

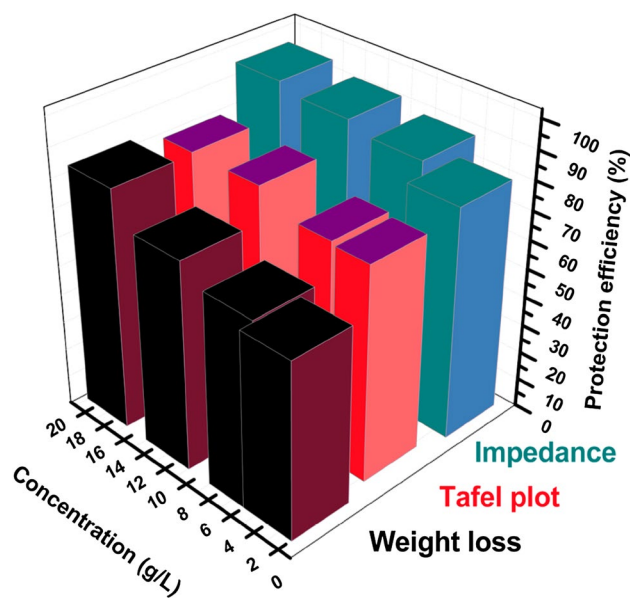
An Environmentally Friendly Approach Towards Mitigation of Al Corrosion in Hydrochloric Acid by Yellow Colour Ripe Arecanut Husk Extract: Introducing Potential and Sustainable Inhibitor for Material Protection

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Abstract The effect of extracts of yellow colour ripe arecanut husk on the inhibition of Al corrosion in 0.5 M HCl solution was examined through weight loss and electrochemical techniques. Weight loss results show that yellow colour ripe arecanut husk extract acts as a good adsorption green corrosion inhibitor for Al metal in 0.5 M HCl solution. The protection efficiency of the inhibitor varied with the inhibitor concentration. The protective action of the yellow colour ripe arecanut husk extract increased with the increase in its amounts in 0.5 M HCl solution. The Langmuir isotherm model shows that yellow colour ripe arecanut husk extract inhibits the Al corrosion in 0.5 M HCl solution by adsorption mechanism. Tafel curves proved that yellow colour ripe arecanut husk extract is mixed-type inhibitor. Results of Nyquist curves confirmed that adsorption of yellow colour ripe arecanut husk extract species led to an enhance in charge transfer resistance values and decrease in the values of double-layer capacitance. Both scanning electron microscopy and atomic force microscopy techniques are used to examine the Al surface morphology without and with the yellow colour ripe arecanut husk extract.

Graphical Abstract Protection efficiency obtained by chemical and electrochemical techniques at different inhibitor concentrations.



Keywords Yellow colour ripe arecanut husk extract · Weight loss · Protection efficiency Langmuir adsorption model · Nyquist plot

1 Introduction

Al is the most commonly used metal in several industries due to its forming and good working properties, high stiffness, lightness, high electrical conductivity and mechanical strength, low density, ease of recycling, innocuousness and ductility [1–4]. Generally, hydrochloric acid solutions are used for the electrochemical or chemical etching, pickling and descaling of Al [5, 6]. Even though aluminium has a natural protective oxide

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film, this film is an etching, when the aluminium metal is exposed to acid solutions leading to a series of electrochemical reaction [7]. Hence, it can be essential to add potential corrosion inhibitors to reduce the Al corrosion rate in acid solutions.

The literature survey concluded that organic species are the well-known corrosion inhibitors for the numerous metals in acid solutions. The electron-rich elements present in the organic compounds determine the adsorption ability of molecules on the surface of the metal. Usually, organic molecules adsorb chemically or physically on a metal surface and effectively isolate the metal surface from the corrosive ions.

Attentiveness has grown towards the employment of green extracts as potential corrosion inhibitor for the various metals in acid solutions because of their non-toxic, biodegradable and greater protection efficiency nature than the synthetic organic compounds. Inhibitors extracted from natural products are readily accepted, available, renewable sources and friendly for atmosphere and human. Many studies concerning the metal corrosion inhibition using plant extracts have been carried out on acid systems [8–21].

