Red Arecanut Seed Extract as a Sustainable Corrosion Inhibitor for Aluminum Submerged in Acidic Corrodent: An Experimental Approach Towards Zero Environmental Impact

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Abstract

The effect of the red arecanut seed (RAS) extract on the corrosion of aluminum in 0.5 M hydrochloric acid environment is reported by weight loss, electrochemical (Tafel plot and impedance spectroscopy), scanning electron microscopy and atomic force microscopy studies. The weight loss study indicated that, protection efficiency of the red arecanut seed extract is directly proportional to its concentration and inversely proportional to solution temperature and aluminum contact time in the test solution. Langmuir adsorption isotherm is best fitted model explaining the adsorption of red arecanut seed extract constituents on aluminum surface in 0.5 M HCl system. The results obtained from Tafel curves indicated the mixed inhibition role of red arecanut seed extract. The impedance spectroscopy technique indicated that, red arecanut seed extract reduces the speed of aluminum corrosion by charge transfer process. The scanning electron microscopy and atomic force microscopy images of aluminum specimens clearly give clues about the adsorption of plant constituents on the surface of the aluminum metal.

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1 Introduction

Prevention of aluminum corrosion in hydrochloric acid solution is very much essential due to its economic considerations, unique characteristics and prevalent applications in industrial units. When an HCl solution is in contact with aluminum metal, HCl solutions destroys the natural protective layer formed on the metal surface. Hence, free metal surface is directly exposed to corrosive solutions and undergoes dissolution. Among the numerous techniques used to prevent metal (including Al) degradation, the use of inhibitors is the best practical and low cost method and which is greatly acceptable in several industrial sections. Inhibitors are the chemical substances contains electron rich elements in their constituents, these species adsorb on metal surface and form a protective film which is responsible for the metal corrosion inhibition process [1-6].

Previously, many organic compounds have been reported as potential corrosion inhibitors for various metals in different environments. These organic compound blocks the metal corrosion process by adsorption mode [7-11]. Even though these compounds show the robust anticorrosive property, the majority of these species are expensive and unsafe to living creatures. Hence, the use of organic compounds which possessing toxic and high cost nature as corrosion inhibitors has been banned in industrial units [12-14]. Due to these reasons; scientific community focuses on inexpensive and non toxic corrosion inhibitors. Hence, the focus is shifted towards natural products (plant extracts) because of their cheap, biodegradable and eco-friendly characteristics [15, 16].

Several scientists studied the plant products such as root, seed, husk, stem, leaves and flower extracts as effective corrosion inhibitors for numerous metals in different systems. The presence of nitrogen, phosphorous, oxygen and sulfur in plant constituents plays very important role in the corrosion control process. These plant constituents prevent the corrosion process by obstructing the electrode active sites by forming insoluble complexes (adsorbed film) on the surface of the electrode. Red arecanut seed extract possessing electron rich elements in their polyphenols (gallic acid, epicatechin, procyanidin B₁, catechin, and leucocyanidin), flavonoids (rutin), fatty acids (eicosanoic

acid, arachidonic acid, palmitic acid, oleic acid, and myristic acid, and linoleic acid), alkaloids (guvacine, isoguvacine, arecoline, guvacoline, arecaidine, and arecolidine) and tannins (tannic acid) groups [17-22]. There is no report on red arecanut seed extract as a corrosion inhibitor for any metal in any system. Therefore, in the present study, we selected red arecanut seed and studied its constituent's inhibition role.

In the present investigation, the inhibition property of the red arecanut seed extract on aluminum metal in 0.5 M hydrochloric acid environment was tested by gravimetric (weight loss), potentiodynamic polarization (Tafel plot), AC impedance spectroscopy, scanning electron microscopy (SEM) and atomic force microscopy (AFM) techniques.

2 Materials and Experimental Sections
2.1 Materials and test solution

The test was performed on aluminum metal (A-63400) having the nominal chemical composition (in weight %), Mg 0.4-0.9, Fe 0.6, Cu 0.1, Si 0.3-0.7, Mn 0.3, Zn 0.2, Tl 0.1, Cr 0.2 and remainder Al. 0.5 M hydrochloric acid solution was prepared through standard procedure.

