Inhibition of Acid Corrosion of Mild Steel Using *Delonix regia* Leaves Extract

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Abstract. The inhibition efficacy of aqueous extracts of the leaves of *Delonix regia* (DR) in 1 M HCl and 0.5 M H₂SO₄ have been investigated using weight loss, electrochemical and surface probe techniques. DR extract inhibited mild steel corrosion in both acidic environments via adsorption of the extract organic matter on the metal/solution interface. Potentiodynamic polarization results indicate that DR is a mixed type inhibitor in both acidic environments, whereas the impedance results revealed adsorption of the DR species on a corroding steel surface. Increase in inhibition efficiency was concentration dependent. The adsorption followed Langmuir adsorption isotherm. Scanning electron microscopy (SEM) results revealed the formation of a protective layer adsorbed on a mild steel surface in the acid solutions.

Introduction

Introduction of corrosion inhibitors into acid solutions commonly employed in cleaning iron and steel surfaces is a useful method to protect such surfaces from corrosion damage [1, 2]. As regards to the adsorption strength of these inhibitors, certain factors are of significant consideration, including: the nature and surface charge of the metal, composition of the electrolyte and the structure of the inhibitor [3, 4]. Most effective inhibitors are organic compounds that contain heteroatoms like nitrogen, sulfur and oxygen in a conjugated system [5-10]. The inhibitors function at the interface between the metal and aqueous corrosive solution, their interaction with the metal surface through adsorption, results in a modification in the mechanism of the electrochemical process. Polar functional groups are regarded as the reaction center that stabilizes the adsorption process [11], this helps to reduce the corrosion susceptibility of the metal surface [9, 12]. As a result, the service life of the metal is prolonged. The requisite electronic structural characteristics of corrosion inhibitors such as substituted heterocycles like imidazoles, pyridines, furans, thiophenes, isoxazoles, and others are readily found in plant extracts [13].

Considering the increasing ecological awareness and strict environmental regulations, attention is now focusing on the development of substitute environmentally friendly alternatives to hazardous chemical processes. Such development will definitely depend on the only practical sustainable source of materials – plant(biomass) extracts - and encourage the use of these extracts as sources of alternative benign chemical substances.

Considering this fact, we have over time studied the possibility of applying biomass extracts directly in solving materials corrosion problems and as potential replacements for the costly and toxic chemicals in use presently. The justification here is that some phytochemical constituents of plant extracts including tannins, proteins, polysaccharides, polycarboxylic acids, alkaloids, etc., possess electronic structures similar to those of conventional organic corrosion inhibitors and some have been reported to function as inhibitors of metal corrosion [12-20]. Most organic inhibitors are known to function via adsorption, involving both specific and non specific interactions with the metal surface. Therefore, all the components are involved in the inhibition process.

The leaf extract of *Delonixregia* (DR) has been studied in this work for its inhibiting efficacy on mild steel corrosion in aqueous acidic environments. *Delonixregia* possesses a lot of medicinal qualities and the decoction of the leaves is traditionally used in treating gastric problems, body pain, and rheumatic pains of joints. Ethanolic extracts of the flower and bark were investigated to anti-inflammatory activity. The leaves are reported to possess antibacterial and antimalarial properties. *Delonix regia* contains carbohydrates, alkaloids, flavanoids, cardiac glycosides, anthraquinone glycosides, saponins, steroids and tannins [21 – 25].

Experimental

Materials preparation

Mild steel specimens with chemical composition (weight %) C -0.30, Si – 0.30, Mn – 0.30, P – 0.045, S – 0.050, Cr – 0.064, Cu – 0.040, Ti – 0.04 and the balance Fe were used for the experiments. The metal specimens were wet-polished with silicon carbide abrasive paper (from grade #150 – #1000), degreased in acetone and dried in warm air. All chemicals and reagents used in preparation of the test solutions were of analytical grade (Sinopharm Chemical Reagent Co., Ltd). The aggressive solutions were prepared using HCl and H₂SO₄ (Sinopharm Chemical Reagent Co., Ltd) and double distilled water. Stock solutions of the crude extract were prepared by refluxing weighed (40g) amount of the dried and ground leaves of DR in absolute ethanol (500 ml) for three hours. This was used without further purification. The amount of material extracted into solution was quantified by comparing the weight of the dried residue with the initial weight of the dried plant material before extraction. Inhibitor test solutions were prepared in the concentration range 50-1200 mg/L from the stock solution.

