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Protection of mild steel in 3 M HCl solution by Areca root extract: Development of novel green and sustainable corrosion inhibitor

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ABSTRACT

The corrosion inhibition effect of Areca root extract on the mild steel (MS) surface in the 3 M HCl solution was examined by gasometric, colorimetric, Tafel plot, impedance, and atomic absorption spectroscopy techniques. Gasometric studies show that the extract behaves as a green corrosion inhibitor in the 3 M HCl system on the MS surface. The protection rate enhances with a rise in the plant extract concentration. Colorimetry studies show that minimum weight loss observed at 0.4 g/L of plant extract. The minimum weight loss of MS in the 3 M HCl solution is an indication of the protection property of Areca root extract. Atomic absorption spectroscopy technique shows that increase in the amount of Areca root extract increases the protection inhibition property. Mixed corrosion inhibition property of Areca root extract was confirmed from the potentiodynamic polarization technique. The trend of charge transfer resistance values with different amounts of plant extract also supports the corrosion inhibition property of Areca root extract. SEM studies fully support the gasometric, colorimetric, Tafel plot, impedance, and atomic absorption spectroscopy techniques.

Keywords: *Areca Root Extract, Mild Steel, Gasometric, Colorimetry, Atomic Absorption Spectroscopy*

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INTRODUCTION

Hydrochloric acid solutions generally used in the industries for chemical pickling, cleaning, petrochemical, and oil well acidizing process. MS metals are greatly corroded during several industrial operations. Corrosion of MS affects the growth of industries, which causes huge economic losses. Hence, discovering the new methods for MS corrosion control has become a hot topic of the present research. Many corrosion prevention methods existed such as electroplating, design modifications, hot-dip galvanization, passivation, cathodic protection, anodic protection, coatings, paintings, lubrication, and corrosion inhibitors. Among these, the corrosion inhibitor is an attractive method for corrosion prevention. Corrosion inhibitors are the chemical species, which generally added in the small concentrations (g/L or mg/L or ppm) to the corrosive solutions to prevent the MS dissolution or disintegration process [1-5]. The utilization of organic compounds has been planned as a solution to the MS corrosion problem. The organic compounds containing the nitrogen, sulfur, oxygen and phosphorous can dramatically reduce the mild steel corrosion rate. Majority of synthetic organic species is highly efficient in the hydrochloric systems for MS, but they are costly and causes ecological and health risks. Hence, because of environmental hazards of synthetic organic species, the corrosion scientist interest

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focused on the non-toxic and cheaper corrosion inhibitors [6-10]. In this view, effective green corrosion inhibitors obtained from the different types of plant products (root, leaves, flower, fruits, and stem) are attracting_high attention for the researchers. Green chemicals present in the plant extract can be obtained by simple procedures at a low cost. Plant extracts generally inhibit the MS corrosion process by adsorption process [11-16]. Authors, Alexander I. Ikeuba and Peter C. Okafor studied corrosion inhibition effect of saponins extract of Gongronema latifolium and ethanolic extracts of leaves of G. latifolium (EEGL) on the mild steel surface in the sulfuric acid system with the aid of hydrogen evolution technique in the solution temperature range of 30-60 °C. Authors reported the protection efficiency of saponins extract of Gongronema latifolium is 96.5 % and ethanolic extracts of leaves of G. latifolium are 93.7 % [17].

The corrosion protection property of methanolic almond peel extract and aqueous almond peel extract of mild steel in the hydrochloric acid system was reported by Hassane Lgaz, Preeti Tiwari, Ill-Min Chung, Gopal Ji, and Rajiv Prakash in 2019. The study revealed that methanolic extract exhibits more corrosion protection properties other than the aqueous extract. The results are further confirmed by atomic force microscopy technique [18]. Authors Zahra Sanaeia et al., in 2019 studied the corrosion inhibition property of Rosa canina fruit extract in 1 M hydrochloric acid system by using potentiodynamic polarization and AC impedance spectroscopy technique. Authors reported maximum protection efficiency of Rosa canina fruit extract is 86 % at 800 ppm of corrosion inhibitor. The corrosion inhibition property of Rosa canina fruit extract enhanced water contact angle [19]. The schematic representation of adsorption of plant extracts species on the metal (MS) surface as shown in Fig 1.

