Anti-corrosion Properties of Areca Palm Leaf Extract on Aluminium in 0.5 M HCl Environment

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ABSTRACT

The aluminium corrosion inhibition behaviour in the presence of Areca palm leaves (AL) extract in hydrochloric acid (0.5 M) medium was examined by chemical (mass loss), AC impedance spectroscopy, potentiodynamic polarization and surface (scanning electron microscopy) methods. Aluminium protection rates increased with an increase in the amount of AL extract to the 0.5 M HCl system and decreased with increasing in the contact time of aluminium metal and temperature of 0.5 M HCl solution. Arrhenius equation was applied in the determination of activation energy values. The other activation parameters such as activation entropy and enthalpy values were obtained from the transition state plot. The adsorption of AL extract species on the Al surface in 0.5 M HCl solution follows the Langmuir adsorption mechanism. Tafel curves reflect the mixed (both anodic and cathodic) inhibition behaviour of green inhibitor (AL extract species) on electrode surface (Al) in 0.5 M system. Impedance method indicates that the Al dissolution in hydrochloric acid environment was fully hindered by charge transfer process. The Al surface morphology was examined by applying scanning electron microscopy (SEM) and atomic force microscopy (AFM) techniques.

KEYWORDS

Aluminium, Areca palm leaves, weight loss, Arrhenius equation, Langmuir adsorption.

1. Introduction

Hydrochloric acid solutions are widely utilized in oil well acidification, removal of scale and rust in metallurgy, acid pickling, de-scaling and cleaning of boilers in industrial processes. During this time, Al surface is greatly affected by corrosion.[1–4] To hinder the effect of hydrochloric acid solution on the electrode surface, corrosion researchers added inhibitors to the hydrochloric acid solution during the several industrial processes. Application of inhibitors as corrosion inhibitor is the simplest and viable technique to protect the Al surface in a hydrochloric acid environment. Al metal treatment with synthesized compounds has been planned to enhance the anticorrosion action.

In practice, copious synthesized species are being applied for the prevention and control of Al disintegration in hostile fluid systems. These synthesized species strongly defending the Al surface from the acid solution by forming the adsorption film on the metal surface *via* active centres existed in the species. The inhibition capacity mainly depends on the nature of synthesized species, nature corrosive environment and nature of the metal.

Several classes of inhibitors are toxic in nature and noxious to the both environment and living creatures. Hence, these specific reasons strongly hinder their utility as corrosion inhibitors in several industrial processes. Because of increased environmental risks and awareness of health, many scientists (especially corrosion researchers) have paid more attention towards the effective non-toxic inhibitors.[5–12]

Green species (natural products) are cheaper compared to synthetic compounds. The electron-rich species molecules in the plants were extracted by simple methods. Previously, many researchers reported the green corrosion inhibition property of natural extracts on Al metal in hydrochloric acid environment.[13–18]

At present, the majority of Areca palm waste products (including Areca leaves) is disposed by burning, which generally results in loss of valuable plant nutrients and potential source of organic matter. The Areca leaves are a good source of organic dung. The use of Areca waste products, especially Areca leaves (dried) in the field of corrosion science has not realized in the world. Areca leaves extract mainly contains electron rich species molecules such as lignin, cellulose, ursolic acid, 3β acetyl ursolic acid and hemicelluloses.[19,20] There is no report on potential corrosion inhibition property of Areca leaf extract for any central metal in any corrosive systems. Therefore, in the present case, we selected Areca leaf extract for the protection of Al surface from hydrochloric acid solution. The experimental methods such as gravimetric (mass loss), potentiodynamic polarization (Tafel curves) and impedance techniques are used to explain the Al corrosion inhibition mechanism. Further, scanning electron microscopy (SEM) and atomic force microscopy (AFM) tool was employed for the Al surface study in uninhibited (without Areca leaf extract) and inhibited (with Areca leaves extract) conditions.

2. Experimental Section

2.1. Material Preparation

Table 1 represents the chemical composition of Al-type.[21] The Al surface was polished with sandpaper in order to get smooth and rust free surface. Dust particles on the Al surface were removed by treating the Al surface with acetone. Finally, wiped aluminium metals are carefully stored.

2.2. Preparation of Green Inhibitor

1 g L^{-1}, 2 g L^{-1}, 4 g L^{-1} and 6 g L^{-1} of inhibitor concentration were prepared by extracting the 180 g of Areca leaves powder with 350 mL of acetone in Soxhlet extraction chamber for 7 h.

The presence of functional groups in the Areca leaf extract was examined by Fourier-transform infrared spectroscopy tool.

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Table 1 Chemical composition of Al specimens.

Element	Fe	Si	Tl	Zn	Mn	Cr	Mg	Cu	Al
wt%	0.6	0.3–0.7	0.1	0.2	0.3	0.2	0.4–0.9	0.1	Remainder (96.9–97.8 %)

2.3. Weight Loss Measurements

The cleaned and pre-weighed Al surface was suspended in uninhibited and inhibited systems. The loss in the weight of Al metal with respect to different immersion time (1, 2, 3, 4, 5 and 10 h) and different solution (0.5 M HCl) temperatures (303, 308, 313, 318 and 323 K) were noted. The experiment was repeated three times in order to confirm the truth of the results until the concordant value was obtained. The Al corrosion rate in mild penetration per year was calculated by[22],

$$\text{Corrosion rate} = \frac{534W}{ATD} \qquad (1)$$

where W = Al weight loss (mg), A = Al area (sq inch), T = Al immersion time (h) and D = Al density (g cm^{-3}).

