

Investigation of *Podranea ricasoliana* Leaves Extract as Green Corrosion Inhibitor for Mild Steel in 1M HCl

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Abstract

The inhibition effect of *Podranea ricasoliana* leaves extract (PRL) on mild steel corrosion in 1M HCl has been studied by mass loss and potentiodynamic polarization. The results infer that the inhibition efficiency increased with increase in concentration of the plant extract and corrosion rate increased with increase in temperature. Experimental results were fitted into various adsorption isotherms. Electrochemical impedance results confirmed the inhibitive nature and tafel polarization studies indicated mixed mode of inhibition of the PRL extract. Based on the experimental studies, a possible mechanism was suggested. Surface analytical techniques were carried out using FT-IR and Scanning electron microscope (SEM). The overall result suggested that PRL extract could serve as a effective inhibitor to minimise corrosion..

Keywords: *Podranea ricasoliana*, corrosion inhibition, Tafel Polarization, SEM, FT-IR

1. Introduction

Acid solutions are more prone to corrosion to any metals but they are widely used as pickling descaling and cleaning agents for the removal of undesirable oxide films and corrosion products [1]. A common method followed to inhibit corrosion of metals in an

aggressive medium is the addition of some compounds to the medium

which is in contact with surface in order to reduce corrosion rate [2]. Several organic compounds containing O,S and N as heteroatoms have been reported as corrosion inhibitors for mild steel in acidic environment [3-8]. Nowadays, the uses of some chemical inhibitors have been limited because of some reasons: their synthesis is often very expensive and they can be hazardous for human beings and environment as well (9). Recently, plant extracts are employed as corrosion inhibitors as they are biodegradable and eco-friendly in nature. Literature survey revealed the use of several plant extracts as corrosion inhibitors in different media [10-13]. The use of plant extracts have drawn attention because they are nontoxic, environmentally acceptable, inexpensive, readily available and renewable sources of materials [14-16].

Our research team also investigated the plant extracts for corrosion inhibition studies which include sprouted seeds of *Phaseolus aureus*, *Cocos nucifera* Shell, seed extract of *Cyamopsis tetragonoloba*, petiole extract of *Cocos nucifera*, staminate flower extract of *Cocos nucifera* and, leaf extract of *Ervatamia*

coronaria, Cocos nucifera L. Peduncle, leaf extract of Mundulea sericea, leaves extract of Dodonaea Viscosa(L) [17-26]. Therefore, the present focus is to investigate the corrosion behavior of mild steel in 1 M HCl solution in the presence of PRL using weight loss, polarization resistance, Tafel polarization, and electrochemical impedance techniques.

Podranea ricasoliana with its glossy foliage and abundance of attractive pink flowers is a very showy plant, well known to many Asian gardeners. **Ahmed Mohamed El-Moghazy(2004)** isolated fourteen compounds using different chromatographic techniques and identified by means of physical, chemical and various spectroscopic methods[31]. The isolated compounds are: four sterols and triterpenes, six iridoid glucosides (two of them are new), two phenylpropanoid glycosides, one flavonoid and one phenolic acid.

The aim of the present work is to study the effect of *Podranea ricasoliana leaves* (PRL) extract on the behaviour of mild steel corrosion in 1M HCl solution.

2. Materials and Methods

2.1 Preparation of Specimen

Mild steel (MS) specimens of the composition C-0.089%, Mn-0.215%, P-0.017%, Mo-0.021% and remaining % of Fe were chosen for the present study. Mild steel specimens of size 1X5 cm² were used for weight loss studies while MS of 1 cm² were used for electrochemical studies. A hole was drilled in the specimen, mechanically polished, degreased, washed with de ionized water then thoroughly dried and kept in desiccators for weight loss tests.

2.2 Preparation of *Podranea ricasoliana* inhibitor

The selected eco-friendly plant extract of *Podranea ricasoliana* leaves (PRL) was prepared by refluxing 25g of leaves in 500ml of 1M HCl for three hours and kept overnight for cooling. The cooled extract was filtered and made up to 500ml with 1M HCl to get the stock solution. (5% extract).