Adsorptive attractions between the MS and adsorbate (plant extract species) explain the corrosion inhibition action of plant extract species. Hence, in the present investigation selected Areca root extract. The Areca root extract contains groups such arecoline, guvacine, arecolidine, procyanidin B1, isoguvacine, arecaidine, gallic acid, guvacoline, epicatechin, catechin, leucocyanidin, rutin, lignin, ursolic acid and 3b acetyl ursolic acid [20-23]. These groups are electron rich centers, which are expected to be formed adsorption film on the MS surface in the 3 M HCl environment. The experimental methods used for MS corrosion inhibition test are gasometric, colorimetric, Tafel plot, impedance, and atomic absorption spectroscopy techniques. Surface studies were carried out by scanning electron microscopy (SEM) technique.

MATERIALS AND METHODS

Preparation of materials

99.03 % of MS metal was polished with different grade of sandpapers (500-2000). MS sample was washed with double distilled water and dried with acetone. Freshly collected Areca root was dried in the sunlight for 10 days. The extract of Areca root (AR) was prepared by the Soxhlet extraction technique by placing 250 g of Areca root powder in the 350 ml of double distilled water for about 7 hours. After evaporation of water, AR extract is stored in the desiccator. The Areca root extract amount used in the present investigation is 0.1 g/L, 0.2 g/L, 0.3 g/L, and 0.4 g/L. The corrosive solution in the present investigation is 3 M HCl, which is prepared according to the standard procedure. Functional groups present in the Areca root extract were examined through a Fourier transform-Infrared spectroscopy (FT-IR) technique. FT-

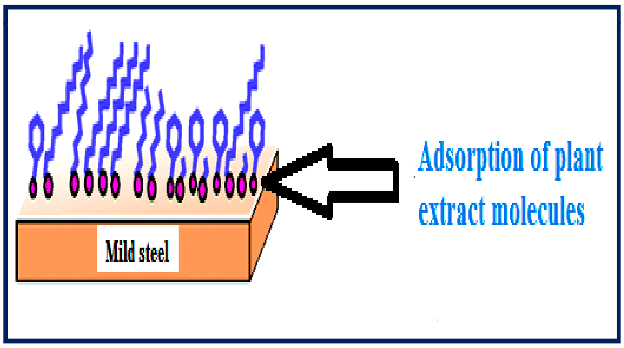


Fig. 1. Schematic representation of adsorption of plant extracts species on the mild steel surface

IR spectra of solid of Areca root extract, which is generated on the MS surface in the 3 M HCl solution after 24 hours was measured in between the 4000- 400 cm^{-1}. Ultraviolet-visible absorption spectroscopy observation was carried out for MS in the 3 M HCl solution in the absence and presence of the Areca root extract for the MS metal.

Gasometric studies

Gasometric technique gives rapid and consistent means of assessing the corrosive inhibiting performance of MS in the 3 M HCl solution as a function of the immersion period. In the gasometric technique, MS corrosion rate in the 3 M HCl solution was characterized by the evolution of hydrogen gas. The evolution of hydrogen gas is due to the disintegration process. The MS metal was placed in the conical flask, which consisting of 3 M HCl solution and side-arm burette. The burette is filled with double distilled water. The conical flask placed in the thermostat. Gasometric studies were performed at 60 ^{o}C.

The protection efficiency can be calculated from the following equation,

$$\text{Inhibition efficiency} = \frac{V_a - V_p}{V_a},$$

where, V_a= Amount of H_2 gas liberated in the uninhibited system, and V_p= Amount of H_2 gas liberated in the inhibited system.

Colorimetry studies

Colorimetry experiment was performed on the MS surface in the 3 M HCl solution without and with 0.1 g/L, 0.2 g/L, 0.3 g/L and 0.4 g/L of Areca root extract. The amount of MS in 3 M HCl solution and absorbance was noted by a colorimeter.

