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Research Article

Impediment Effect of *Galinsoga parviflora* (Quick Weed) on Mild Steel Corrosion in 1 M HCl

P. Divya[1], R. Saratha[1] and S.V. Priya[2]*

[1]Department of Chemistry, Avinashilingam Institute for Home Science and Higher Education for Women **University**, Coimbatore, Tamil Nadu, India
[2]Department of Chemistry, Kongu Engineering College, Centre for Environmental Research, Perundurai, Tamil Nadu, India

Abstract

The effectiveness of *Galinsoga parviflora* (Quick Weed) leaf (GP) as corrosion inhibitor of mild steel in 1 M HCl solution was studied by weight loss, potentiodynamic polarization curves and electrochemical impedance spectroscopy. The corrosion rate of mild steel and inhibition efficiencies were calculated. The efficiency of the inhibitor was found to increase with increase in concentration of the inhibitor and the inhibition efficiency of above 90% was obtained at 6% V/V of the inhibitor. The optimum temperature for maximum efficiency (99.04) is 323 K at 6% V/V concentration of the extract. The inhibitive actions of plant extract are discussed on the basis of adsorption of stable complex at the mild steel surface. Theoretical fitting of different isotherms were tested to clarify the nature of adsorption. Polarization curves revealed that GP acts as a mixed type inhibitor.

Keywords: Corrosion inhibitors, Mild steel, Weight loss method, Polarization

***Correspondence**
Author: S.V. Priya
Email: viswa.shanmugam84@gmail.com

Introduction

One of the very important methods of minimizing corrosion today is the use of corrosion inhibitor. Inhibitors are extensively used in metal pickling and vary in type according to the acid used in the pickling operation. During pickling, corrosion inhibitors are added to the solution in order to reduce the degree of metal attack and rate of acid consumption. Several authors conducted a lot of studies to find effective inhibitors for metals in different media. [1-4]. Most of the corrosion inhibitors are expensive synthetic chemicals having hazardous properties to living creatures and environment. Research activities in recent times are geared towards finding alternative corrosion inhibitors to replace non-biodegradable and toxic organic or inorganic compounds. Naturally occurring substances satisfy this need [5-9]. These are readily available, cost effective, renewable source of materials, eco-friendly and biodegradable. The present research is to replace highly toxic substances with less or non-toxic environmentally safe materials for corrosion resistance. Our previous works [10, 11] reported a successful use of Crossandra infundibuliformis leaves and Citrus aurantiifolia leaves extract as corrosion inhibitors for mild steel in 1 M HCl respectively. The present work is another trial to find a naturally occurring cheap and environmentally safe substance that could be used for inhibiting the corrosion of mild steel in 1 M HCl medium. The leaves of Galinsoga parviflora is selected since it occurs naturally and is easily available. It is grown as ornamental plants and so there is ample availability for this plant.

Material and Methods

The sheet of cold rolled mild steel, which is commercially accessible in the market, were appliance interested in specimens of vicinity 7.5×2 cm^2. Fissures were bradawl on the interior of single end of every specimen for deferral. These specimens were degreased, sterile with emery tabloid and rinsed with deionised water. The specimens were gathered in desiccators in the absence of humidity prior to their utilization for the experiment. The composition of mild steel has been analyzed using ARL 3460 metal analyzer (Optical emission spectrometer). It was found to contain the following composition. Silicon-0.013%, Carbon-0.100%, Phosphorus-0.026%, Molybdenum-0.016%, Chromium-0.059%, Manganese-0.27%, Nickel-0.012%, Sulphur-0.011% and Iron-99.493%.

Acid emulsions are extensively used in assorted traders for pickling of ferrous alloys and steels. They are too worn in lubricate fabrication to inspire and amplify lubricate and air surge and to descale coated in invention wells. Therefore the revise of corrosion and impendence on mercantile mild steel in acid medium is of greatly engrossed and it has expanded enormous magnitude in the meadow of industrial and manufacturing concerning electrochemical practices. 1 M Hydrochloric acid is preferred for the study. All the solutions intended for the research were prepared with commercial quality reagents in deionised water.