The Areca leaves extract protection efficiency (inhibition capacity) was calculated by using the following equation,

$$\text{Protection efficiency} = \frac{W_1 - W_2}{W_1} \times 100 \qquad (2)$$

where W$_2$ = Al weight loss in protected state and W$_1$ = Al weight loss in unprotected state.

The Al surface topography after treatment with 0.5 M HCl and 0.5 M HCl plus Areca leaf extract were examined through SEM and AFM technique.

2.4. Electrochemical Measurements

The electrochemical tests (CHI660C work station) were carried out by three electrode configurations (Al = working electrode, pt = auxiliary electrode and calomel = reference electrode). For potentiodynamic polarization technique, potential of ±200 mv was applied with scan rate 0.01 V/s. Impedance study was carried out with the frequency of the range in between 10^5 to 1 Hz using 0.01 V amplitude. The electrochemical experiment was repeated and concordant values were reported.

3. Results and Discussion

3.1. Weight Loss Technique

The loss of Al weight sample in 0.5 M HCl system with and without various Areca leaves extract concentrations at different immersion period and solution temperature was evaluated. Tables 2 and 3 represent the results of mass loss (gravimetric

Table 2 Gravimetric parameters at room temperature (303 K).

Time /h	Concentration /g L^{-1}	Corrosion rate $\times 10^{-4}$ /mpy)	Inhibition efficiency
1	Blank	8.689 ± 0.36 %	
	1	2.458 ± 0.016 %	74.037 ± 0.022 %
	2	1.941 ± 0.023 %	77.147 ± 0.007 %
	4	1.448 ± 0.033 %	82.767 ± 0.0256 %
	6	0.892 ± 0.062 %	89.493 ± 0.0113 %
2	Blank	11.414 ± 0.0369 %	
	1	3.437 ± 0.074 %	73.150 ± 0.018 %
	2	2.651 ± 0.014 %	73.698 ± 0.023 %
	4	1.897 ± 0.019 %	83.138 ± 0.017 %
	6	1.450 ± 0.032 %	84.287 ± 0.0298 %
3	Blank	16.450 ± 0.021 %	
	1	4.578 ± 0.049 %	69.019 ± 0.020 %
	2	4.484 ± 0.028 %	71.187 ± 0.027 %
	4	3.458 ± 0.061 %	77.770 ± 0.0372 %
	6	2.465 ± 0.023 %	83.033 ± 0.0213 %
4	Blank	18.481 ± 0.017 %	
	1	6.197 ± 0.042 %	64.913 ± 0.0240 %
	2	5.524 ± 0.018 %	68.865 ± 0.016 %
	4	3.627 ± 0.008 %	82.785 ± 0.025 %
	6	3.356 ± 0.042 %	83.262 ± 0.010 %
5	Blank	24.433 ± 0.009 %	
	1	8.545 ± 0.015 %	63.672 ± 0.024 %
	2	7.349 ± 0.010 %	72.684 ± 0.030 %
	4	6.330 ± 0.033 %	71.904 ± 0.035 %
	6	4.623 ± 0.043 %	82.659 ± 0.026 %
10	Blank	42.431 ± 0.002 %	
	1	16.605 ± 0.016 %	58.734 ± 0.021 %
	2	13.202 ± 0.031 %	67.1 ± 0.014 %
	4	12.600 ± 0.013 %	72.942 ± 0.019 %
	6	9.539 ± 0.011 %	75.050 ± 0.027 %

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measurement) technique. The introduction of 1 g L⁻¹, 2 g L⁻¹, 4 g L⁻¹ and 6 g L⁻¹ of Areca leaves extract to the 0.5 M HCl solution greatly reduces the aluminium corrosion rate. This suggests that protection of Al metal occurs by adsorption of Areca leaves extract at active sites of Al surface. The minimum aluminium corrosion rate (maximum protection efficiency) was observed at 6 g L⁻¹ of Areca leaf extract. The number of species adsorbed on the Al surface was increased with increase in Areca leaf extract concentration. Hence, maximum protection was achieved at 6 g L⁻¹ of Areca leaf extract.

The increase in Al immersion time leads to increase in corrosion rate values (decrease in protection efficiency values), which is probably due to adsorbed Areca leaves extract species on the Al surface undergo degradation (desorption) and protective film loses its stability leaving the Al surface in unprotected condition. Hence, the greater Al surface is attacked by corrosive ions (hydrochloric acid). Therefore, high Al dissolution was observed with increasing the immersion time from one hour to ten hours. It is also observed that the Al corrosion rate in hydrochloric acid solution enhanced with an increase in temperature; this may be due to increase in desorption rate and decreased desorption rate of Areca leaves extract species on the surface of metal. The desorption process weakens the Al-Areca leaves extract interaction. As a result, protection efficiency decreases with increases in solution temperature.[23]

Activation energy (Ea) values can be identified from the Arrhenius plots of aluminium corrosion rate (Fig. 1). Transition state plot (Fig. 2) was used to determine the activation enthalpy (ΔH^*) and activation entropy (ΔS^*) values. The obtained activation parameters are placed in the Table 4.