The protection efficiency was calculated from the equation below,

$$\text{Protection efficiency} = \frac{(W_1 - W_2)}{W_1} \times 100,$$

Where, W_1= Weight loss of MS without inhibitor, and W_2= Weight loss of MS with inhibitor.

Atomic absorption spectroscopy

Atomic absorption spectroscopy was used to calculate the amount of dissolved MS in the 3 M HCl solution. The inhibition efficiency can be calculated from the following relation,

$$\text{Inhibition efficiency} = \frac{B - A}{B} \times 100,$$

Where B= Amount of dissolved MS in 3 M HCl solution without inhibitor and amount of dissolved MS in 3 M HCl solution with inhibitor.

Electrochemical studies

The electrochemical techniques such as Tafel plot and impedance spectroscopy were used to study the corrosion inhibition property of Areca root extract. Current-potential curves were recorded with three-electrode cell (Pt= counter electrode, a saturated calomel= reference electrode, and mild steel= working electrode). Tafel plots were carried out with ± 200 mV at a scan rate of 0.01 Vs^{-1}. Impedance studies were performed in the frequency range of 10^{5} – 1 Hz.

Scanning electron microscopy

Surface studies were carried out by scanning electron microscopy (SEM) technique for MS surface without and with inhibitor for an immersion period of one hour.

FT-IR and UV-Visible spectroscopy

FT-IR spectroscopy carried out on the Areca root extract and protective film of Areca root extract on the mild steel in the 3 M HCl solution (with an immersion period of 3 hours) in the wavelength range of 4000 cm^{-1} to 1000 cm^{-1}. UV-Visible spectroscopy carried out in order to confirm the adsorption property of Areca root extract molecules on the mild steel surface in 3 M HCl solution without and with 0.4 g/L of Areca root extract with an immersion period of 3 hours at ambient solution temperature.

RESULTS AND DISCUSSION

Gasometric studies

The gasometric studies carried out at 60 ^{o} C and results of gasometric studies are presented in Fig 2 and Table 1. From the Fig. 2 and Table 1, it is clear that amount of hydrogen gas evolution decreases with enhancement in the Areca root extract, which indicates the prevention of degradation of MS metal in the 3 M HCl solution. The gasometric results show that protection efficiency enhances with the rise in the Areca root extract in 3 M HCl solution and corrosion inhibition effect more pronounced at 0.4 g/L of Areca root extract. The presence of Areca root extract generally prevents the acceleration of hydrochloric acid ions. The slow motion of corrosive ions in the 3 M HCl solution decreases the evolution of hydrogen (H_2) gas, which gave rise

Table 1. Gasometric results

Concentration (g/L)	Immersion period in hours	Volume of hydrogen gas evolved in ml	Protection efficiency (in percentage)
Blank	3	25.0	
0.1		5.0	80.00
0.2		4.0	84.00
0.3		3.1	87.60
0.4		1.3	94.80
Blank	6	40.0	
0.1		7.0	82.50
0.2		6.1	84.75
0.3		5.0	87.50
0.4		3.0	92.50
Blank	9	60.0	
0.1		11.0	81.66
0.2		10.0	83.33
0.3		8.0	86.66
0.4		6.0	90.00
Blank	12	87.3	
0.1		15.0	82.81
0.2		11.0	87.39
0.3		10.0	88.54
0.4		8.0	90.83
Blank	24	150.8	
0.1		30.0	80.10
0.2		27.0	82.09
0.3		22.0	85.41
0.4		20.0	86.73

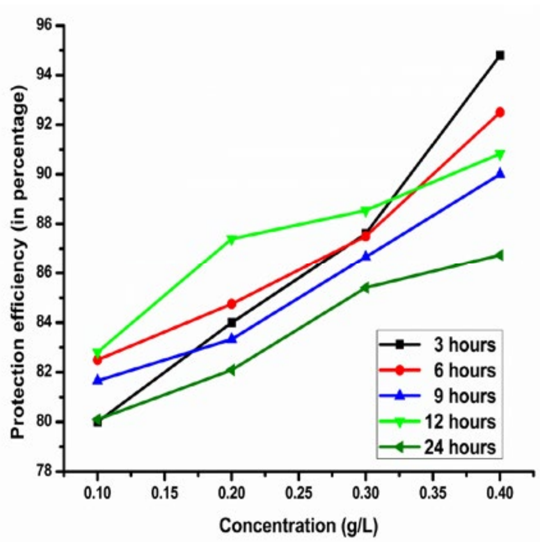


Fig. 2. Relationship between protection efficiency and concentration of the inhibitor at different immersion period