Plant substance which is freely accessible and ecological is certain to perform as inhibitor. The selected plant substance is, *Galinsoga parviflora* leaves (Mookuthi Poo).The leaves of GP are available in all over Tamilnadu. For this study the leaves are collected from North Coimbatore area in Tamilnadu. The leaves were dimness dehydrated and pulverized.

The extract was prepared by refluxing 25 g of pulverized leaves in 500 mL of 1 M Hydrochloric acid for 3 hours and reserved it over night and was filtered and made equal to 500 mL with the same acid. From this reserve solution, various concentrations of the inhibitor were prepared. The refluxing setup is shown in **Figure 1**.

Figure 1 Refluxing method.

Mass loss method

Mild steel specimens precisely weighed aiding Denver balance were smarmy immersed in triplicate in 250 mL of 1 M Hydrochloric acid in the dearth and existence of assorted concentrations of the extract. At the terminate of the analysis, the specimens were saturated in Sodium carbonate, sluiced with deionised water, dehydrated, desiccated for half an hour and subsequently recrudesced. The slaughter in weight was premeditated from the divergence among the earlier and terminates of the trial weights. The typical of the triplicate values were aided for corrosion rate computation. The mass loss method was espoused for assorted concentrations of the extract, distinct time periods of immersion and at prominent temperatures. The experiment was carried out in laboratory is represented in **Figure 2**.

Figure 2 Thermostat.

Electrochemical Studies

Electrochemical experimentations were conceded out in a glass cell with a competence of 100 mL. A platinum electrode and a saturated calomel electrode (SCE) were used as counter electrode and reference electrode correspondingly. The working electrode (WE) was mild steel slip used for Mass loss method but lacquered as to expose an area of 1 cm^2.

Potentiodynamic polarization method

Potentiodynamic polarization was done using a Biological 5.4 version with a software envelope of Corrware, Corrview, Z-plot and Z-view. The a.c impedance dimensions were executed at corrosion potentials (Ecorr) over a frequency range of 10 khz to 20 mhz, with signal amplitude on perturbation of 10 mv. Nyquist were acquired from the results of these experiments.

Electrochemical measurement unit and three electrode cell

Corrosion potentials and corrosion current densities were unwavering by extrapolating the cathodic and anodic Tafel regions from the potentiodynamic polarization curves, the intersect gives the corrosion current and corrosion potential. The electrochemical unit is shown in the **Figure 3**.

Figure 3 Biological 5.4 version

Inhibition Efficiencies (%) were calculated as follows,

Tafel method

$$I.E\,\% = \frac{Icorr - I'corr}{Icorr} \times 100$$

Where, Icorr is the corrosion current density in the absence of the inhibitor, I'corr is the corrosion current density in the presence of the inhibitor.

Linear polarization method

$$I.E\% = \frac{Rp\,(inh) - Rp(blank)}{Rp\,(inh)} \times 100$$

Where, Rp(inh) is the polarization resistance in the presence of inhibitor, Rp(blank) is the polarization resistance in the absence of inhibitor

Impedance Studies

Consequences of the charge transfer resistance Rct were acquired from the plots by influential the difference in the values of impendence at low and high frequencies. The inhibition efficiency is calculated from the charge transfer resistance using the formula,

$$I.E\% = \frac{R\,(ct/inh) - R(ct)}{R\,(inh)} \times 100$$

Where, Rct and Rct/inh are the charge transfer resistance values in absence and in the presence of the inhibitor, correspondingly.