2.2 Preparation of inhibitor

The inhibitor is prepared from the red arecanut seed (see Fig. 1). Different chemical constituents from 130 grams of red arecanut seed powder was extracted by Soxhlet technique for 7 hours duration by using 470 ml of distilled water. The different concentrations such as 2 (g/L), 4 (g/L), 8 (g/L) and 12 (g/L) was prepared and applied for corrosion studies. The presence of functional groups in the red arecanut seed extract was analyzed through Fourier transform infrared spectroscopy technique.

Fig. 1 Red arecanut seed used for the present investigation

2.3 Weight loss technique

The cleaned Al specimens were suspended in 100 ml of 0.5 M HCl solution without and with the red arecanut seed extract of different concentrations. The test is performed at 303, 308,313, 318 and 323 K. The loss in the weight of Al metal was noted with respect to different immersion time intervals (1, 2,3,4,5, and 10 h) by removing the corrosion products on aluminum surfaces. The test is carried out 3 times to confirm the reproducibility of the results.

The electrode (aluminum) corrosion rate in mils penetration per year (mpy) was evaluated by following mathematical relation,

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

where, W= aluminum weight loss in 0.5 M HCl solution (milligrams), A= aluminum surface area (in square inches), T = exposé time of aluminum in 0.5 M HCl solution (hours), D = electrode (aluminum) density (grams per cubic centimeter).

The protection efficiency of red arecanut seed extract in percentage was calculated by using below equation,

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

where, W_1 = aluminum weight loss in 0.5 M HCl solution, and W_2= aluminum weight loss in 0.5 M HCl solution plus red arecanut seed extract of different concentrations.

2.4 Electrochemical measurements

To understand the nature of corrosion and corrosion inhibition process, the electrochemical test (CHI 660 C) was carried out with three electrode systems, in which aluminum metal served as working electrode; platinum electrode acts as auxiliary electrode and saturated calomel electrode worked as a reference electrode. Before taking any readings, the aluminum specimen was submerged in 0.5 M HCl solution in the absence and presence of different concentrations of red arecanut seed extract at the open circuit potential for about one hour in order to attain a steady stable state. The degree of potentiodynamic polarization curve (Tafel plots) was recorded at the applied potential range in between +200 *mv* to -200 *mv* with scan rate of 0.01 *V/s*. Nyquist plots were obtained by applying frequency in the range between the 100000 to 1 *Hz* with an amplitude of 0.01 *V*.

2.5 Surface study

The surface morphology of the aluminum specimen immersed in 0.5 M HCl solution in the absence and presence of the red arecanut seed extract for 2 *h* duration was examined through scanning electron microscopy (SEM) and atomic force microscopy (AFM) techniques.

3 Results and Discussion
3.1 Weight loss studies

From weight loss parameters (Tables 1 and 2), it is clear that, the protection efficiency of red arecanut seed extract gradually increases with increase its amount, while aluminum corrosion rate decreases. The increase in protection efficiency is due to increasing in the adsorption of plant extract molecules (red arecanut seed extract) on the surface of the aluminum. The adsorption of plant extract molecules on aluminum surface creates a protective film which prevents the further attack of hydrochloric acid solutions on metal surface.

Table 1 Weight loss parameters at laboratory temperature

Time (h)	Concentration (g/L)	Corrosion rate × 10⁻⁴ (mpy)	Inhibition efficiency in percentage (%)
1	Blank	8.699	
	2	2.899	66.666
	4	2.416	72.222
	8	1.933	77.777
	12	1.449	83.333
2	Blank	11.116	
	2	3.866	65.217
	4	3.141	71.739
	8	2.658	76.086
	12	1.933	82.608
3	Blank	16.110	
	2	5.638	65.000
	4	4.833	70.000
	8	4.349	73.000
	12	3.222	80.000
4	Blank	18.124	
	2	6.404	64.666
	4	5.437	70.000
	8	4.954	72.666
	12	3.624	80.000
5	Blank	24.166	
	2	8.699	64.000
	4	7.249	70.000
	8	6.863	71.600
	12	4.833	80.000
10	Blank	42.291	
	2	16.916	60.000
	4	13.049	69.142
	8	12.566	70.285
	12	9.666	77.142

Table 2 Effect of 0.5 M HCl solution temperature on the protection efficiency (with contact time of one h)