Gravimetric experiments

Gravimetric experiments were conducted on test coupons of dimension 3 x 3 x 0.14 cm, these were abraded using Silicon carbide paper (up to 1000 grit), rinsed with distilled water, dried in acetone and warm air, weighed and stored in moisture free desiccators prior to use, these were suspended using glass rods and hooks under total immersion conditions in 300ml beaker containing the test solutions at room temperature. All tests were made in aerated and unstirred test solutions. In order to ascertain the weight loss with respect to time, test coupons were retrieved at 24 h intervals progressively for 168 hrs; immersed in 20% NaOH solution containing 200g/L of zinc dust, scrubbed with bristle brush, washed, dried and reweighed. The weight loss was taken as the difference between the initial and final weights of the coupons. Measurements were undertaken using a FAJA weighing balance of range 0.0001 to 200g.

Electrochemical measurements

Electrochemical tests were conducted using a PAR-2273 Advanced Electrochemical System workstation, with a conventional three-electrode corrosion cell. A platinum sheet and a saturated calomel electrode (SCE) were used as a counter and reference electrodes, respectively. A metal specimen fixed in epoxy resin with a surface area of 1 cm^2 exposed to the test solution, served as the working electrode. Electrochemical measurements were carried out in aerated and unstirred solutions at the end of 1800 s of immersion, which allowed the OCP values to attain steady state. Temperature was fixed at $30 \pm 1^\circ \text{C}$. Impedance measurements were performed at corrosion potentials (E_{corr}) over a frequency range of 100 kHz - 0.1 Hz, with a signal amplitude perturbation of 5 mV. Potentiodynamic polarization studies were conducted in the potential range $\pm 250 \text{ mV}$ versus corrosion potential at a scan rate of 0.333 mV/s [28].

Scanning Electron Microscopy

The XL-30FEG scanning electron microscope was used to study the surface morphology of the metal specimens after immersion in 1 M HCl and 0.5 M H_2SO_4 in the absence and presence of DR. Mild steel specimens of dimensions $3 \text{ cm} \times 3 \text{ cm} \times 0.25 \text{ cm}$ were prepared as mentioned earlier (Section 2.1) and immersed for 24 h in both acidic solutions, in the absence and presence of 1200 mg/L DR. The specimens were then removed and cleaned with double distilled water, dried in warm air and submitted for SEM analysis.

Results and discussion

Analysis of the results of the weight loss measurements

Figures 1 and 2, shows the results of weight loss measurements against time for the corrosion of mild steel in 0.5 M H_2SO_4 and 1 M HCl solutions in the absence and presence of different concentrations of aqueous extract of DR for different immersion times (1-7 days) at 303K. This study was performed to evaluate the stability of the extract over time. Close scrutiny of the plot revealed that the rate of mild steel corrosion was significantly reduced in the presence of DR extracts for all immersion times, indicating retardation of the metal dissolution process. The data presented here are means of a triplicate determinations with standard deviations < 0.001 . This behavior reflects the inhibitory effect of this extract against the mild steel corrosion in the studied environments.

Fig. 1 Variation of weight loss with time for mild steel corrosion in 0.5 M H_2SO_4 solution in the absence and presence of DR at 30°C .

Fig. 2 Variation of weight loss with time for mild steel corrosion in 1 M HCl solution in the absence and presence of DR at 30°C.

Careful examination of the plots in Figures 1 – 4, reveals fact that the metal specimen manifests higher corrosion susceptibility in 0.5 M H_2SO_4 . The observed phenomenon shows that the acid anions influence the corrosion process in different ways. The lower weight loss values observed in 1 M HCl environment is attributable to the ability of chloride ions to replace hydroxyl ions adsorbed on the metal surface, thus reducing the catalytic effect. The plots (Figures 3 and 4) clearly reveal a general reduction in weight loss in both 1 M HCl and 0.5 M H_2SO_4 due to DR addition, suggesting a corrosion inhibiting effect of the extract. The inhibiting effect becomes more pronounced with increasing DR concentration, indicating a dependence of the inhibition process on the amount of inhibiting species present in the system.

Fig. 3 Variation of weight loss with concentration for mild steel corrosion in 0.5 M H_2SO_4 solution in the absence and presence of DR at 30°C.