Phytochemical Constituents Analysis

In the present study, the phytochemical constituents typically present in the extract was determined as detailed below:

- ***Carbohydrates:*** To the test solution added small amount of Anthrone, few drops of concentrated sulphuric acid and heated. A black precipitate was obtained. Confirmed the presence of Carbohydrate.
- ***Alkaloids:*** To the test solution added 2 mL of dilute hydrochloric acid, shaken and decants the aqueous layer and added few drops of Wagner reagent. A red precipitate was obtained. Confirmed the presence of Alkaloids.
- ***Flavanoids:*** To the test solution in alcohol added small amount of Mg powder, 3 drops of 2 N concentrated hydrochloric acid and heated and cooled. A Yellow precipitate was obtained. Confirmed the presence of Flavanoids.
- ***Saponins:*** To the test solution added water and shaken it well. A foamy layer was formed for 1 cm. Confirmed the presence of Saponins.
- ***Tannins:*** To the test solution added water and lead acetate. A white precipitate was obtained. Confirmed the presence of Tannins.
- ***Phytosterol:*** To the test solution in chloroform and add conc. sulphuric acid. A golden yellow layer is formed below. Confirmed the presence of Phytosterol.

The phytochemical analysis was done in the laboratory is shown in **Figure 4**.

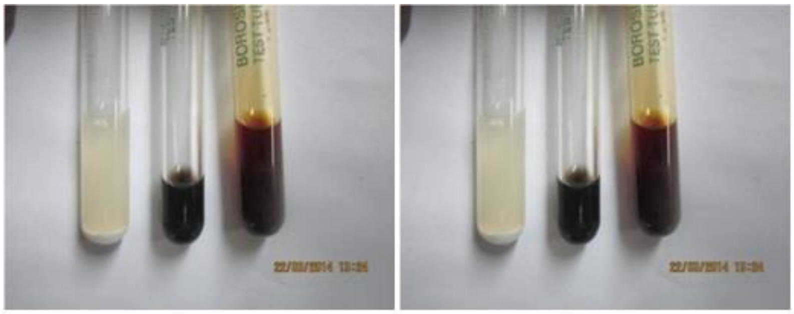


Figure 4 Phytochemical Analysis

Weight loss measurements
Effect of inhibitor concentration

The inhibition efficiency of various concentrations of the extracts of GP for the dissolution of mild steel in 1 M Hydrochloric acid is obtained from weight loss measurement and presented in the **Table 1**. From Table, it is evident that the inhibition efficiency increased (93.71-98.08%) with increase in concentration of the extract. This behaviour may be attributed to the adsorption of the phytochemical constituents on the metal surface [12]. The adsorption of the phytoconstituents on the metal surface makes a barrier for mass and charge transfer thus protecting the metal surface from corrosion. The degree of protection increases with increasing surface fraction occupied by the adsorption molecules. The maximum efficiency (98.08%) is obtained at 6% V/V. Analyzing the table thoroughly it seems that inhibition efficiency does not vary much from 5.5% V/V (97.77%).

Table 1 Variation of inhibition efficiency of GP extract in 1M Hydrochloric acid at different concentrations and various immersion periods.

Conc. of the extract (% v/v)	Immersion period in hours / Inhibition efficiency (%)				
	1	**3**	**5**	**24**	**48**
2.5	26.32	56.75	60.86	94.19	93.71
3	38.17	57.33	59.97	95.75	95.77
3.5	34.45	59.58	61.52	96.18	95.99
4	51.07	60.06	66.07	96.49	96.91
4.5	51.84	63.10	69.92	96.84	97.24
5	56.89	64.83	76.46	97.03	97.66
5.5	62.17	69.36	78.83	97.47	97.77
6	78.73	71.64	83.57	97.08	98.08

Effect of immersion period

The variation of inhibition efficiency of GP with immersion period is given in the Table 1. It is seen that the inhibition efficiency is increased (93.71-98.08%) with immersion time (1 h – 48 h) and attained a maximum at 48 h. The results show that the extract acts as a good inhibitor in 1 M Hydrochloric acid. The increase in inhibition efficiency in the period of immersion may be due to the increase in adsorption of phytoconstituents of the extract on the surface of mild steel with time.