Temperature (K)	Concentration (g/L)	Protection efficiency in percentage (%).
303	2	66.666
	4	72.222
	8	77.777
	12	83.333
308	2	65.000
	4	70.000
	8	75.000
	12	80.000
313	2	63.636
	4	68.181
	8	72.727
	12	77.272
318	2	60.000
	4	64.000
	8	68.000
	12	72.000
323	2	59.259
	4	62.962
	8	66.666
	12	70.370

It is also observed that, protection efficiency of red arecanut seed extract decreased with an increase in 0.5 M HCl solution temperature from 303 K to 323 K. An increase of 0.5 M HCl solution temperature increases the process of hydrogen gas evolution and hence metal dissolution rate increases. The decline in the protection efficiency values may be attributed due to reduced adsorption and increased desorption process, which is due to the high thermal motion of red arecanut seed extract molecules in 0.5 M HCl solution at elevated temperatures. This reflects the physical adsorption, which is associated with weak Vander Waals force existing between the red arecanut seed extract molecules and Al surface. The increase in the immersion time of Al metal in 0.5 M HCl solution increases the metal corrosion rate, which indicates the instability of protective film for the longer contact period.

Activation energy (Ea*), activation enthalpy (ΔH^*) and entropy (ΔS^*) are required to understand the Al corrosion inhibition process. For these purposes Arrhenius plots (Fig. 2, for activation energy) and transition state plots (Fig. 3, for activation enthalpy and entropy) were used.

The activation The calculated values were tabulated in Table 3. energy value in uninhibited 0.5 M hydrochloric acid is low compared to inhibited 0.5 M hydrochloric acid. The higher activation energy values in the presence of red arecanut seed extract molecules strongly support the results of weight loss studies and confirms the physisorption mechanism (protection efficiency decreases with an increase in 0.5 M HCl solution temperature).

The endothermic Al dissolution process in uninhibited 0.5 M HCl and inhibited 0.5 M HCl solution was confirmed by positive activation enthalpy values. The improvement of activation entropy values is an indication of a decrease in the disorderness of the system (when shifted from reactants to activated complex).

The adsorption isotherm models are required to understand the inhibition mechanism, which give details about the molecular interaction between active electrode surfaces and red arecanut seed extract molecules. In the present study, the results of weight loss studies are best fitted to the Langmuir adsorption model (Fig. 4).
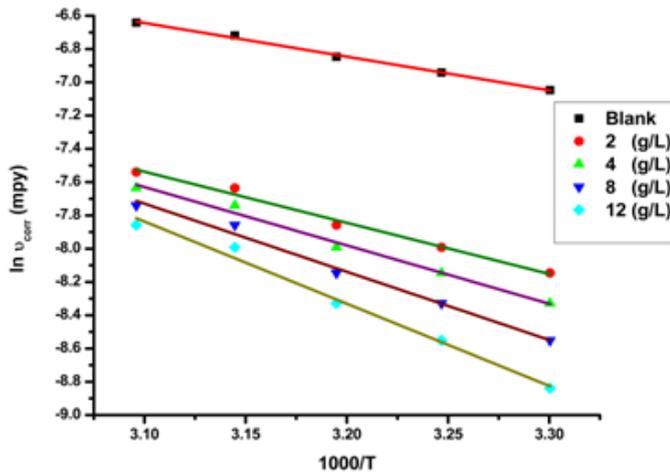
Fig. 2 Arrhenius plots (aluminum in 0.5 M HCl solution without and with red arecanut seed extract)
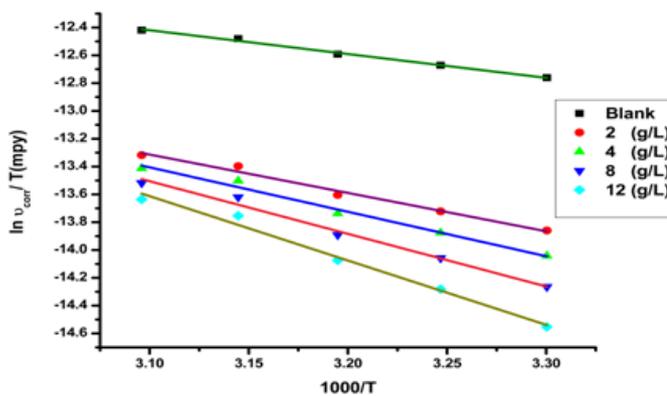
Fig. 3 Transition state plots
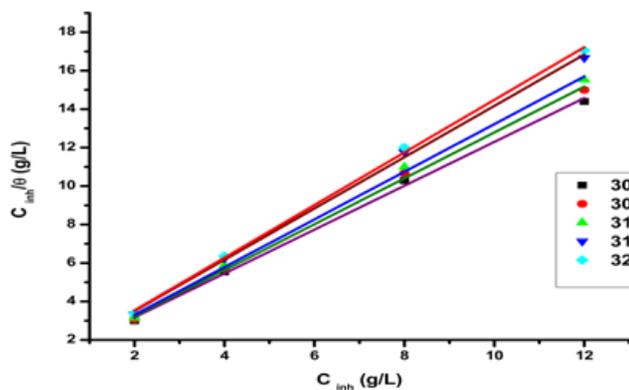
Fig. 4 Langmuir isotherm plot