Fig. 4 Variation of weight loss with concentration for mild steel corrosion in 1 M HCl solution in the absence and presence of DR 30°C

Inhibition Efficiency

The inhibitive effect of DR in retarding the dissolution of mild steel in 0.5 M H₂SO₄ and 1 M HCl environment was determined by comparing the weight loss of the metal in the blank acid solution and in the presence of DR. Figures 5 and 6 show the relationship between the inhibition efficiency and different concentrations of DR in the acidic solutions. Close observation of the plot reveals that efficiency increases with an increase in concentration.

The inhibition efficiency (IE %) of DR extract on mild steel corrosion was calculated from the weight loss data (g) according to the equation:

$$IE(\%) = \left(1 - \frac{W_1}{W_2}\right) \times 100 \quad (\text{Eq. 1})$$

where W_1 and W_2 are the weight losses in inhibited and uninhibited corrodents, respectively. The high IE% values obtained indicate a strong adsorption of the constituents of DR species on a corroding metal surface.

Fig. 5 Variation of Inhibition Efficiency (IE) with concentration for mild steel corrosion in 0.5 M H₂SO₄ solution in the presence of DR 30°C.

Fig. 6 Variation of Inhibition Efficiency (IE) with concentration for mild steel corrosion in 1 M HCl solution in the presence of DR 30°C.

Electrochemical Measurements

It has been established the corrosion reaction is an electrochemical process, for this reason electrochemical measurements are the most appropriate for understanding in-depth mechanistic insights into corrosion systems.

Potentiodynamic polarization measurements

Polarization experiments were carried out to investigate the influence of DR on the anodic and cathodic half reactions of the corrosion process.

Parts a and b of Figure 7 show typical polarization curves for mild steel specimens in 0.5 M H₂SO₄ and 1 M HCl, in the absence and presence of DR. Accordingly, the mild steel specimen in both acidic environments shows active dissolution with no distinctive transition to passivation within the studied potential range.

The obtained electrochemical parameters, namely, corrosion potential (E_{corr}), and corrosion current density (I_{corr}) were obtained and their values are presented in Table 1. The data presented therein reveals that in both acidic environments the I_{corr} decreases in the presence of the inhibitor compared to the uninhibited solution and the trend continues with an increase in the concentration of the inhibitor. In 0.5 M H₂SO₄ and 1 M HCl the polarization curves in presence of DR (Figure 7 a and b) show evidence of inhibition. It is obvious that DR has no significant effect on E_{corr} , however, a shift of both the cathodic and anodic curves to lower corrosion current densities was observed and this effect becomes more significant with increasing DR concentration in the studied environments.

The results obtained show that the rate of mild steel dissolution in 0.5 M H₂SO₄ is higher than that in 1 M HCl, which is in agreement with previous reports [26,27]. The differences in the polarization profiles in 1 M HCl and 0.5 M H₂SO₄ solutions possibly suggest that the acid anions influenced the processes in different ways.

The values of the corrosion current density in the absence and presence of AA were used to determine the inhibition efficiency from polarization data as follows:

$$IE\% = \left(\frac{I_{\text{corr}(bl)} - I_{\text{corr}(inh)}}{I_{\text{corr}(bl)}} \right) \times 100 \quad (\text{Eq. 2})$$

The values obtained are presented in Table 1. The observed reduction in anodic and cathodic corrosion current densities shows that the inhibitor reduced the mechanism of H_2 gas evolution reaction [28] and also the anodic dissolution of mild steel. If the displacement in E_{corr} is greater than 85 mV with reference to E_{corr} (in the absence of the inhibitor) the inhibitor may act as a cathodic or anodic type and if the displacement is less than 85 mV the inhibitor may be regarded as a mixed-type. In our result, displacement of E_{corr} is less than 85 mV; therefore, DR is classed as a mixed-type inhibitor.

Fig. 7 Potentiodynamic polarization curves of mild steel in: (a) 0.5 M H_2SO_4 and (b) 1 M HCl solutions in the absence and presence of DR at $30^\circ C$.