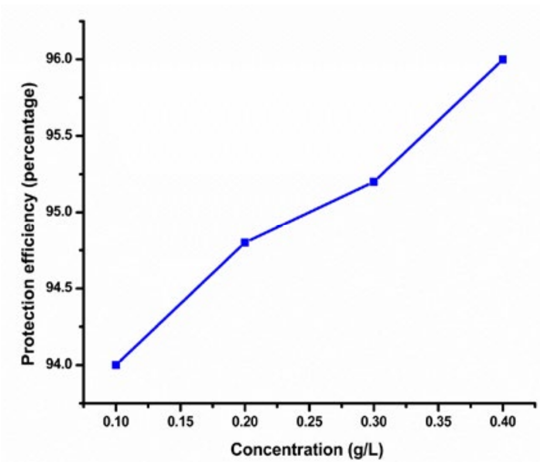


Fig. 3. Colorimetry results (Concentration vs. Protection efficiency)

to low disintegration of MS. The Areca root extract adsorbed on the MS surface in 3 M HCl solution and prevents the MS surface from the dissolution of metal. The maximum energy barrier for the MS corrosion in protected solution indicates that the adsorbed protective layer blocks the mass/charge transfer reaction taking on the surface of the MS. The high protection efficiency was observed at 3 hour immersion period at 60 ° C. The value of high protection efficiency obtained is 94. 8 %. The protection efficiency reduces with a rise in the contact time due to desorption process.

Colorimetry studies

The results of the colorimetry are shown in Fig. 3 and Table 2. Table 2 shows that, with an increase in the Areca root extract amount, the Fe content in the 3 M HCl solution decreases, which indicating the lower MS corrosion rate. This observation is due to adsorption of Areca root extract species on the surface of MS in 3 M HCl solution. The protection efficiency obtained from the colorimetry technique follows the same trend of results of gasometric studies. The low protection efficiency at the low amount of Areca root extract is due to unstable protective layer on metal surfaces. The stability of protective film or layer enhances with Areca root extract amounts and the most stable protective film formed at 0.4 g/L of Areca root extract. Therefore, maximum protection efficiency observed at 0.4 g/L of Areca root extract.

Atomic absorption spectroscopy

Atomic absorption measures the concentration of Fe^{+2} ions in the 3 M HCl solution when MS submerged in the corrosive solution with an immersion period of two hours. The disintegration of MS was measured by evaluating the dissolved Fe in the 3 M HCl solution without and with an inhibitor. The atomic absorption spectroscopy results are shown in Table 3. The increase in the protection efficiency of the corrosion inhibitor on the MS surface in 3 M HCl solution is due to adsorption of Areca root extract molecules [Fig. 4]. The formed invisible protective layer blocks the generation of corrosion products on the surface of MS in the 3 M HCl solution. The atomic absorption spectroscopy results are in good agreement with the colorimetry and gasometric results.

Tafel plot studies

The cathodic and anodic Tafel plots obtained from the potentiodynamic polarization studies are shown in Fig. 5. The various Tafel plot parameters are shown in Table 4. The protection efficiency of the Areca root extract can be obtained from the following equation

$$\text{Protection efficiency} = \left[1 - \frac{i'_{corr}}{i_{corr}}\right] \times 100$$

where, i'_{corr} = Mild steel corrosion current density

Table 2. Colorimetry studies

C (g/L)	Optical density	3 hour Weight of MS in grams	Protection efficiency
Bare	0.351	25×10^{-3}	
0.1	0.024	1.5×10^{-3}	94.00
0.2	0.022	1.3×10^{-3}	94.80
0.3	0.020	1.2×10^{-3}	95.20
0.4	0.017	1×10^{-3}	96.00