The greater activation energy values in the protected system as opposed to unprotected system recommended that adsorbed inhibitor species on the Al surface leads to increase the energy barrier of electrode (Al) dissolution, resulting in the reduction of Al dissolution in 0.5 M HCl. Hence, Al surface was protected by Areca leaf extract species. The obtained positive ΔH^* values reflected the endothermic Al disintegration process in 0.5 M hydrochloric acid system.

The direction of ΔS^* values directed towards the positive side is an indication of increase of disordeness of the system.

The basic thermodynamic information, knowledge between

Table 3 Effect of 0.5 M solution temperature on the protection efficiency with contact time of one hour.

Temperature /K	Concentration /g L⁻¹	Protection efficiency in percentage
303	1	74.037 ± 0.022 %
	2	77.147 ± 0.007 %
	4	82.767 ± 0.0256 %
	6	89.493 ± 0.0113 %
308	1	71.900 ± 0.024 %
	2	75.611 ± 0.014 %
	4	81.104 ± 0.011 %
	6	82.772 ± 0.019 %
313	1	67.768 ± 0.014 %
	2	71.300 ± 0.0189 %
	4	75.718 ± 0.018 %
	6	80.894 ± 0.011 %
318	1	66.838 ± 0.0206
	2	70.338 ± 0.0217
	4	74.005 ± 0.013 %
	6	81.738 ± 0.019 %
323	1	65.005 ± 0.015 %
	2	71.341 ± 0.015 %
	4	70.640 ± 0.010 %
	6	77.258 ± 0.0141 %

Table 4 Activation parameters.

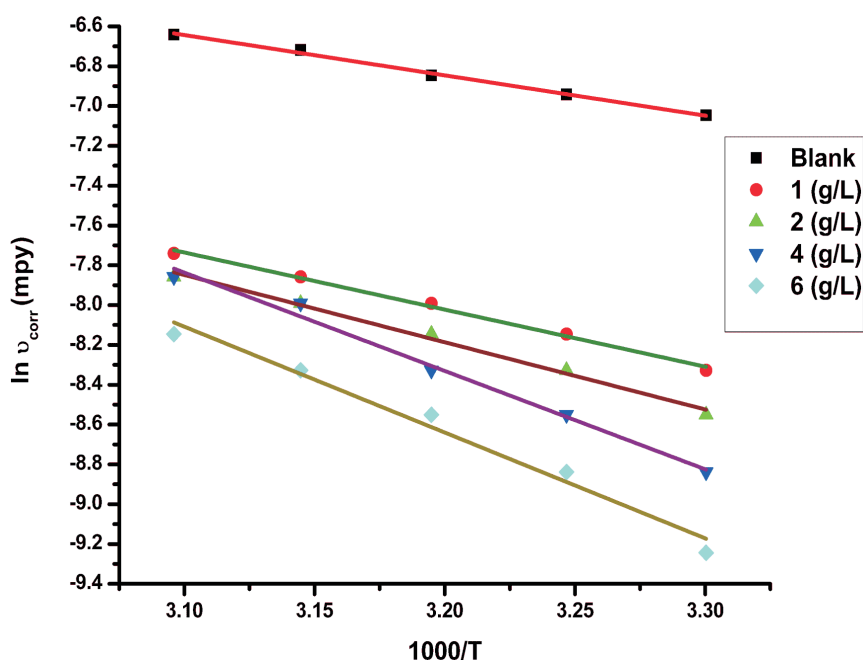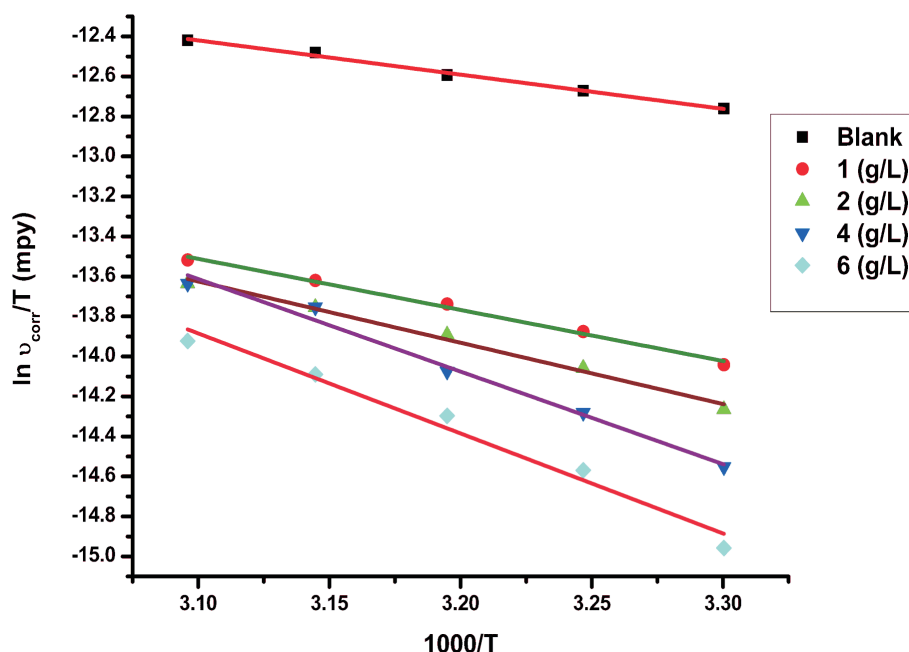| C /g L⁻¹	Ea* /kJ mol⁻¹	ΔH^* /kJ mol⁻¹	ΔS^* /J mol⁻¹ K⁻¹
Blank	16.830	14.230	–313.901
1	23.846	21.246	–301.232
2	28.084	25.483	–289.036
4	41.071	38.471	–248.672
6	44.194	41.593	–241.253

Fig. 1 Arrhenius plots.