Table 1 Polarization Parameters for Mild Steel in 0.5 M H_2SO_4 and 1 M HCl in the Absence and Presence of DR at $30^\circ C$

System	E_{corr} (E vs SCE)	I_{corr} ($\mu A\ cm^2$)	IE
0.5 M H_2SO_4	- 482.5	168	
50 mg/L DR	- 495.2	115	31.5
1200 mg/L DR	- 479.2	30.6	81.7
1 M HCl	- 476.7	75.8	
50 mg/L DR	- 462.6	47.7	37.1
1200 mg/L DR	- 471.2	15.9	79

Electrochemical impedance spectroscopy measurements

Figure 8 (a) and (b) represent the Nyquist plots for mild steel in 0.5 M H₂SO₄ and 1 M HCl in the absence and presence of different concentrations of DR. The experiment was carried at the open circuit potential. In a 0.5 M H₂SO₄ solution, the Nyquist plots in the presence of DR are characterized by a capacitive loop at high-to-medium frequency. The capacitive loop at high frequencies shows the phenomenon associated with the double electric layer. This arises from the time constant of the electrical double layer and charge transfer in the corrosion process [29].

In 1 M HCl environment, the corresponding Nyquist plots show single semicircles for all systems over the frequency range studied, relating to one time constant in the Bode plots. The high frequency intercept with the real axis in the Nyquist plots is assigned to the solution resistance (R_s) and the low frequency intercept with the real axis is ascribed to the charge transfer resistance (R_{ct}). It is evident in Figure 7 (a) and (b) that introduction of DR to the acidic environments result to an increase in the charge transfer resistance which points towards inhibition of the corrosion process.

In order to determine the numerical values of the various impedance parameters presented in Table 2, the impedance spectra were analyzed by fitting to the equivalent circuit model $R_s (Q_{dl} R_{ct})$ which was used before to model the mild steel/acid interface [30 -33]. An example of the equivalent circuit model is presented in Figure 9. From the data presented in Table 2, it is clear that introduction of DR lead to an increase in the R_{ct} values at all concentrations in 0.5 M H₂SO₄ and 1 M HCl. The values of double-layer capacitance (C_{dl}) were determined using the relation;

$$f(Z_{im(max)}) = \frac{1}{2\pi C_{dl} R_{ct}} \quad (\text{Eq. 3})$$

Due to a modification on introduction of DR, the values of C_{dl} decrease more than observed in the absence of the inhibitor. Charge transfer resistance and double layer capacitance also show the opposite trend, according to Helmholtz model:

$$C_{dl} = \frac{\epsilon \epsilon_0 A}{\delta} \quad (\text{Eq. 4})$$

ϵ represents the dielectric constant of the medium, ϵ_0 is the vacuum permittivity, A is the electrode area, and δ is the thickness of the interfacial layer. The decrease in C_{dl} value which occurs due to a decrease in the dielectric constant and/or an increase in the double layer thickness can be related to the adsorption of DR species on a corroding metal surface. An increase in resistance with inhibitor concentration, suggests enhanced adsorption of DR species on a steel surface and efficient blocking of the steel surface [34]. Also, the inhibition efficiency ($IE\%$) from EIS data was determined by comparing the values of the charge transfer resistance in the absence and presence of DR using relation;

$$IE\% = \left(\frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \right) \times 100 \quad (\text{Eq. 5})$$

The obtained inhibition efficiency values are shown in Table 2. Though the result follows the same trend, however, a slight variation in the computed corrosion rate and inhibition efficiency values was observed from polarization and impedance measurements. This could be ascribed to the processes associated with the different techniques. For example, the mild steel surfaces were held close to the equilibrium corrosion potential for impedance measurements, but taken far away from the equilibrium potential during polarization measurements, which should influence the measured values from both techniques (35).

Fig. 8 Electrochemical impedance parameters of mild steel in: (a) 0.5 M H_2SO_4 and (b) 1 M HCl solutions in the absence and presence of DR at 30°C .

Fig. 9 Equivalent circuit for modeling impedance result

Table 2 Electrochemical Impedance Parameters of Mild Steel in 0.5 M H₂SO₄ and 1 M HCl in the Absence and Presence of DR at 30°C

System	R _s (Ω cm ²)	R _{ct} (Ω cm ²)	n	C _{dl} (μΩ ⁻¹ S ⁿ cm ⁻²)	IE (%)
0.5 M H₂SO₄	2.303	29.8	0.88	1.131E-4	
50 mg/L DR	2.512	39.9	0.89	5.144E-5	25.3
1200 mg/L DR	2.832	85.2	0.89	4.371E-5	65
1 M HCl	1.738	102.6	0.89	2.340E-5	
50 mg/L DR	1.826	160.5	0.89	1.050E-5	36.1
1200 mg/L DR	1.854	300.1	0.88	6.595E-6	60.5

