrow M^{n+} + ne^- \tag{10}$$

(2) Cathodic reaction which results in hydrogen evolution in acid media

$$2H^+ + 2e^- \rightarrow H_2 \tag{11}$$

According to the polarization results, EAL follows the mixed inhibition mechanism. Some of the organic molecules present in EAL may get protonated, and these cationic forms may adsorb directly on the cathodic sites of the mild steel and reduces the hydrogen evolution reaction. On the other hand, EAL may adsorb on anodic sites of mild steel through the π electrons of the aromatic rings and lone pair of electrons of heteroatoms and thereby inhibit the anodic dissolution of mild steel. Hence by following the above mechanism, EAL shows mixed inhibition behavior on mild steel surface in 1 M HCl solution.

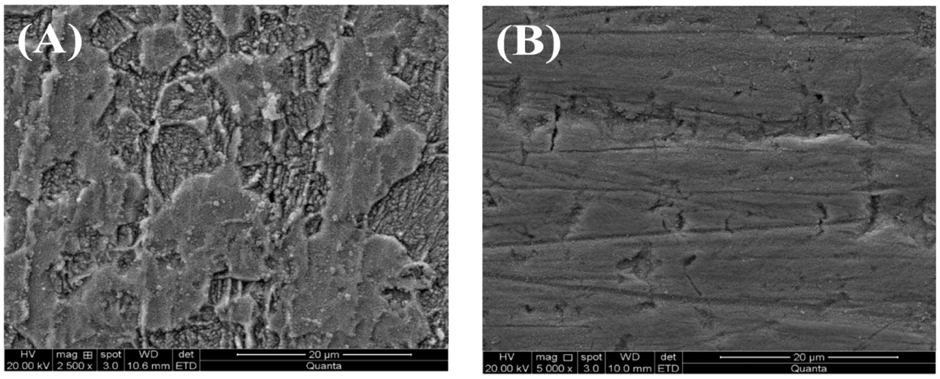

Figure 8. SEM images of mild steel exposed to 1 M HCl solution in the absence (A) and presence (B) of 1000 ppm EAL.

4. CONCLUSION

The ethanol extract of *Acalypha torta* leaves acts as a mixed inhibitor for mild steel in 1 M HCl. The protection efficiency of EAL increases with concentration and decreases with temperature. The adsorption of the extract follows Langmuir adsorption isotherm. The presence of EAL decreases the anodic current as observed in the CA experiments and the percentage inhibition efficiency values obtained from weight loss measurements are comparable with those obtained from electrochemical measurements.

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Notes

The authors declare no competing financial interest.

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