Temperature study

Temperature has a greater impact on the rate of metal dissolution. Temperature effect on acidic corrosion, most often in Hydrochloric acid has been the object of larger number of investigations. Corrosion rate approximately doubles for every 100 °C rise in temperature for corrosion accompanied by hydrogen gas evolution as in normal chemical reactions. The results of the experimental study on the effect of temperature on mild steel corrosion in 1 M Hydrochloric acid in the presence of various concentrations of the extract of GP is given in the **Table 2** and represented in **Figure 5**. The IE increased with increase in temperature till 323 K and decreased with further increase in temperature. The decrease in efficiency at 333 K and 343 K may be due to desorption of the molecules of the extract from the surface of the mild steel after adsorption till 323 K. The optimum temperature for maximum efficiency (99.04) is 323 K at 6 % V/V concentration of the extract. At each temperature, the IE increased with increase in concentration of the extract.

Table 2 Inhibition efficiency of GP extract in 1M Hydrochloric acid at different concentrations and various temperatures.

Conc. of the extract (% v/v)	Temperature (K) and Inhibition efficiency (%)				
	303	**313**	**323**	**333**	**343**
2.5	26.32	77.4	88.8	91.1	90.1
3	38.17	82.4	96.2	91.8	90.7
3.5	34.45	83.1	97.2	92.3	93.2
4	51.07	85.6	97.5	92.9	93.5
4.5	51.84	86.7	98	93.7	93.5
5	56.87	88.3	98.9	94.8	94.4
5.5	62.17	92.6	99.1	95.8	94.9
6	78.73	94.7	99.4	96.2	96.2

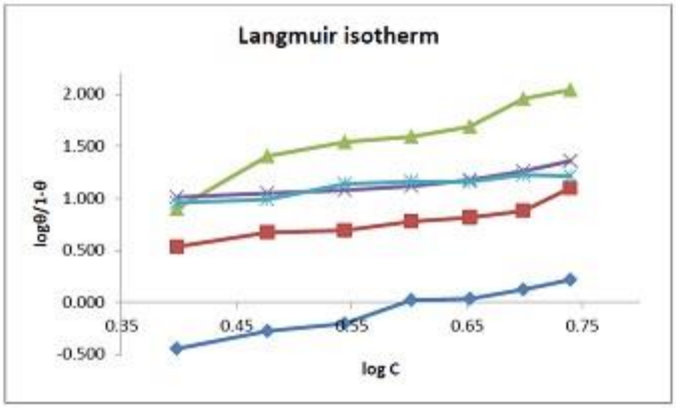


Figure 5 Langmuir adsorption isotherm for the dissolution of mild steel in 1M Hydrochloric acid in the absence and in the presence of various concentration of GP extract.

Adsorption isotherm

The adsorption characteristic of the extract is evaluated by plotting surface courage against inhibitor concentration using the common adsorption isotherms namely Langmuir, Freundlich and Temkin isotherms.

All these isotherms are of the general form;

$$F(\theta,x)\exp(-2a\theta) = kc$$

Langmuir adsorption isotherm

The plot of log θ/1-θ vs log C for the dissolution of mild steel in 1 M Hydrochloric acid in the absence and in the presence of various concentrations of GP extract at different temperature is presented in the Figure 5. The adsorption of the extract follows Langmuir isotherm with perfect linearity at 333 K and 343 K for 1 M Hydrochloric acid as adsorption follows Langmuir isotherm implies monolayer adsorption of the phytoconstituents present in the extract on the mild steel surface. Though the Langmuir plots are linear the deviation of slopes from unity can be attributed to the molecular interaction among adsorption species [13].

Freundlich adsorption isotherm

The plot of log θ vs log C for the dissolution of mild steel in 1 M Hydrochloric acid in the presence of various concentrations of GP extract with various temperatures is shown in the **Figure 6**. The adsorption coefficient, slope and linear correlation coefficients from the regression between log θ and log C for 1 M Hydrochloric acid are listed in the **Table 3**.