2.3 Mass loss study

Mass loss method

Pre weighed test pieces were immersed in triplicate in 100 ml of the solution containing various concentration of the inhibitor and in the absence of inhibitor for a

predetermined time period. The test specimens were removed and then washed with de-ionised water, dried and reweighed. From the initial and final masses of the specimen, the mass loss was calculated as per ASTM[27] G1-2[28]. From the mass loss, corrosion rate, inhibition efficiency and surface coverage were determined using the following relationship.

$$\text{Corrosion Rate (mpy)} = \frac{K \times W}{DAT} \quad (1)$$

K = Constant-3.45*10⁶ (mpy), W = Mass loss in grams; D = Density of mild steel in mg / cm³; A = Area of the specimen in cm²; T = Exposure time in hours

$$\text{Inhibitor Efficiency (\%)} = \frac{CR_{blank} - CR_{inh}}{CR_{blank}} \times 100 \quad (2)$$

CR_{blank} = Corrosion rate of mild steel in acidic medium, CR_{inh} = Corrosion rate of mild steel in the presence of inhibitor.

$$\text{Surface Coverage (\theta)} = \frac{W_o - W}{W_o} \quad (3)$$

W_o = Mass loss of mild steel without inhibitor (blank); W = Mass loss of mild steel in the presence of the inhibitor.

2.4 Electrochemical studies

Electrochemical studies were carried out using conventional three electrode cell with platinum as counter electrode, saturated calomel electrode (SCE) as reference electrode and sample (MS) as working electrode. The Bio-Logic electro chemical analyzer interfaced with an IBM computer was used for the polarization study. Before starting the measurements the electrode potential was allowed to stabilize for 30 minutes. The impedance diagrams are given in Nyquist representations [29]. The impedance and polarization parameters such as double layer capacitance (C_{dl}), charge transfer resistance (R_{ct}), corrosion current (I_{corr}), corrosion potential (E_{corr}), anodic Tafel slope (b_a) and cathodic Tafel slope (b_c) are calculated.

2.5 Characterization techniques

The FT-IR and SEM analysis were carried out on the surface of MS before immersion and this after immersion in acidic solutions with and without the optimal concentration of *Podranea ricasoliana* leaves extract (PRL).

3. Results and discussion

3.1 Mass loss measurements

The data obtained for the corrosion behaviour of mild steel in 1 M HCl solution containing PRL within the concentration range of 0.1–0.8% at different time of immersion (1/2h, 1h, 3h,6h and 24h) are presented in Fig. 1. It is seen from the table, that the corrosion rate decreases significantly in the presence of the extract compared to the blank acid solution and is also found to be dependent on the concentration of the extract. For instance the % inhibition efficiency values of 95.7% are obtained at the highest extract concentration for PRL in 1M HCl, at three hours period of immersion. The enhanced IE at higher concentration is due to the increase in the surface coverage of mild steel surface.

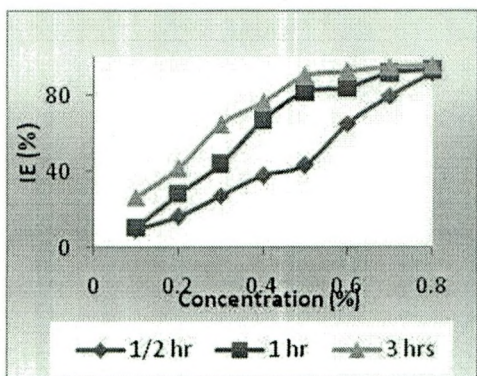


Figure 1 - IE as a function of concentration

Examination of Fig.2 revealed a decreasing trend in inhibition efficiency with increasing temperatures for all the system studied. This suggests possible desorption of some of the adsorbed inhibitor from the metal surface at higher temperatures. Such behaviour reflects that the phytochemical constituents were physically adsorbed on the metal surface [30]. Also the corrosion rate increased with increase in temperature and greater rate of the mild steel specimen was recorded at 326K both in the absence and presence of PRL inhibitor. Maximum inhibition efficiency of 94.6% at 0.8% concentration at 316K has been observed. The corrosion-inhibiting effect of PRL extract can be attributed to the adsorption of phytochemical

constituents including sterols, triterpenes, iridoid glucosides, phenylpropanoid glycosides, flavonoid and phenolic acid [31] on mild steel surface.