Table 3. AAS results

Concentration (g/L)	Amount of mild steel dissolved in 3 M HCl solution (g)	Protection efficiency (in percentage)
Blank	0.050	
0.1	0.010	80.00
0.2	0.005	90.00
0.3	0.004	92.00
0.4	0.003	93.60

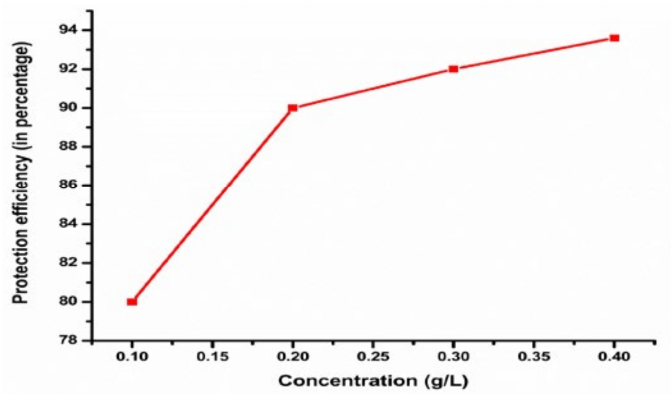


Fig. 4. Variations of protection efficiency at different amounts of plant extract

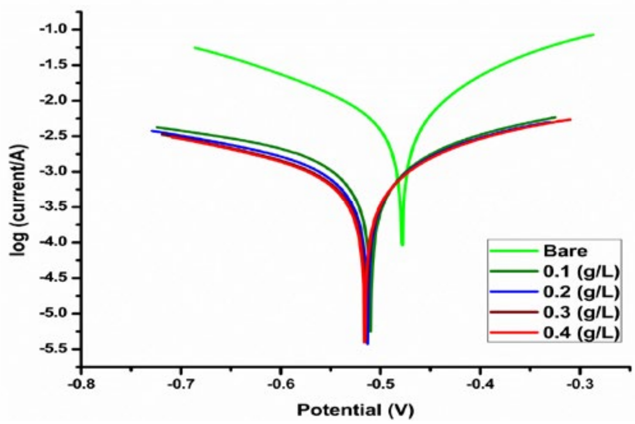


Fig. 5. Tafel plots

Table 4. Tafel results

Concentration (g/L)	Corrosion potential (mV)	Cathoidc Tafel slope (V/dec)	Anodic Tafel slope (V/dec)	Corrosion current (A)	Protection efficiency
Blank	-478	5.792	7.183	0.006398	
0.1	-510	3.852	5.278	0.001292	79.806
0.2	-513	4.016	5.340	0.001074	83.213
0.3	-515	4.091	5.496	0.0009563	85.053
0.4	-516	4.134	5.552	0.0009185	85.643

(protected) and i_{corr} = Mild steel corrosion current density (unprotected).

From Table 4, it is noticed that corrosion current density values decrease with a rise in the concentration of Areca root extract, which confirms the protection inhibition property of Areca root extract. The increase in the Areca root extract retards the MS corrosion process by adsorption process at active sites of MS surface. Further, it is noticed that the introduction of Areca root extract to the 3 M HCl solution causes no change in the corrosion potential (E_{corr}), cathodic (βc) and anodic Tafel plot (βa) values. According to literature, if a change in corrosion potential value exceeds 85 mV, then the green corrosion inhibitor is classified into cathodic, anodic or mixed type. In the present research, the Areca root extract is considered to be a mixed type. This is because maximum displacement in the corrosion potential value is 38 mV. The reduction in the corrosion current density values related to the adsorption of Areca root extract on the MS surface, which blocks the MS metal against the dissolution process. The maximum protection efficiency obtained from the potentiodynamic polarization technique is 85.643 %.

Impedance spectroscopy technique

Fig. 6 shows a Nyquist plot of MS in 3 M HCl solution without and with 0.1 g/L, 0.2 g/L, 0.3 g/L and 0.4 g/L of Areca root extract. The Nyquist plots are not perfectly semicircle which is ascribed to frequency dispersion, which is mainly due to the heterogeneity of the MS surface.