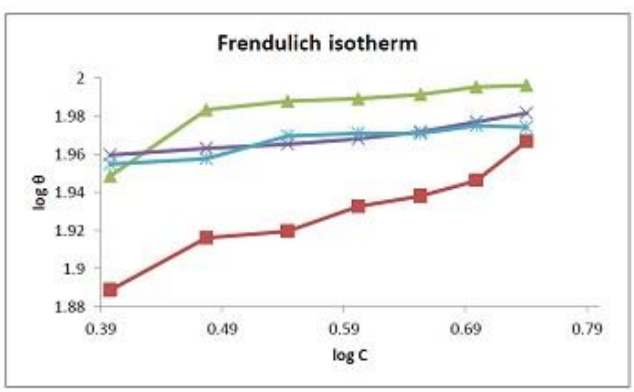


Figure 6 Freundlich adsorption isotherm for the dissolution of mild steel in 1M Hydrogen acid in the absence and in the presence of various concentration of GP extract.

Table 3 Linear regression analysis of Freundlich adsorption isotherm for the dissolution of mild steel in 1 M Hydrochloric acid in the presence of GP extract at different temperature.

Extract	Temperature in K	Slope	Intercept	Correlation coefficient
GP	303	2.5	9.2	5.8
	313	2.3	8.4	5.3
	323	2.4	8.3	5.4
	333	2.5	8.5	5.5
	343	2.1	7.2	4.7

Temkin adsorption isotherm

The plots of θ vs log C for the mild steel dissolution in 1 M Hydrochloric acid in the presence of GP extract are represented in the **Figure 7**. The applicability of Temkin isotherm verifies the assumption of monolayer adsorption on a uniform homogeneous metal surface with an interaction in the adsorption layer.

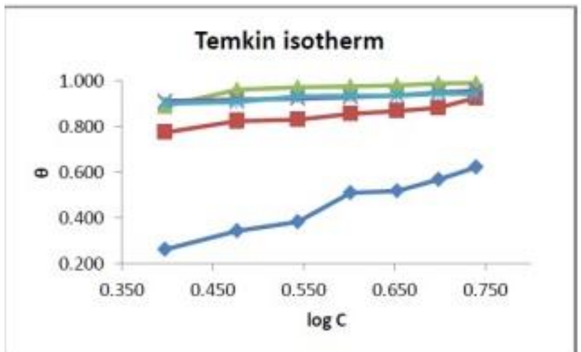


Figure 7 Temkin adsorption isotherm for the mild steel dissolution in 1M Hydrochloric acid in the presence of various concentrations of GP extract at different temperatures.

Thermodynamic functions

Thermodynamic functions for the dissolution of mild steel in 1 M Hydrochloric acid in the absence the presence of various concentrations of the inhibitor were obtained by applying the Arrhenius equation and transition state equation are represented in the **Table 4**.

Table 4 Thermodynamic functions for the dissolution of mild steel in 1M Hydrochloric acid in the absence and in the presence of various concentrations of GP extract at various temperature.

Conc.of the extract (%v/v)	-ΔGads $kJmol^{-1}$				
	303K	**313K**	**323K**	**333K**	**343K**
Blank	13.17	15.21	15.61	16.65	16.84
2.5	12.21	13.42	13.75	14.17	14.92
3	12.38	12.38	12.74	13.09	13.81
3.5	10.88	12.65	12.03	12.38	12.99
4	10.39	12.09	12.45	11.80	12.37
4.5	9.95	10.63	10.97	12.33	11.85
5	9.59	10.27	10.61	10.93	11.41
5.5	9.28	9.94	10.27	10.57	11.05
6	9	9.65	9.96	10.26	10.73

Arrhenius plot

The dependence of logarithm of the corrosion rate on the reciprocal of temperature for Hydrochloric acid are presented in the **Figure 8**.