Scanning electron microscopy

The surface micrographs of mild steel dipped in the absence and presence of optimum concentration of DR (1200 mg/L) in both acidic solutions are shown in Figure 10. Careful scrutiny of the picture, reveal that the surface of the mild steel appears rough due to active dissolution in the absence of the inhibitor. However, the roughness reduced significantly after the introduction of DR to the acid environments. The findings show that the presence of DR molecules reduced the active dissolution of mild steel by formation of a protective film on its surface. Examination of the images presented in Figure 10 shows that the metal surface was more protected in 0.5 M H₂SO₄ [Figure 10a (ii)] compared to 1 M HCl (Figure 10b (ii)) environment, which is in agreement with the values of inhibition efficiency obtained in both environments.

Fig. 10 Scanning electron micrographs of a mild steel surface immersed for 24 h in: a(i) 0.5 M H₂SO₄, b(i) 1 M HCl, a(ii) presence of 1200 mg/L DR (0.5 M H₂SO₄), b(ii) presence of 1200 mg/L DR (1 M HCl).

Adsorption considerations

In 0.5 M H_2SO_4 and 1 M HCl solutions, some of the constituents of DR are protonated whereas the rest remain in the molecular form. So, the observed inhibiting efficiency of DR can be ascribed to the participation of both protonated and molecular species in the adsorption process. Again, the mixed type inhibition mechanism suggested by the potentiodynamic polarization data gives evidence of adsorption of protonated and molecular species from the DR extract in 0.5 M H_2SO_4 and 1 M HCl solutions. The positive surface charge on mild steel in 0.5 M H_2SO_4 and 1 M HCl environment hinders the adsorption of protonated species, as a result of electrostatic repulsion. However, in 1 M HCl environment, the chloride ions present can be strongly adsorbed on the metal surface and thus facilitate adsorption of protonated species. The observed effect is not outstanding with sulfate ions, ensures a pronounced adsorption of protonated inhibitor species on the mild steel surface dipped in 1 M HCl , while in 0.5 M H_2SO_4 environment the metal specimen is more predisposed to adsorption of non protonated species, yielding the inhibition efficiencies obtained.

Adsorption of DR species on a corroding metal surface can be explained by using a suitable isotherm, which explains the variation of experimentally obtained values of the amount of adsorbed substance by unit area of the metal surface with its concentration in bulk solution at a constant temperature. For us to determine the adsorption mode of DR molecules on the metal surface in the different acid environments experimental data obtained from gravimetric result were tested by fitting to several adsorption isotherms (not shown) and the best fit was obtained from the Langmuir isotherm. If we assume a direct relationship between inhibition efficiency and the degree of surface coverage values (θ) defined as $\theta = \text{IE}/100$ for different inhibitor concentrations, data derived from gravimetric measurements were adapted to determine the adsorption characteristics of DR on mild steel in different acid solutions according to the Langmuir equation.

$$C/\theta = 1/b + C \quad (\text{Eq. 6})$$

Figure 11 shows the plot of C/θ against C to be linear in both acid environments, with slopes of 0.9942 (0.5 M H_2SO_4), and 1.318 (1 M HCl) respectively, showing that the adsorption of DR species onto a mild steel surface obeys Langmuir adsorption isotherm. The observed deviations of the slopes from the predicted values of 1.00 can be attributed to the interactions between adsorbate species on the metal surface as well as changes in the adsorption heat with increasing surface coverage, factors which were not taken into consideration in deriving the isotherm.

Fig. 11 Langmuir adsorption isotherms for DR extract on mild steel in 0.5 M H_2SO_4 and 1 M HCl solutions

Conclusions

The Delonixregia extract was found to be a good inhibitor for corrosion of mild steel in the acidic environments (0.5 M H₂SO₄ and 1 M HCl). The inhibition efficiency was found to increase with increased inhibitor concentration. Potentiodynamic polarization results indicate that DR is a mixed type inhibitor, affecting both the anodic metal dissolution reaction and the cathodic hydrogen evolution reaction, whereas the impedance results revealed adsorption of the DR species on a corroding steel surface. The adsorption of the DR on mild steel surface followed Langmuir adsorption isotherm. Thus, indicating that there was a homogeneous layer of adsorption on the mild steel surface.

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