Fig. 2 Transition state plots (without and with Areca leaf extract).

the Areca leaves extract and the Al surface can be studied by adsorption models. Surface coverage (θ) values obtained from the gravimetric method were used to study the different adsorption model. In the present investigation, the surface coverage and inhibitor concentration values are fitted well to Langmuir adsorption model (Fig. 3). The various thermodynamic parameters obtained from this plot are shown in Table 5.

The equilibrium constant of the inhibitor adsorption (K_{ads}) values increase with increasing solution temperature from 303 K to 323 K. The higher K_{ads} values clearly proved the strong adsorption of Areca leaves extract molecules on Al surface. The introduction of Areca leaves extracts to the 0.5 M HCl solution caused –ve free energy of adsorption (ΔG°_{ads}) values, which certified that chemical constituents of Areca leaves adsorb spontaneously on the Al surface in a hydrochloric acid environment. The ΔG°_{ads}

Table 5 Thermodynamic parameters.

Temperature /K	K_{ads} /L g^{-1}	ΔG°_{ads} /kJ mol^{-1}
303	2577.851	–37.195
308	2647.113	–37.877
313	2723.163	–38.565
318	2796.889	–39.252
323	2874.389	–39.943

values obtained with the system were close to –40 kJ/mol, indicating that adsorption of Areca leaves extract species on the surface of the Al is more chemical phenomena than the physical phenomena.
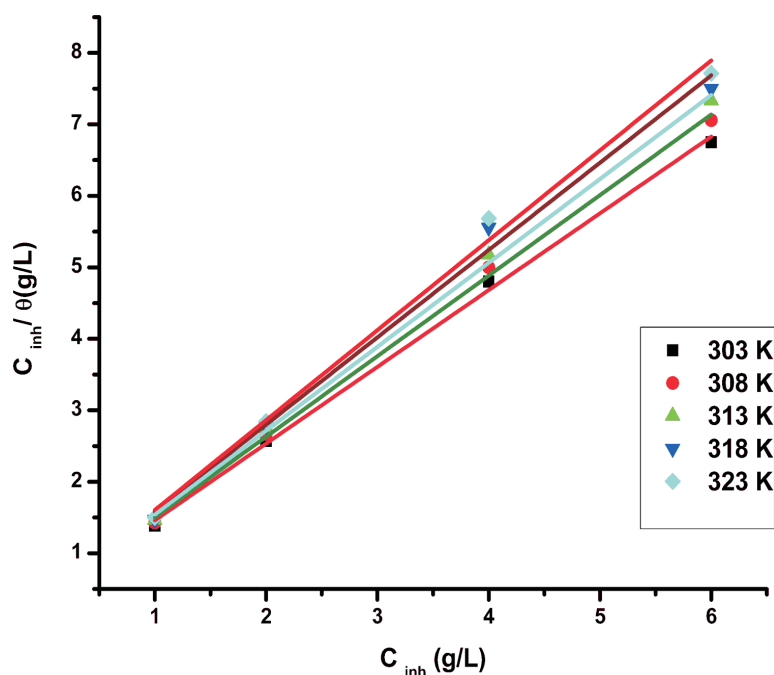

Fig. 3 Langmuir adsorption model.

3.2. Electrochemical Measurements

3.2.1. *Potentiodynamic Polarization (Tafel Plot)*

Figure 4 shows the Tafel curves[24,25] (cathodic and anodic) for unprotected and protected system after the stabilization period in the 0.5 M HCl condition. The anodic (βa) and cathodic (βc) Tafel constants, corrosion current density (i_{corr}) and corrosion potential (E_{corr}) values are obtained Tafel plots. The ability of an Areca leaf extract toward Al corrosion inhibition (protection efficiency) was evaluated by following relation,

$$\text{Protection efficiency} = 1 - \left[\frac{i'_{CORR}}{i_{CORR}} \right] \times 100 \qquad (3)$$

where i'_{corr} = Al corrosion current density value in 0.5 M HCl plus inhibitor condition and i_{corr} = Al corrosion current density value in bare condition.

All above data are presented in Table 6. The shape of potentiodynamic polarization curves (Tafel curves) is same in both unprotected and protected systems, but the plots are moves towards lower i_{corr} values in the protected system (with the addition of different concentrations of Areca leaf extract), which confirming that the species of Areca leaves extract retard the Al corrosion rate by adsorbing on electrode surfaces. Hence, protection efficiency increases with increase in Areca leaves concentration. The molecules of Areca leaves extract causes marginal changes in the corrosion potential, anodic and cathodic Tafel constant values with respect bare solution, implying that Areca leaves extract inhibit the both anodic and cathodic reactions (mixed inhibition property) of Al in 0.5 M HCl system through the adsorption mechanism.