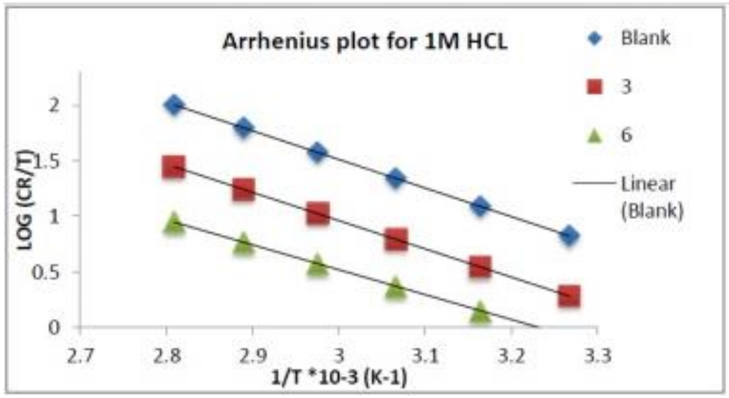


Figure 8 Arrhenius plot for the mild steel dissolution in 1M Hydrochloric acid in the absence and in the presence of various concentrations of GP extract at different temperatures.

Activation parameters

The activation energy E_a, Entropy of adsorption ΔS_{ads} and Enthalpy of adsorption ΔH_{ads} in the case of pure 1 M Hydrochloric acid in the presence of different concentrations of GP extract are shown in the **Table 5**. The adsorption of the phytoconstituents of the extract on the surface of the mild steel is conformed from the negative values of E_a. The increase in the activation energy in the presence of the extract when compared to that in the absence is suggestive of formation of adsorption film of physical nature [14]. The negative enthalpy change indicates that, the adsorption process is exothermic in nature [15] and the values ranges between 31 J/K/mol – 48 J/K/mol for 1 M Hydrochloric acid.

Table 5 Activation parameters for the dissolution of mild steel in 1M Hydrochloric acid in the absence and the presence of various concentration of GP extract.

Conc.of the extract (v/v)	$-E_a$ kJ/mol	ΔS_{ads} J/K/mol	$-\Delta H_{ads}$ kJ/mol
3	40.0	35.1	45.5
3.5	41.5	39.5	46.3
4	42.2	34.5	48.6
4.5	41.2	35.4	40.7
5	43	38.7	43.2
5.5	41.9	34.8	72.5
6	42.1	32.7	48.5

$\Delta G = 2.303\ RT\ (1.74 + \log(\theta/1-\theta) - \log C)$, the ΔG for corrosion adsorption was calculated using the above formula and the values are given in the Table 5.

The table reveals that ΔG values are negative and less than the threshold values of -40 KJ/mol required for chemical adsorption. The adsorption of the acid extracts of GP extract on the surface of mild steel is spontaneous and supports the mechanism of physical adsorption [16]. ΔS_{ads} values do not shown any gradual increase or decrease with respect to inhibitor concentration. This may be attributed to the fact that adsorption of the phytoconstituents is not only depended on the concentration but it also influenced by other factors like presence of inhibitor constituents among themselves as well as with the other constituents present in the corrosive media, orientation of the constituents etc.

Electrochemical studies

The polarization curves for the dissolution of mild steel in 1 M Hydrochloric acid in the absence and presence of GP extract are shown in the **Figure 9**. The values of corrosion kinetic parameters corrosion current (E_{corr}), Tafel slope constants, linear polarization resistance (R_p) are recorded in **Table 6**. It is noted from the Table 6, the values of I_{Corr} decrease with increase in the concentration of GP extract in 1 M Hydrochloric acid. The decrease in I_{Corr} with increase in extract concentration is associated with a shift in corrosion potential E_{Corr} to less negative values. The addition of the extract prevents acid attack on the mild steel suggesting that the extract act as a mixed type inhibitor. From Table 6, it is also clear that the Rp values are higher for inhibited system indicating the formation of an insulated adsorption layer. They inhibit corrosion by blocking the active sites of mild steel.