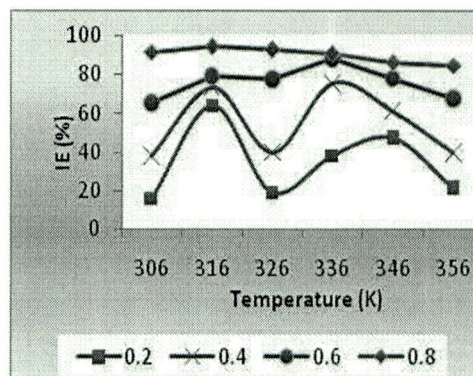


Figure 2 - IE as a function of temperature

3.2 Potentiodynamic polarization studies

Potentiodynamic polarization curves of MS in 1M HCl in the presence and absence of PRL extract is shown in fig-3. This figure indicates that corrosion potential values shifts to the positive values in the presence of the extract. Values of E_{corr} indicate that all the inhibitor behave as mixed type inhibitor by controlling the cathodic hydrogen evolution as well as anodic metal dissolution. Decrease in I_{corr} values and increase in R_p values with increase in concentration of inhibitor shows that the process has taken place by adsorption of the extract on MS surface. Further inspection of table 2 also reveals that both the anodic and cathodic Tafel slopes decrease upon addition of inhibitor and decrease further with increasing inhibitor concentration. This behaviour suggested that the extract inhibits the corrosion of MS via the adsorption of its molecules on both anodic and cathodic sites consequently, it acts as mixed inhibitor [32].

Table 1- Role of concentration on time and temperature for MS/PRL/HCl

S.No	Conc v/v(%)	TIME					TEMPERATURE				
		½ h	1 h	3 h	6h	24h	316K	326K	336K	346K	356K
		IE (%)			IE (%)	IE (%)	IE (%)	IE (%)	IE (%)	IE (%)	IE (%)
1	0.1	9	10	26.6	59.2	13.2	53.9	14.3	25.3	40.8	8.7
2	0.2	15.9	28	41.6	76.9	28.2	63.8	19.1	38.2	46.9	20.7
3	0.3	27.2	44	64.4	81.1	41.9	68.2	27.1	52.9	54.3	28.5
4	0.4	37.8	67	76.3	83.5	49	73.4	39.6	75.3	61.2	39.1
5	0.5	43.1	82	89.8	84.9	64.1	76.9	74.9	87.8	61.9	53.2
6	0.6	65.1	84	92.2	87.1	77.4	79.2	77.8	88.3	78	67.4
7	0.7	79.5	92	95	89.3	85.1	90.6	89.3	89.4	80.3	69.2
8	0.8	91.6	93	96	91	90.2	94.6	92.8	90.8	86.1	84.8

Table 2 – Potentiodynamic results 1M HCl for different concentration of PRL of MS/CI/PRL

Parameters	Blank	0.30%	0.50%	0.70%
E _{corr} mV/sec	501	473	490	456
I _{corr} (Amp/cm ²)	6589.157	2499.88	1256.74	924.76
b _a mV/dec	207.4	97.5	111.3	101.5
b _c mV/dec	203.2	220.4	200.9	192.1
IE(%)	-	62.06	80.93	85.97
R _p (Ω/cm ²)	7.14	26	37	45
IE (%)	-	72.54	80.7	84.13

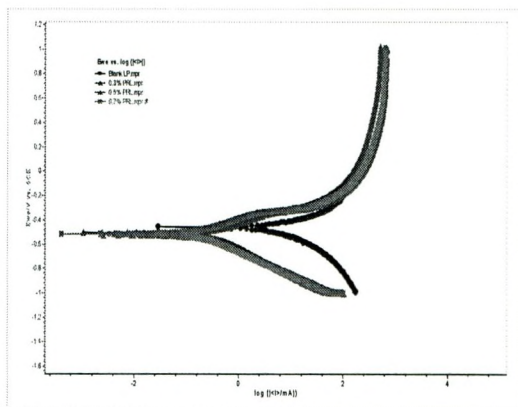


Figure 3 – Polarization curve of MS/HCl/PRL

3.3 ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

EIS is one of the most successful techniques used to evaluate the effectiveness of an inhibitor. The impedance spectra expressed exhibit one single depressed semicircle. It is clear from fig 4 that the diameters of the semicircle increases with the increase in concentration of PRL extract[32]. The semi-circular appearances show that the corrosion of MS is controlled by the charge transfer. The deviation from

the semicircle refers to the frequency dispersion of interfacial impedance [33]. This behaviour is ascribed to the in homogeneity of the metal surface arising from surface roughness. Consequently the high R_{ct} values of inhibited electrodes can be explained by the build-up of protective layers and the effective barrier on MS surface.