3.2.2. *Impedance Spectroscopy Technique*

The Nyquist curves[26,27] for Al obtained without and with Areca leave extract concentrations in 0.5 M HCl solution at laboratory temperature are represented in Fig. 5. The obtained plots are not semicircle due to roughness and non-homogeneity of Al surface. The charge transfer resistance (R_{ct}), surface heterogeneity factor (n), frequency (f_{max}), chi-square (χ^2) values and double layer capacitance (C_{dl}) values are obtained from Nyquist plot and are presented in Table 7.

From the charge transfer resistance values, the corrosion inhibition efficiency was calculated according to the following equation,

$$\text{Corrosion inhibition efficiency} = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100 \quad (4)$$

where, R_{ct} = value of charge transfer resistance in inhibited free system, and $R_{ct(inh)}$ = value of charge transfer resistance in inhibiting system.

Quality of the fit was determined based on chi-square (χ^2) values. In present study, the obtained chi-square (χ^2) values are in between the range 0.001412–0.033260, which clearly indicates the proposed circuit is the best fit.

The data presented in Table 7 show that the value of R_{ct} in 1 g L^{-1}, 2 g L^{-1}, 4 g L^{-1} and 6 g L^{-1} of Areca leaves extract is high compared to bare (0.5 M) solution and maximum value is obtained at 6 g L^{-1} of Areca leaves extract is attributed to the existence of insulating adsorbed film at electrode (aluminium)–electrolyte (0.5 M hydrochloric acid) solution interface. Stability of insulating protective layer increases with improving the inhibitor concentration. Hence, maximum Al protection was
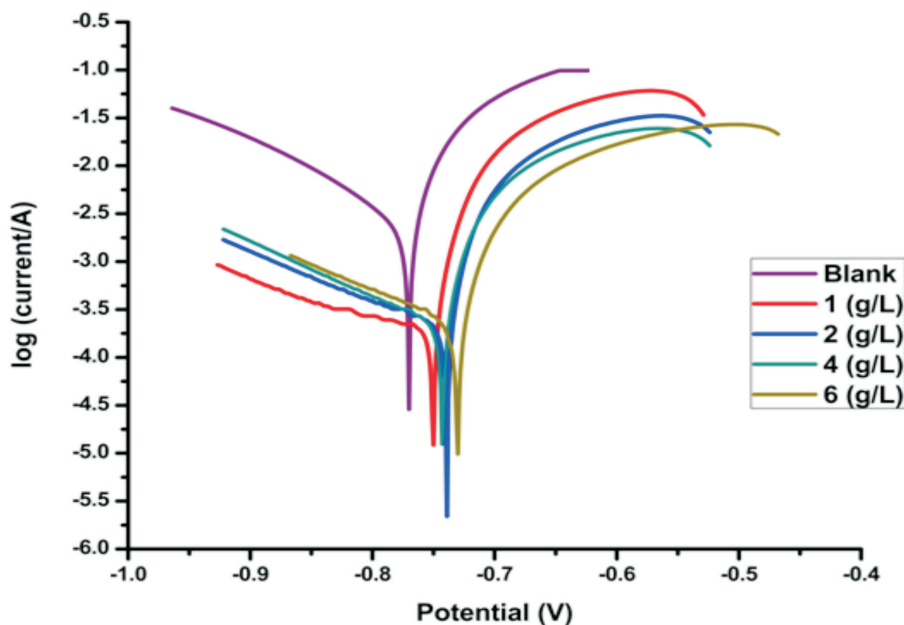

Fig. 4 Tafel plots without and with inhibitor.

Table 6 Potentiodynamic polarization (Tafel study) results.

Concentration /g L^{-1}	E_{corr} /mV	$i_{corr} \times 10^{-3}$ /A	βc /V dec^{-1}	βa /V dec^{-1}	Protection efficiency
Blank	−770	8.916	6.679	6.031	
1	−750	1.824	3.398	6.694	79.542
2	−739	1.338	5.050	6.332	84.993
4	−743	1.189	5.694	6.153	86.664
6	−730	1.057	5.175	6.640	88.114

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Table 7 Nyquist plot parameters.