Table 3 Activation parameters

C (g/L)	Ea^* (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J mol^{-1} K^{-1})
Blank	16.830	14.230	-313.901
2	25.534	22.933	-294.352
4	29.168	26.568	-283.849
8	34.062	31.462	-269.507
12	41.071	38.471	-248.672

The calculated parameters of the Langmuir adsorption model are placed in Table 4. The high equilibrium constant of the adsorption process (K_{ads}) values indicates the strong adsorption of red arecanut seed extract molecules on aluminum surface in 0.5 M HCl system. The negative standard adsorption free energy (ΔG^o_{ads}) values indicating the adsorption process of red arecanut seed extract molecules on the surface of the aluminum in 0.5 M HCl solution are spontaneous [23]. The ΔG^o_{ads} values obtained in the present case fall in between the range -35 to -37 kj/mol showing that, the adsorption of red arecanut seed extract constituents on the Al surface in 0.5 M HCl solution is both physical and chemical adsorption.

Table 4 Thermodynamic parameters

Temperature (K)	K_{ads} (L g^{-1})	ΔG^o_{ads} (kJ mol^{-1})
303	1119.106	-35.093
308	1160.025	-35.764
313	1203.224	-36.439
318	1198.207	-37.010
323	1246.416	-37.698

3.2 Electrochemical measurements
3.2.1 Potentiodynamic polarization (Tafel plot studies)

Tafel studies were carried out in order to investigate the effect of the red arecanut seed extract on cathodic and anodic reaction of aluminum. Fig. 5 represents the potentiodynamic polarization curves without and with different concentrations of red arecanut seed extract.

The corrosion current density (i_{corr}) values obtained from polarization curves are used to calculate the protection efficiency as by following equation,

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

where, i'_{corr} = the value of corrosion current density in 0.5 M HCl plus red arecanut seed extract condition and i_{corr} = the value of corrosion current density in 0.5 M HCl condition.
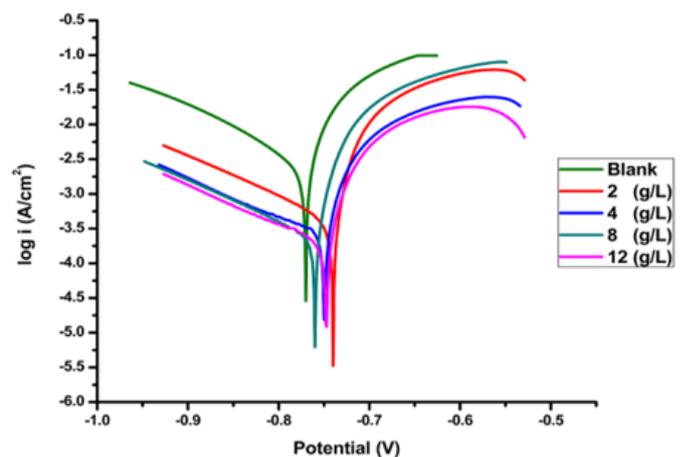
Fig. 5 Tafel plots

The various parameters obtained from polarization curves are placed in Table 5. This indicates that, the shape of Tafel plots in unprotected system and protected system remains the same which indicates that, the addition of different amounts of red arecanut seed extract effectively hinders the corrosion current density values and did not change the aluminum corrosion inhibition mechanism process.