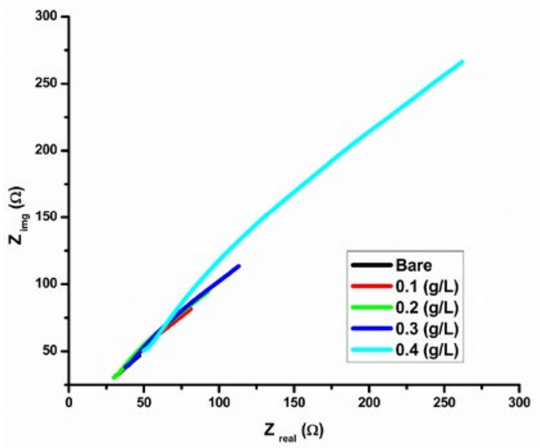


Fig. 6. Nyquist plots

The corrosion inhibition property of Areca root extract was calculated from the following equation,

$$\text{Protection efficiency} = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100$$

where, R_{ct} = Charge transfer resistance value (unprotected system) and $R_{ct(inh)}$ = Charge transfer

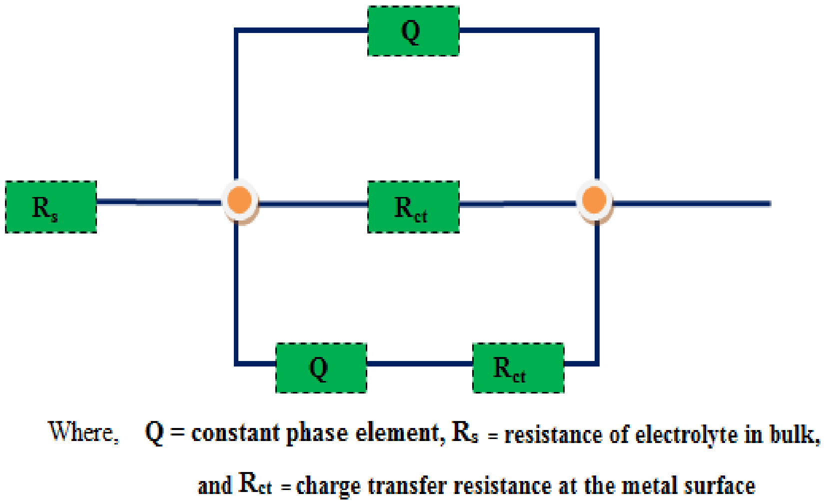


Fig. 7. Equivalent circuit of model R (QR (QR))

Table 5. Nyquist plot results

Concentration (g/L)	Charge transfer resistance (Ω)	Protection efficiency (%)
Blank	37.8	
0.1	71.24	46.939
0.2	90.35	58.162
0.3	97.58	61.262
0.4	262.5	85.600

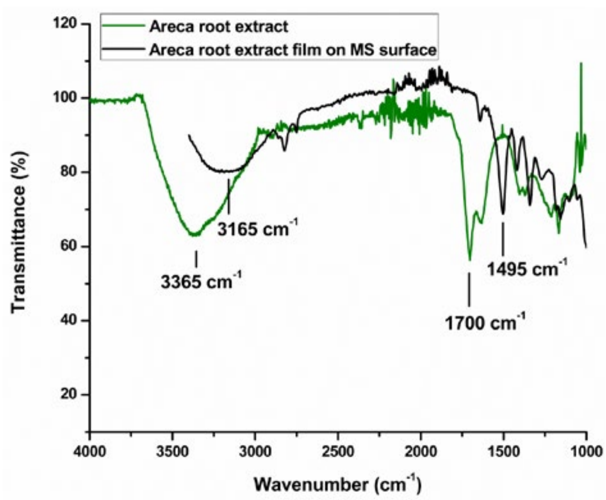


Fig. 8. FT-IR spectrum of Areca root extract and Areca root extract film on MS surface

resistance value (protected system).

The charge transfer resistance values are obtained from using the ZSimpWin 3.20 software. Among the many electrical circuits present in the ZSimpWin 3.20 software, the obtained Nyquist plots are well fit into the electrical equivalent circuit R (QR(QR)). The model of the R (QR (QR)) electrical equivalent circuit is shown in Fig. 7.