From Table 3, it is clear that the values of C_{dl} , decrease with an increase in the inhibitor concentration, this is due to an increase in surface coverage by the inhibitors i.e the thickness of protective layer increases, which results higher value of inhibition efficiency.

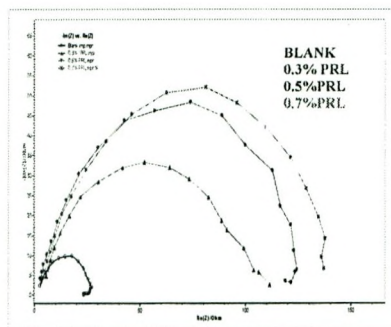


Figure 4- Impedance curve for MS/HCl/PRL

Table 3 - Impedance parameters for MS/HCl/PRL

Conc. (v/v) (%)	R_{ct} ($\Omega \text{ cm}^2$)	IE (%)	C_{dl} ($\mu \text{ F cm}^2$)	θ
Blank	22.59	-	173	-
0.3	115.8	80.49	108	0.3757
0.5	119.8	81.14	92	0.4682
0.7	126.5	82.14	50	0.711

The adsorption of inhibitors on the MS surface decreases its electrical capacity, because they displace the water molecules and other ions originally adsorbed on the surface of mild steel [32]. With the help of the double layer capacitance C_{dl} , θ can be calculated using the equation,

$$\theta = 1 - \frac{C_{dl}(\text{inhibited})}{C_{dl}(\text{blank})} \quad (4)$$

where, $C_{dl}(\text{inhibited})$ and $C_{dl}(\text{blank})$ are the double layer capacitance in the presence and absence of the inhibitor respectively.

Adsorption isotherm

Adsorption isotherm has been generally studied since organic molecules present in plant extract inhibit corrosion by adsorption at the metal solution interface. To determine the adsorption mode and its parameters, various isotherms were tested. A plot of $\log(\theta / (1-\theta))$ Vs $\log C$ and θ Vs $\log C$ gave a straight line which obviously shows that the adsorption of PRL can be fitted to Langmuir and Temkin adsorption isotherm.

KINETIC PARAMETERS

The effect of temperature on the inhibited acid-metal reaction is highly complex, because many changes occur on the metal surface, such as rapid etching desorption of the inhibitor. Generally the corrosion rate increases with the rise of temperature. It was found that the inhibition efficiency decreases with increasing temperature and increases with increasing the concentration of the inhibitor. The activation energy (E_a^*) of the corrosion process has been calculated using Arrhenius equation

$$\log CR = A \exp(-E_a^*/RT) \quad (5)$$

Where CR is corrosion rate and A is Arrhenius constant. The values of activation energies E_a^* can be obtained from the slope of the straight lines of plotting $\log CR$ Vs $1/T$ in the presence and absence of investigated compounds at various temperatures [Fig.5] and are given in Table 3. It was found that the E_a value of the blank was higher than that of the systems studied in the presence of the inhibitor. This is due to the formation of a film of inhibitors on mild steel. The activation energy for the corrosion of mild steel in 1M HCl is found to be around 50-60KJ/mol-1 which is in good agreement with the work carried out by others.

Activation parameters were calculated from Arrhenius equation

$$CR = \frac{RT}{Nh} \exp\left\{\frac{\Delta S_a}{R}\right\} \exp\left\{\frac{\Delta H_a}{R}\right\} \quad (6)$$

Where h is Planck's constant, N is Avogadro number, ΔS_a is entropy of activation, ΔH_a is enthalpy of activation. A plot of $\log(CR/T)$ Vs $1/T$ gave a straight line with a slope of $-\Delta H_a / 2.303R$ and an intercept of

$\log(R/N_h + S_a/2.303R)$ from which the values of ΔH_a and ΔS_a were calculated and listed in table 4.