Table 5 Tafel plot results

Concentration (g /L)	E_{corr} (mV)	$i_{corr}\times(10^{-3})$ (A/cm²)	βc (V dec⁻¹)	βa (V dec⁻¹)	Protection efficiency (%)
Blank	-770	8.916	6.679	6.031	
2	-740	1.919	5.852	6.159	78.476
4	-750	1.400	5.560	6.085	84.297
8	-760	1.340	5.977	6.976	84.970
12	-747	1.076	5.561	5.875	87.931

The decrease in i_{corr} values with improve in red arecanut seed extract concentration is due to enhanced blocked portion on the surface of the aluminum metal by red arecanut seed extract constituent's. Therefore, protection efficiency increases, whereas the corrosion rate decreases with an increase in the amount of red arecanut seed extract constituents.

The corrosion potential (E_{corr}) and anodic (β_a) and cathodic (β_c) Tafel constants not shifted towards any particular side is an evidence of mixed corrosion inhibition [24, 25] properties of red arecanut seed extract molecules on aluminum metal in 0.5 M HCl solution.

3.2.2 AC impedance spectroscopy technique

The various parameters obtained from Nyquist plots [26-30] (Fig. 6) were presented in Table 6. The protection efficiency was determined by comparing the charge transfer resistance values without and with red arecanut seed extracts constituents as follows,

$$\text{Corrosion inhibition efficiency} = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100, \quad (3)$$

where, R_{ct} = charge transfer resistance value in uninhibited 0.5 M HCl solution, and $R_{ct(inh)}$ = charge transfer resistance in inhibiting 0.5 M HCl solution.

The value of charge transfer resistance increases prominently and the value of double layer capacitance (C_{dl}) decreases with improvement in plant extract constituents in 0.5 M HCl solution, showing that plant extract species functions through adsorption at active aluminum -0.5 M HCl solution interface. The high charge transfer resistance values and low double layer capacitance values reflect the stumpy aluminum corrosion rate. Thus, a decline in aluminum corrosion rate and enhancement of adsorption occurs with the improve of red arecanut seed extract concentration. In the present study, the surface heterogeneity

factor (n) values in the range 0.695-1, which clearly indicates that, inhibition of aluminium corrosion occurs via charge transfer phenomena. These results indicate the effective corrosion inhibition property of red arecanut seed extract for aluminum metal in 0.5 M hydrochloric acid solution.
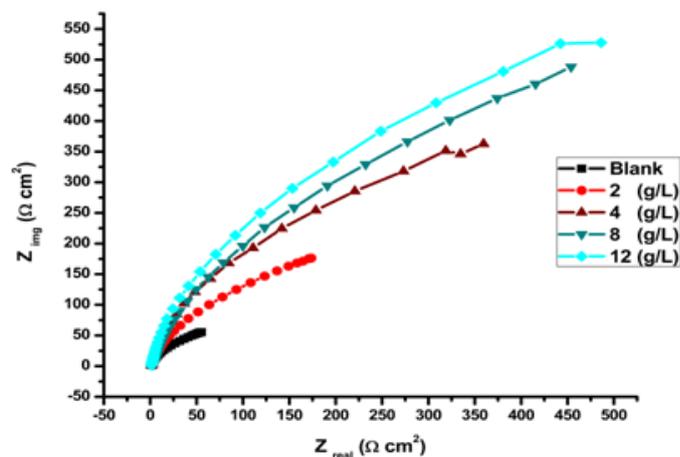
Fig. 6 Nyquist plots

Table 6 Nyquist plot parameters

Concentration (g/ L)	n	R_{ct} (Ω cm²)	C_{dl} (µF/cm²)	Protection efficiency (%)
Blank	1	55.94	291.355	
2	0.914	187.6	154.609	70.181
4	0.995	366.8	140.492	84.749
8	0.695	568.1	109.919	90.153
12	1	646	116.822	91.340