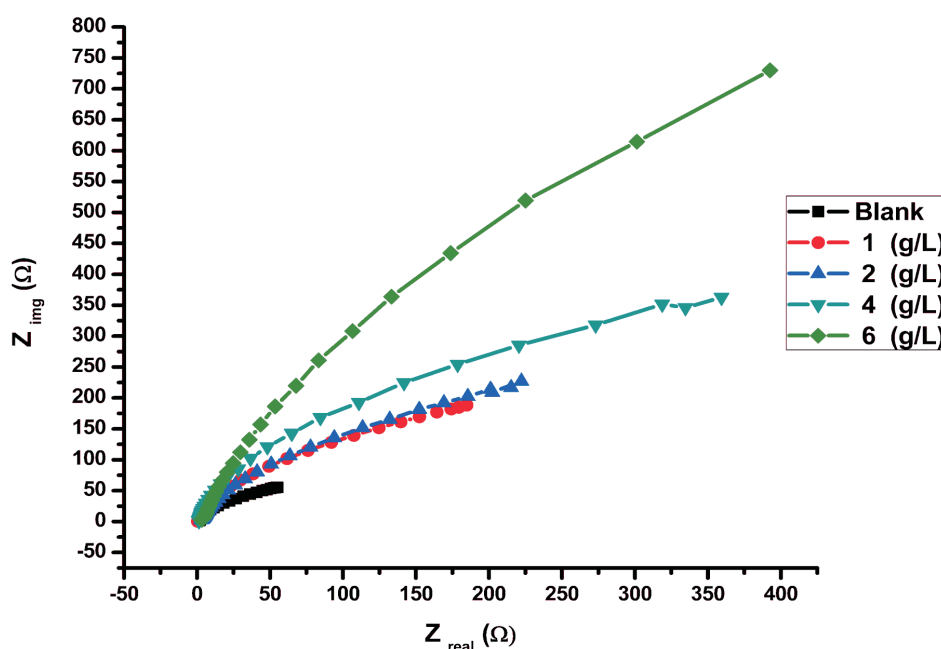| Concentration /g L^{-1}	n	R$_{ct}$ /Ω	C$_{dl}$/μF	χ^2	Protection efficiency
Blank	1	55.94	291.355	0.001479	
1	1	197.2	147.082	0.033260	71.632
2	0.9055	233.2	124.376	0.001712	76.012
4	0.9953	366.8	140.492	0.007258	84.749
6	0.8975	1887	71.513	0.001412	97.035

Fig. 5 Nyquist plots.

successfully achieved at highest Areca leaf extract concentration. The decrease in C$_{dl}$ value leads to enhance in double layer thickness, which shows that Areca leaves extract species retards the Al dissolution by adsorption at the Al/0.5 M HCl solution interface. The variation in the C$_{dl}$ values confirms the substitution of H$_2$O molecules by Areca leaf extract species. The obtained n values range 1–0.8975 showing that inhibition of Al corrosion in 0.5 M HCl solution was occurring through charge transfer phenomena.

3.3. FT-IR Spectroscopy

Functional groups in organic molecules play very important role in the metal corrosion inhibition process. FT-IR spectroscopy was used in order to investigate the functional groups present in the Areca leaves extract (Fig. 7). The obtained results are shown in Table 8.

3.4. Surface Study

3.4.1. *Scanning Electron Microscopy (SEM) Analysis*

Figure 8a,b shows the SEM images of Al after 2 hours immersion in 0.5 M HCl environment without and with Areca leaves extract condition, respectively. With no inhibitor (without Areca leaf extract), the Al surface was highly damaged due to direct attack by corrosive ion metal surface resulting in the large of cracks in the Al surface. With inhibitor (Areca leaves extract), the Al surface was significantly improved with smooth surface, indicating the reduction of the Al corrosion rate.

Table 8 FT-IR results.

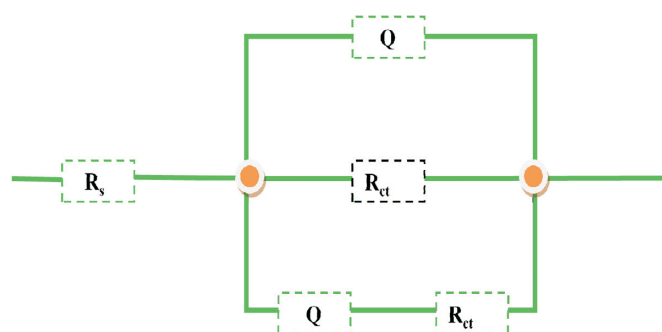| Bands	Groups
3402 cm^{-1}	monomeric hydroxyl
1710 cm^{-1}	carbonyl
2343 cm^{-1}	O-C=O
2920 cm^{-1}	C-H stretching vibrations
1086 cm^{-1}	C-O