For the sake of comparison of MS corrosion rate, impedance studies were also carried out. The impedance results are shown in Table 5. According to this table, it is observed that the charge transfer resistance values enhance with the rise in the Areca root extract from 0.1 g/L to 0.4 g/L. The charge transfer resistance (R_{ct}) values are directly proportional to the Areca root extract concentration. The charge transfer resistance values enhance with a rise in the concentration of the Areca root extract and depressed semi-circle in the absence and presence of the inhibitor shows that the MS corrosion inhibition property is due to the charge transfer process. The maximum protection efficiency observed from the impedance studies is 85.600 %.

FT-IR spectroscopy techniques

FT-IR spectroscopy technique is a powerful tool which can be used to examine the type of bonding of green corrosion inhibitors which is adsorbed on the MS surface. During this investigation, the FT-IR spectroscopy technique is used to support the MS corrosion inhibition property of Areca root extract in the 3 M HCl solution. The IR bands of AR extract and protective film are shown in Fig. 8 (a, b). The shift of IR bands in the Areca root extract and a protective film (from 3365 cm^{-1} to 3165 cm^{-1}, 1700 cm^{-1} to 1495 cm^{-1}) confirms the adsorption of Areca root extract on the surface of MS in 3 M HCl solution.

UV-Visible spectroscopy

UV-Visible spectroscopy is one of the spectroscopic techniques and gives information about the adsorption of extracted organic molecules on the metal surface. UV-Visible absorption spectra obtained for a 3 M HCl solution containing 0.4 g/L of Areca root extract before and after 3 hour immersion of mild steel at room temperature are shown in Fig 9. The electronic absorption spectra of Areca root extract showed broad bands (200 nm and 250 nm) in the UV region. There was a small change in the values of absorbance and no significant change in the shape of the spectra before and after immersion of MS which suggested that Areca root extract is adsorbed on the MS surface and prevents from the corrosion.

Scanning electron microscopy technique

MS surface studies were carried out by scanning electron microscopy (SEM) technique. SEM photographs are shown in Fig. 10 a, b. Without Areca root extract, the MS surface becomes very rough, this is due to a direct attack of 3 M HCl solution on the MS surface. Whereas in the presence of corrosion inhibitor, the surface becomes very smooth, this is due to adsorption of Areca root extract on the MS surface in the 3 M HCl solution. The surface variation in the SEM photographs clearly shows the corrosion inhibition property of Areca root extract.

Mechanism

Studying the influence of Areca root extract for the corrosion of mild steel in 3 M HCl solution is very important. The corrosion protection mainly depends on the number of active sites, concentration, stability and molecular mass of the chemical substance. The special elements in the plant extract species such as P, S, N and O atoms give a tendency to reduce the or inhibit metal dissolution process. The Areca root extract can be used as a corrosion inhibitor due to a greater tendency of adsorption of Areca root extract molecules over the mild steel in the 3 M HCl