3.3 FT-IR spectroscopy

The presence of functional moieties in the red arecanut seed extract was identified through FT-IR spectroscopy technique and is shown in Fig. 7 and Table 7.
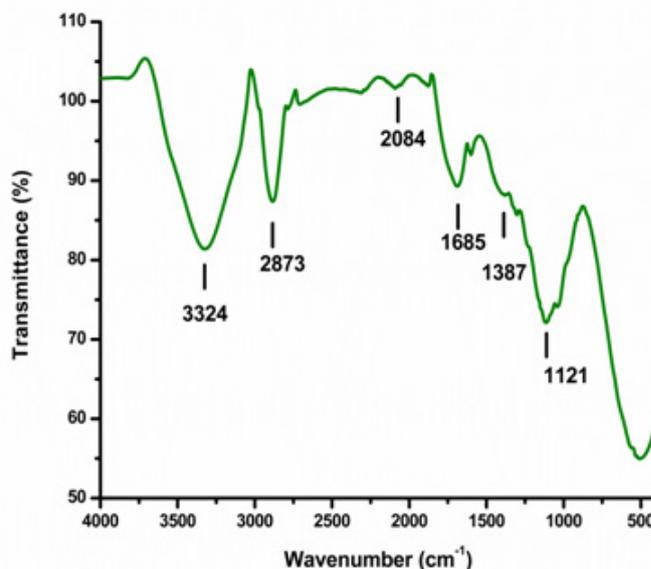
Fig. 7 IR spectrum of red arecanut seed extract

Table 7 FT-IR results

Bands	Groups
3324 cm^{-1}	monomeric hydroxyl
1685 cm^{-1}	carbonyl
1121 cm^{-1}	C-O
2084 cm^{-1}	O-C=O
2873 cm^{-1}	C-H stretching
1387 cm^{-1}	vibrations

Without inhibitor With inhibitor

Fig. 8 SEM images (aluminum metal in 0.5 M HCl solution)

3.4 Scanning electron microscopy (SEM) technique

The morphologies of aluminum surface submerged in the 0.5 M hydrochloric acid corrosive system without and with red arecanut seed extract constituents are shown in Fig. 8. The cracked aluminum surface in uninhibited hydrochloric acid solution indicates that, aluminum metal was badly corroded in the unprotected system by the hydrogen evolution process. But, in the case of inhibited hydrochloric acid solution, the cracked portions are changed to smooth layer which is due to coverage of red arecanut seed extract molecules on aluminum surface in 0.5 M HCl solution. Hence, a small aluminum portion is exposed to a corrosive solution.

3.5 Atomic force microscopy (AFM) technique

To support the SEM results, we also carried out AFM analysis and results are presented in Fig. 9 a, b and Table 8. The reduction in the roughness values (both average and root mean square roughness) in the presence of the plant extract clearly indicates the inhibitory action of green species.

(a)

Table 8 AFM results

Concentration	Average roughness (Sa)	Root mean squre roughness (Sq)
Bare	1036.6 nm	1290. 6nm
12 (g/L)	82.825 nm	133.36 nm

3.6 Comparison

The maximum protection efficiency obtained from weight loss and electrochemical techniques is shown in Table 9. From this table, it is clear that, the protection efficiency obtained from the gravimetric method is slightly different from the electrochemical methods. This is because; results obtained from the weight loss studies are reported as average values, whereas as electrochemical results are reported as instantaneous values. The weight loss studies were carried out for one hour duration and electrochemical studies are carried with a stabilization period of 30 minutes. The experimental condition of weight loss technique is different from electrochemical technique. These are the main reason for the observed deviation in the protection efficiency values.