Fig. 6 Electrical circuit (R (QR (QR)) model, where, R$_s$ = resistance of electrolyte in bulk, R$_{ct}$ = charge transfer resistance at the metal surface and Q = constant phase element.
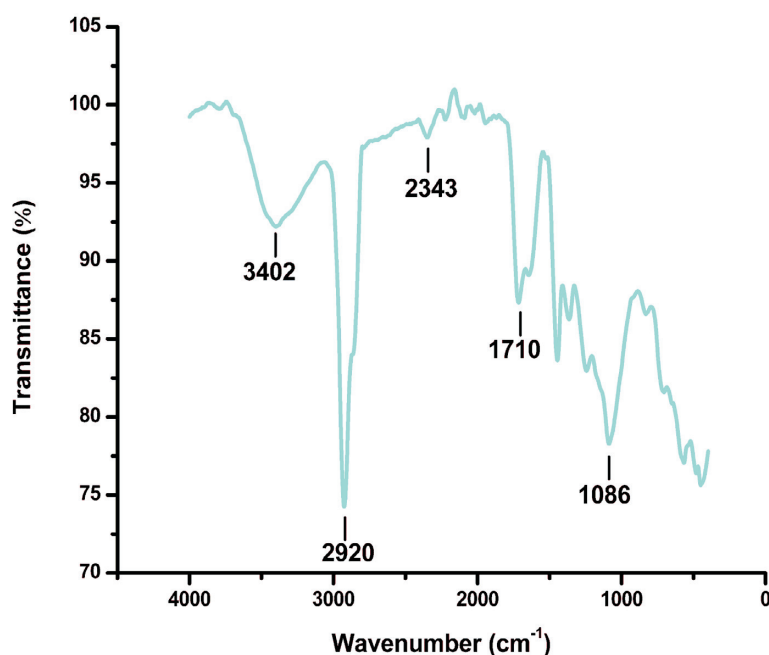
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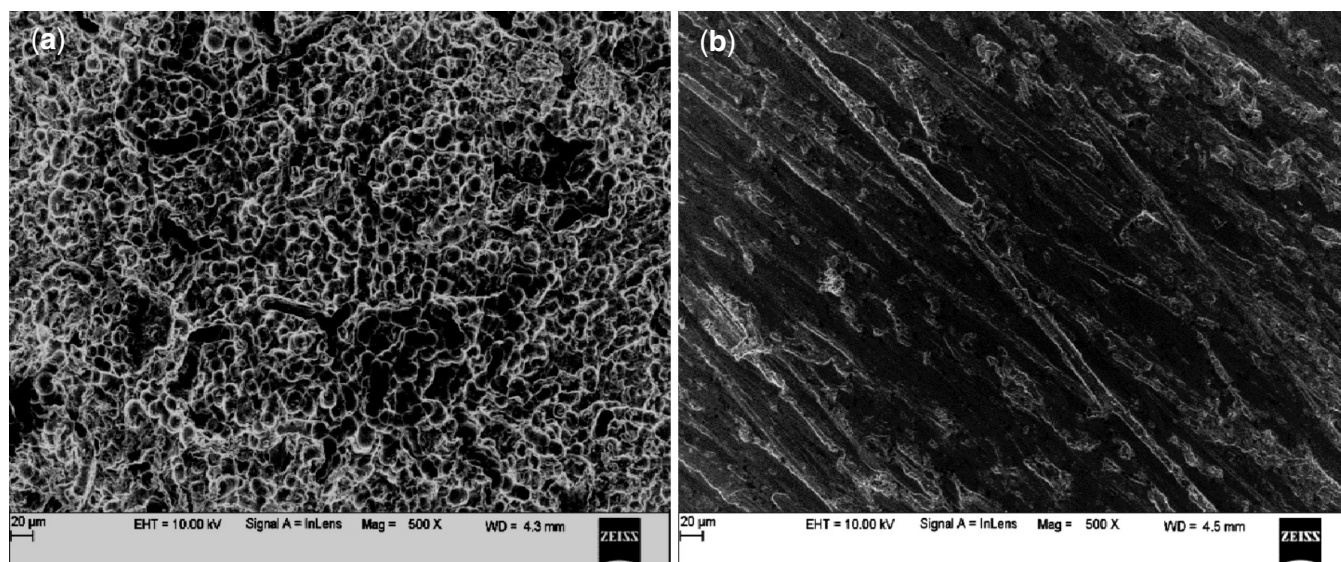**Fig. 7** FT-IR spectrum of Areca leaves extract.

Fig. 8 SEM images of aluminium; (**a**) without Areca leaf extract, (**b**) with Areca leaf extract.

Table 9 AFM results.

Concentration	Average roughness /S_a	Root mean square roughness /S_q
Bare	323.04 nm	395.01 nm
6 g L^{-1}	185.79 nm	275.78 nm

3.4.2. *Atomic Force Microscopy (AFM) Technique*

It is observed that the reduction in the value of average roughness (S_a) and root mean square roughness (Sq) in inhibited system (0.5 M HCl + plant extract species) compared to bare system is an indication of lesser aluminium corrosion rate in inhibited state. Hence, the atomic force microscopy results (Fig. 9 a,b and Table 9) are fully favourable with the results of SEM studies.