It is clear that the values of E_a in the presence of the PRL inhibitor are higher than those in uninhibited acid solution. The increase in E_a in the presence of the inhibitor indicates the physical adsorption mechanism [34]. The decrease in the steel corrosion rate is mostly decided by apparent activation energy

The positive values of ΔH_a for PRL/HCl with and without the inhibitor reveal the endothermic nature of the steel dissolution process and indicate that the dissolution of steel is difficult [35]. Typically, the enthalpy of physisorption process is lower than that of 40 kJ/mol while the enthalpy of chemisorption process approaches 100 kJ/mol [36]. The large negative value of ΔS_a for mild steel in PRL/HCl implies that the activated complex in the rate determining step represents an association rather than a dissolution step. This means that a decrease in disordering takes place on going from reactants to the activated complex.

Table 4 – Adsorption parameters for MS/HCl/PRL

S.NO	Conc. v/v (%)	E_a (kJ/mol)	ΔH_a (kJ/mol)	ΔS_a (J/mol K)
1	Blank	43.3	46.6	-21.3
2	0.2	44.6	47.8	-21.3
3	0.4	43.6	46.4	-29
4	0.6	41.8	44.2	-41
3	0.8	57.9	62.2	6.2

Surface analytical techniques

Scanning electron microscopy

Surface examination using SEM is carried out to investigate the effect of the inhibitor on the surface morphology of the mild steel. Fig 5a shows the SEM image of the mild steel specimen after immersion in 1M HCl solution in the absence of extract. The micrograph reveals that the surface is strongly damaged in the absence of the inhibitor. Fig 5b shows images of the surface of mild steel specimens immersed for the same time interval in 1M HCl solution containing 0.8% PRL. The figure exposes that the surface damage has diminished in the presence of inhibitor. It is reveals that there is a good protective film adsorbed on specimens' surface which is responsible for inhibition of corrosion.



Fig 5A & 5 B: SEM images of Mild steel in the presence of a) 1M HCl and b) 1M HCl + 0.8% PRL extract

FTIR technique

FTIR is a powerful tool for identifying the functional groups associated with the adsorption of an inhibitor. By comparing the FTIR of PRL (Fig. 6a) with that of the corrosion product of mild steel when PRL is used as an inhibitor (Fig. 6b), functional groups responsible for adsorption can be deduced.

Table 5 - Frequencies and adsorption peaks of IR for PRL extract

Pure plant extract	Corrosion product	Frequency Assignment
3410	3464	O-H stretch
2924	-	O-H stretch
2376	-	C≡N stretch
-	2314	C≡N stretch
-	1975	C-H stretch due to Phenyl ring substitution
1627	-	C=C in ring aromatics
1442	-	C=C aromatic stretch
1257	1249	C-O stretch
1072	1018	C-O stretch
-	879	C-H oop due to aromatic compound
-	717	C-H bend

The frequencies and peaks of IR adsorption deduced from the spectra are recorded in Table 5. From the results obtained, the C-O bend at 1072 cm⁻¹ is shifted to 1018 cm⁻¹ and the O-H stretch at 3410 cm⁻¹ is shifted to 3464 cm⁻¹. These shifts imply that there is interaction between the inhibitor's molecules and Fe in mild steel. However, some functional groups including the C=C stretch at 1627 cm⁻¹, 1442 cm⁻¹, the C≡N stretch at 2376 cm⁻¹ are missing in the spectrum of the corrosion product indicating that these bonds might have been involved in bonding [37]. Comparing the FTIR results, it can be stated that the mechanism for inhibition of mild steel by acid extract of PRL involves synergistic adsorption through the aromatic ring double bond and -OH functional groups in sterols, triterpenes, iridoid glucosides, phenyl propanoid glycosides, flavonoid and phenolic acid.