(b)
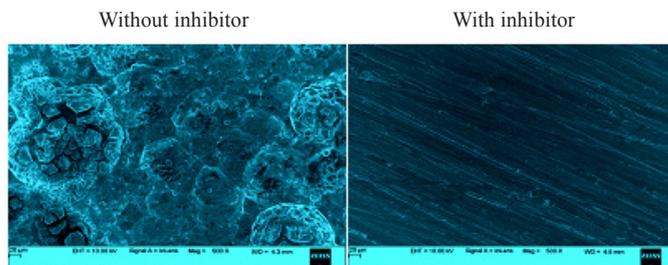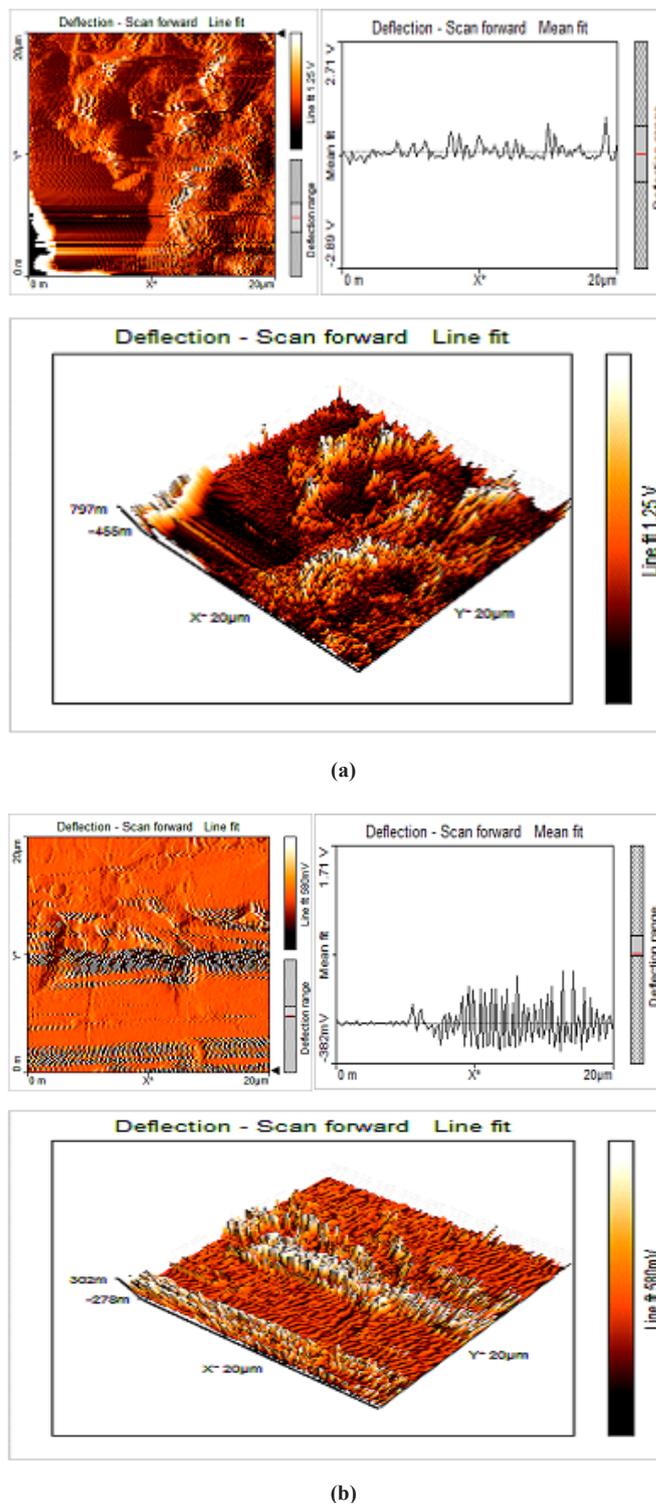
Fig. 9 (a) AFM image without inhibitor **(b)** AFM image with an inhibitor

Table 9 Protection efficiency obtained from chemical and electrochemical techniques

Optimum concentration (g/L)	Techniques (highest protection efficiency)		
	Weight loss	Tafel plot	ACimpedance spectroscopy
12	83.333	87.931	91.340

4 Conclusions

The red arecanut seed extract molecules showed the potential corrosion inhibition property on the surface of aluminum in 0.5 M hydrochloric acid environment. The efficiency of the red arecanut seed extract is increasing with increasing its amount in 0.5 M HCl solution. Slight desorption of red arecanut seed extract constituents on aluminum surface was observed with increasing solution temperature from 303 to 323 K, which indicates the physical adsorption process. Langmuir model is well fitted for studying system. The electrochemical results (both Tafel and AC impedance spectroscopy study) confirm the adsorption of red arecanut seed extract species at aluminum-HCl system interface. The smooth aluminum surface topography (obtained from by SEM and AFM technique) in the presence of plant product supports the inhibitory action of green inhibitor (red arecanut seed extract).

Acknowledgment

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