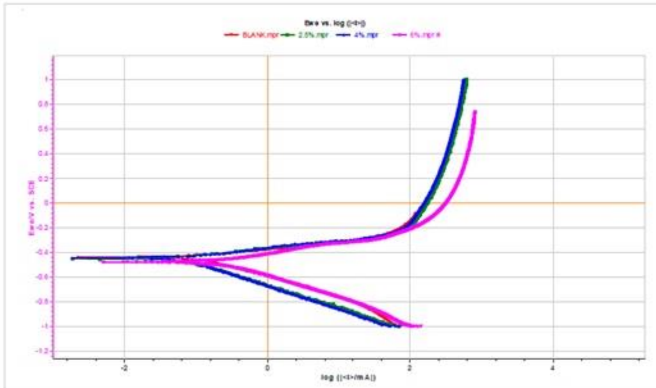


Figure 9 Polarization study for mild steel in 1M Hydrochloric acid.

Table 6 Electrochemical kinetic parameters for the dissolution of mild steel in 1M Hydrochloric acid in the absence and in the presence of various concentrations of GP extract.

Conc. of extract (% V/V)	$-E_{Corr}$ (mV)	I_{Corr} (μA)	R_p (Ohm)	b_a	b_c	IE % I_{Corr} Tafel	IE % R_p Linear
Blank	474.3	152.4	149	84	185		
2.5	442.5	56.6	310	59	181	62.8	51.9
4	440.2	55.4	345	60	183	63.6	56.8
6	433	52.4	352	71	175	65.6	57.6

The Impedance parameters derived from Nyquist plot (**Figure 10**) are tabulated (**Table 7**). Table 7 shows the results of the impedance studies of mild steel immersed in 1 M Hydrochloric acid with and without the plant extract. From the table, it is evident that the Rct values increases with inhibitor concentration implying reduced corrosion rate. Thus it is confirmed that the extract shows good inhibition efficiency.

Table 7 Impedance parameters for mild steel in the absence and presence of GP extract in 1M Hydrochloric acid.

Conc.	Rct ohm cm^2	IE
Blank	155	
2.5	183	16.3
4	280	44.6
6	267	41.9

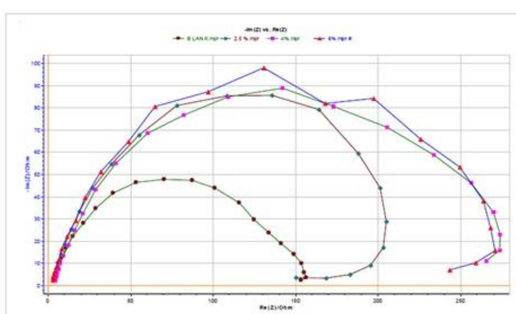
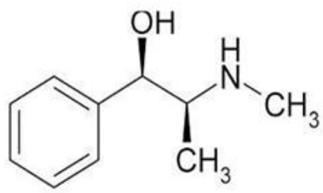


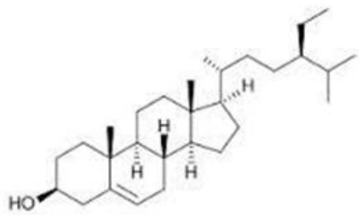
Figure 10 Impedance study of mild steel sample in 1M Hydrochloric acid.

Probable mechanism of inhibition

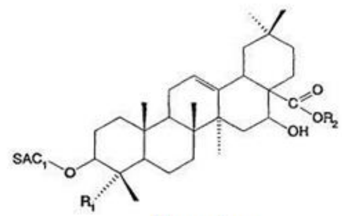
The plant extract investigated in this study is found to contain several organic compounds such as alkaloids, flavanoids, saponins, tannins, phytosterol, Galinsosides. The results of phytochemical analysis were summarized in **Table 8.** These big organic molecules in the plant extract may be adsorbed on the metal surface through their inhibiting the corrosion of mild steel. The adsorption process is proved from adsorption isotherm. Oxygen, Nitrogen, Sulphur atoms and hydroxyl group present in the constituents of the extract, adsorption of the constituents on the mild steel surface is depicted as follows,