Yellow colour ripe arecanut husk extracts are complex mixtures possessing cellulose (high concentration) protopectin, pectin, furfuraldehyde, lignin (high concentration), hemicellulose and traces of tannins [22–24]. These species contain electron-rich elements which are expected to adsorb on the metal surface. Hence, in continuation of the development of eco-friendly corrosion inhibitors, the current study investigates the corrosion inhibition behaviour of yellow colour ripe arecanut husk extract on the Al surface in acid (0.5 M HCl) solution by chemical and electrochemical techniques. Surface studies were also carried out to prove the corrosion inhibition action of yellow colour mature arecanut husk extract on Al in 0.5 M HCl solution.

2 Experimental Details

2.1 Materials Preparation

Al (2.4 cm × 1.1 cm × 0.2 cm) of the type Al-63400 was used in the present investigation, which has the following chemical composition: Cr—0.2%, Mg—0.4–0.9%, Cu—0.1%, Si—0.3–0.7%, Mn—0.3%, Fe—0.6%, Zn—0.2%, Ti and other grain-refining elements—0.1% and Al balance [25]. The Al pieces are polished with sand papers, washed with double-distilled water, degreased with ethanol and air-dried before use. Double-distilled water and analytical-grade HCl were used for the preparation of 0.5 M HCl solution.

2.2 Preparation of Green Inhibitor

Various chemicals from green products can be extracted by different extraction methods. Among the numerous existing extraction methods, the Soxhlet extraction method is a very simple tool for plant chemical extraction. The advantages of Soxhlet extraction technique, compared to other extraction techniques, are that more amounts of different chemicals can be extracted with very small volumes of solvent. This is very important in terms of energy, time and financial inputs. For these reasons, in the present investigation, we selected Soxhlet apparatus for the extraction of chemical constituents from yellow colour ripe arecanut husk.

Soxhlet extraction is the one of the useful technique for the preparation purposes, which consists of water-cooled condenser, thimble holder, round-bottom flask and heating mantle. Figure 1 shows the schematic representation of Soxhlet device. In this, the crude plant products are placed in the thimble holder (covered with clean cotton or filter paper) and solvent is added, and before heating the solvent, the boiling chips (washed with acetone) were added to the distillation flask (round-bottom flask) to avoid bumping of resulting solution which causes damage to the Soxhlet device. The vaporized solvent undergoes condensation in water-cooled condenser, the condensed solvent dropped on thimble containing crude plant products, and solvent extracts the different chemical constituents by its contact with crude sample. When the solvent reaches an overflow level in siphon tube, the whole contents (in sample thimble) are moved into a round-bottom flask (distillation flask). The complete extraction can be achieved by repeating this operation. After the

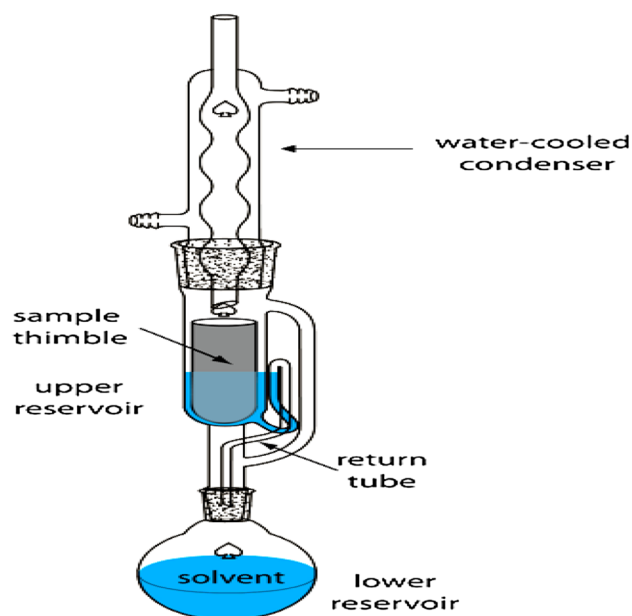


Fig. 1 Schematic representation of Soxhlet device



Fig. 2 Yellow colour ripe arecanut husk

Table 1 Physical parameters of yellow colour ripe arecanut husk extract

Density (g/ml)	Viscosity (centipoise)	Surface tension (dynes/cm)	Refractive index (n_D^{20})
1.009	0.854	92.389	1.333

complete extraction, the resulting extracted solution was cooled and placed in the refrigerator in order to avoid any physical, chemical and biological reactions of the resulting extracted sample.