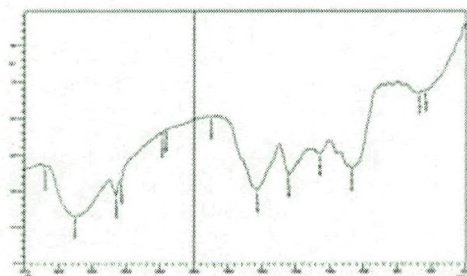


Figure 6A - FTIR spectrum of acid extracts of PRL

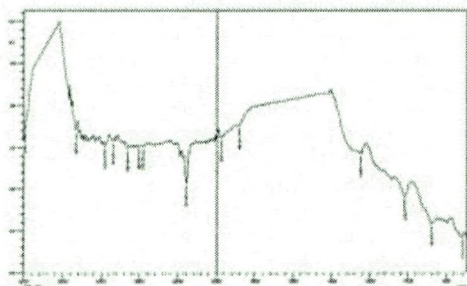


Figure 6B - FTIR spectrum of the corrosion product of mild steel.

Mechanism of corrosion inhibition

The inhibition performance of PRL extract for mild steel in 1 M HCl interface depends on the extent of adsorption and adsorption depends on several factors such as the number of adsorption sites, molecular size,

and mode of interaction with the metal surface and extent of formation of metallic complexes[38]. Literature survey reflects the presence of the phytochemicals namely sterols, triterpenes, six iridoid glucosides, two phenylpropanoid glycosides, one flavonoid and one phenolic acid, Ursolic acid, Leucine and stigmasterol[31]. Accordingly, the inhibitive action of PRL could be attributed to the adsorption of its components on the mild steel surface. In aqueous acidic solution, the organic molecules of PRL exist either as neutral molecules or in the form of protonated organic molecules (cation).

It is not possible to consider a single adsorption mode between inhibitor and metal surface because of the complex nature of adsorption and inhibition of a given inhibitor.

Generally, two modes of adsorption are considered on the metal surface in acid media. In one mode, the neutral molecules may be adsorbed on the surface of mild steel involving the displacement of water molecules from the mild steel surface and the sharing electrons between the N,O atoms and iron. The inhibitor molecules can also adsorb on the mild steel surface on the basis of donor-acceptor interactions between π-electrons of the aromatic ring and vacant d-orbitals of surface iron atoms.

In second, one since it is well known that it is difficult for the protonated molecules to approach the positively charged mild steel surface (H₃O⁺/metal interface) due to the electrostatic repulsion. Since Cl⁻ have a smaller degree of hydration, they could bring excess negative charges in the vicinity of the interface and favour more adsorption of the positively charged inhibitor molecules, the protonated inhibitors adsorb through electrostatic interactions between the positively charged molecules and the negatively charged metal surface. Thus, there is synergism between adsorbed Cl⁻ ions and protonated inhibitors.

Thus, we can assume that inhibition of mild steel corrosion in 1 M HCl is due to the adsorption of extract constituents on the mild steel surface

Table 6: Phytochemical components in PRL

PHYTO CONSTITUENTS	REFERENCE
Sterols	AhmedMohamedEl-Moghazyetal,2004
Triterpenes	
Iridoidandphenylpropanoid Glucosides	
Phenolicacid	
Flavonoid	

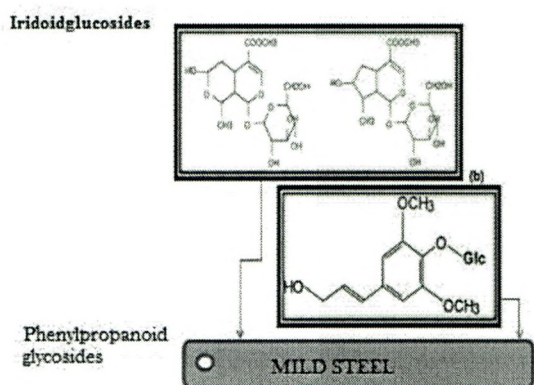


Figure 7—Schematic representation of adsorption model in presence of PRL

4. Conclusions

The results presented in this work show that the PRL extract inhibit the corrosion of mild steel in HCl solution to an appreciable extent. The inhibition efficiencies of the plant extracts increased with increase in extract concentration but decreases with increase in temperature. The experimental data fit into the Langmuir and Temkin adsorption isotherm model. FT-IR and SEM show that the compounds present in plant extracts form corrosion inhibitive layer by reacting with iron ions present on the mild steel surface. Electrochemical measurements show that PRL is a mixed type of inhibitor. The results obtained from mass loss, potentiodynamic polarization, and EIS techniques are in good agreement.

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