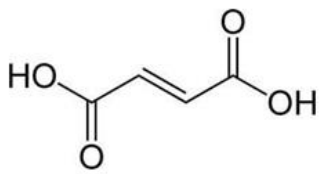
Alkaloids



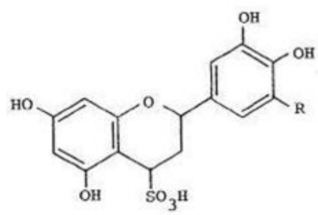
Phytosterol



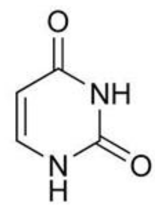
Saponins



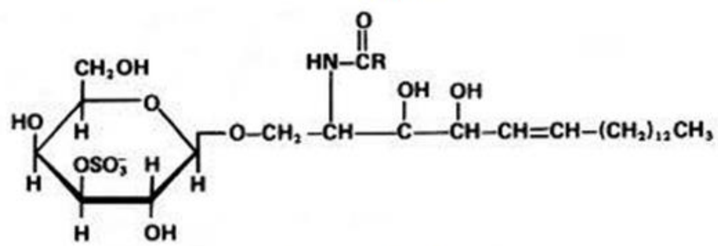
Fumaric acid



Tannins



Uracil



Galinsosides A

Table 8 Phytochemical analysis.

S.no	Experiment	Observation	Inference
1	**Carbohydrates:** To the test solution added small amount of Anthrone few drops of concentrated sulphuric acid and heated.	A black precipitate was obtained.	Confirmed the presence of Carbohydrate
2	**Alkaloids:** To the test solution added 2ml of dilute hydrochloric acid, shaken and decants the aqueous layer and added few drops of Wagner reagent.	A red precipitate was obtained.	Confirmed the presence of Alkaloids.
3	**Flavanoids:** To the test solution in alcohol added small amount of Mg powder, 3 drops of 2N concentrated hydrochloric acid and heated and cooled.	A Yellow precipitate was obtained.	Confirmed the presence of Flavanoids.
4	**Saponins:** To the test solution added water and shaken it well.	A foamy layer was formed for 1cm.	Confirmed the presence of Saponins
5	**Tannins:** To the test solution added water and lead acetate.	A white precipitate was obtained.	Confirmed the presence of Tannins.
6	**Phytosterol:** To the test solution in chloroform and add conc. Sulphuric acid	A golden yellow layer is formed below.	Confirmed the presence of Phytosterol.

Summary and Conclusion

- The plant material selected for the study GP provide as effective inhibitor for corrosion of mild steel in 1 M Hydrochloric acid.
- The efficiency of the inhibitor was found to increase with increase in concentration of the inhibitor and the inhibition efficiency of above 90% was obtained at 6% V/V of the inhibitor.
- The efficiency of the inhibitor increase with increase in immersion period. The optimum immersion period for GP was 48 h. This shows the persistence of the adsorption inhibitor molecules on the mild steel surface over a long period. The inhibitors were effective at the studied range of temperature. There was a trend of increase in efficiency with increase in temperature in higher concentrations of the inhibitors. GP shows maximum efficiency at 333 K in 1M Hydrochloric acid. The energy of activation E_a were negative for the inhibitor in both the acid media suggesting the formation of adsorption film of physical nature.
- Enthalpy of adsorption ΔH_{ads} was negative for the inhibitor in HCl confirming adsorption as an exothermic process.
- The negative values of free energy of adsorption ΔG_{ads} and positive values of entropy of adsorption ΔS_{ads} in all the cases confirm the spontaneous adsorption of the phytochemical constituents of the extract on the mild steel surface and the rate of adsorption is controlled by activation complex.
- Polarization studies suggest mixed type of inhibitor.
- The phytochemical constituents in the extract may be adsorbed on the metal surface and inhibit the corrosion process.

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