4. Conclusions

Areca leaves extract acts as potential corrosion inhibitor for aluminium in a 0.5 M HCl environment. Areca leaves extract has potential efficiency of 89.493 % obtained by gravimetric measurements, 88.114 % by potentiodynamic polarization test and 97.035 % by impedance spectroscopy technique. The positive ΔH^* values strongly reflect the endothermic Al dissolution process. The obtained negative ΔG°_{ads} values indicate the spontaneous adsorption process of Areca leaves extract on the Al surface in 0.5 M HCl system. The SEM and AFM topography greatly favours the results of weight loss, potentiodynamic polarization and impedance studies.
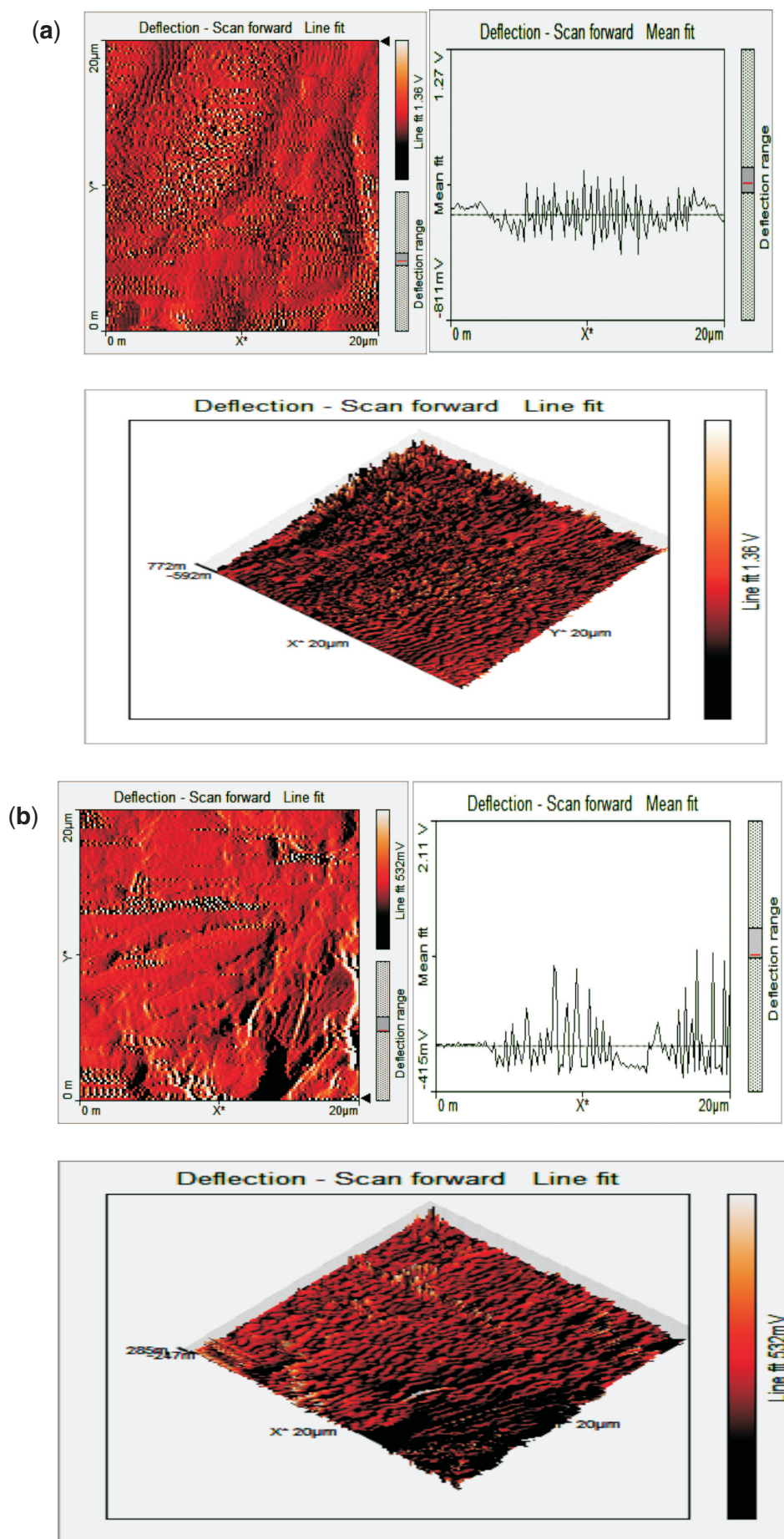
Fig. 9 (**a**) AFM image without plant extract; (**b**) AFM image with plant extract.

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Conflicts of Interest

The authors declare no conflict of interest.

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1. Arrhenius plot

	A	B	C	D	E1	E2
1	Equation	y = a + b*x				
2	Adj. R-Square	0.99508	0.99387	0.99078	0.98593	0.97331
3			Value	Standard Error		
4	B	Intercept	-0.36801	0.22737		
5	B	Slope	-2.0244	0.07111		
6	C	Intercept	1.15577	0.35974		
7	C	Slope	-2.86825	0.11251		
8	D	Intercept	2.62275	0.52047		
9	D	Slope	-3.37792	0.16278		
10	E	Intercept	7.47766	0.94192		
11	E	Slope	-4.9401	0.2946		
12	F	Intercept	8.36993	1.40251		
13	F	Slope	-5.31562	0.43865		

2. Transition state plot

	A	B	C	D	E1	E2
1	Equation	y = a + b*x				
2	Adj. R-Square	0.99313	0.99206	0.98858	0.9839	0.96972
3			Value	Standard Error		
4	B	Intercept	-7.11371	0.22744		
5	B	Slope	-1.71163	0.07113		
6	C	Intercept	-5.58993	0.36517		
7	C	Slope	-2.55549	0.11421		
8	D	Intercept	-4.12295	0.52589		
9	D	Slope	-3.06515	0.16448		
10	E	Intercept	0.73196	0.94432		
11	E	Slope	-4.62734	0.29535		
12	F	Intercept	1.62423	1.40788		
13	F	Slope	-5.00286	0.44033		

3. Langmuir plot

	A	B	C	D	E1	E2
1	Equation	y = a + b*x				
2	Adj. R-Square	0.99021	0.99123	0.99759	0.99787	0.99811
3			Value	Standard Error		
4	E	Intercept	0.35754	0.26413		
5	E	Slope	1.22106	0.06997		
6	F	Intercept	0.3479	0.2575		
7	F	Slope	1.25761	0.06821		
8	B	Intercept	0.38792	0.11505		
9	B	Slope	1.07341	0.03048		
10	C	Intercept	0.37777	0.11336		
11	C	Slope	1.12638	0.03003		
12	D	Intercept	0.36722	0.11132		
13	D	Slope	1.17366	0.02949		