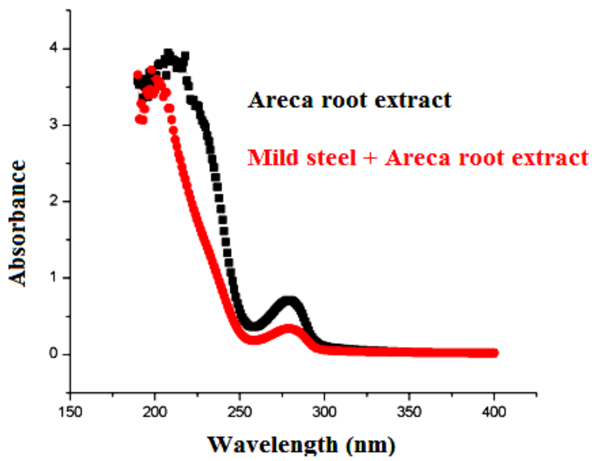
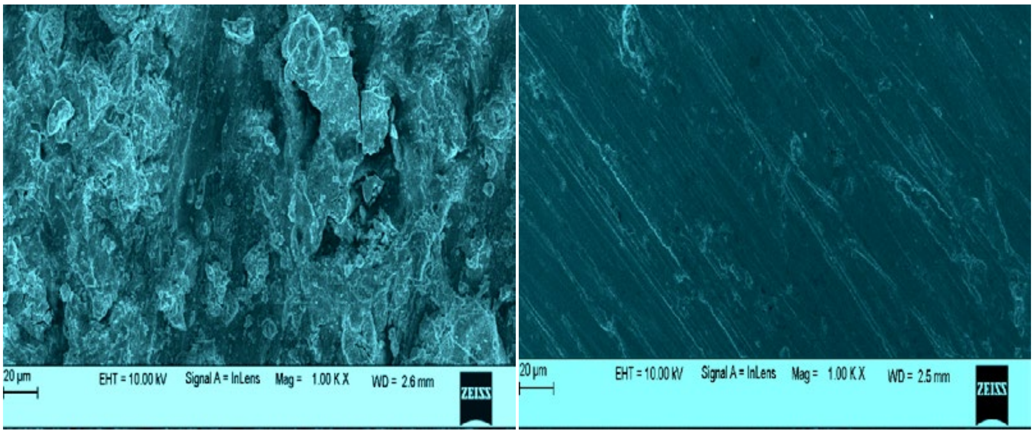


Fig. 9. UV-Visible spectroscopy results



a) b)

Fig. 10. (a, b). SEM photographs, a) without inhibitor, b) with inhibitor

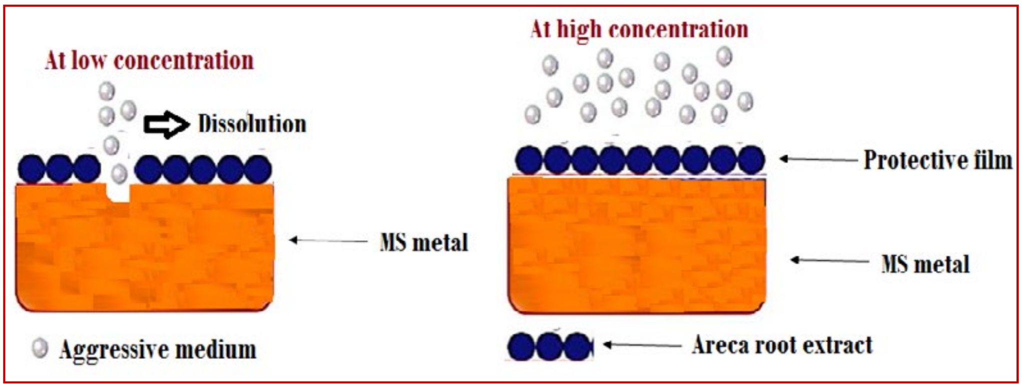


Fig. 11. Schematic representation of adsorption of Areca root extract species on the mild steel in 3 % HCl solution

solution. The existence of functional groups such as –OH, C=O and other groups in ursolic acid,

guvacine, procyanidin B1, arecolidine, arecaidine, arecoline, gallic acid, guvacoline, catechin, rutin, 3b acetyl ursolic acid, lignin, isoguvacine, leucocyanidin, and epicatechin of Areca root extract adsorbed on the MS surface in 3 M HCl system by blocking active site of MS and hinders the MS dissolution in high concentration of Areca root extract (0.4 g/L of Areca root extract) than lower (0.1 g/L of Areca root extract) as shown below [Fig. 11].

CONCLUSION

Results of different methods such as gasometric, colorimetry, atomic absorption spectroscopy, Tafel plot, and impedance spectroscopy technique shows that Areca root extract acted as good corrosion inhibitor for MS in 3 M HCl solution. The protection rate enhances with a rise in the concentration of the Areca root extract for all the five techniques. Tafel plot studies show the mixed corrosion inhibition property of Areca root extract. Impedance studies show that charge transfer process plays a very important role in the MS corrosion inhibition process. Scanning electron microscopy technique results also support the gasometric, colorimetry, atomic absorption spectroscopy, Tafel plot, and impedance results.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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