Yellow colour ripe arecanut husk (Fig. 2) was collected, and 210 g of small pieces of ripe arecanut husk (yellow colour) extract was submerged in one percentage of HCl solution for about 24 h. After that, green chemicals from the yellow colour ripe arecanut husk can be extracted by using a Soxhlet apparatus. The cooled sample is purified and stored in the refrigerator. The concentrations range used for the corrosion test is 3–18 (g/L).

2.3 Characterization of Yellow Colour Mature Arecanut Husk Extract

For the proper identification of yellow colour ripe arecanut husk extract, the physical parameters such as viscosity (centipoise), density (g/ml), refractive index (n_D^{20}) and surface tension (dynes/cm) are calculated and listed in Table 1. Functional groups present in the yellow colour ripe arecanut husk extract were confirmed from the Fourier transform infrared spectroscopy technique.

2.4 Weight Loss (Gravimetric) Studies

The cleaned Al samples were weighed before exposed to 100 ml of 0.5 M HCl solution. An experiment was conducted with 3, 6, 12 and 18 (g/L) of yellow colour ripe arecanut husk

extract. The loss in the weight of Al metal was noted at different solution temperatures, i.e. 303, 308, 313, 318 and 323 K with immersion time of 1 h.

To evaluate the stability of protective film over the Al surface on timescale, gravimetric studies were performed for different contact periods (1, 2, 3, 4, 5 and 10 h) at 303 K. Experiment was repeated for three times, and average values were reported.

The corrosion rate (mpy) and protection efficiency (in percentage) were calculated from the gravimetric results by the following relations [26],

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

where A = aluminium-exposed area in 0.5 M HCl solution (sq inch), W = weight loss of aluminium (mg), D = density of aluminium metal (g cm^{-3}) and T = aluminium contact time in acid solution (h).

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

where W_2 = Al weight loss in 0.5 M HCl solution in the presence of yellow colour mature arecanut husk extract and W_1 = Al weight loss in 0.5 M HCl solution in the absence of yellow colour ripe arecanut husk extract.

2.5 Electrochemical Studies

Electrochemical tests were carried out using CHI-660C workstation and three-electrode system. In the present investigation, we used 1 cm^2 of Al as working electrode, platinum as counter electrode and saturated calomel electrode served as a reference electrode. Tafel curves were obtained in the potential range from -200 to $+200$ mV with respect to open-circuit potential with 0.01 V/s scan (sweep) rate. Nyquist plots were recorded in the frequency range 10^5 –1 Hz with the 0.01 V amplitude.

2.6 Surface Examination

The Al piece exposed for 2 h in 0.5 M HCl solution without and with the 18 g/L of yellow colour ripe arecanut husk extract. After that, the Al piece was taken out and dried. The presence of protective film on the Al surface of the protected condition was confirmed by scanning electron microscopy and atomic force microscopy techniques.

3 Results and Discussion

3.1 Weight Loss Technique

The Al corrosion rate (mpy) and protection efficiency of the yellow colour ripe arecanut husk extract obtained from the weight loss technique are shown in Tables 2 and 3. Rise in the yellow colour ripe arecanut husk extract concentration decreases the Al corrosion rate in 0.5 M HCl system and increases the protection efficiency of the green inhibitor. The decrease in the Al weight loss taking place in 0.5 M HCl solution corresponds with an enhance in the yellow colour ripe arecanut husk extract concentration. This indicates that adsorbed plant extract species protect the Al metal in 0.5 M HCl solution. The table also shows that yellow colour ripe arecanut husk extract protection efficiency decreases with a

Table 2 Gravimetric (weight loss) results at laboratory temperature (303 K) with different immersion times

Time (h)	C (g/L)	Corrosion rate [$\times 10^{-4}$ (mpy)]	Protection efficiency (%)
1	Blank	8.699	
	3	3.383	61.111
	6	2.899	66.666
	12	2.416	72.222
	18	1.449	83.333
2	Blank	11.116	
	3	4.833	56.521
	6	3.866	65.217
	12	3.141	71.739
	18	2.658	76.086
3	Blank	16.110	
	3	7.249	55.000
	6	5.638	65.000
	12	4.833	70.000
	18	4.349	73.000
4	Blank	18.124	
	3	8.216	54.666
	6	6.404	64.666
	12	5.437	70.000
	18	4.954	72.666
5	Blank	24.166	
	3	11.599	52.000
	6	8.699	64.000
	12	7.249	70.000
	18	6.863	71.600
10	Blank	42.291	
	3	24.166	42.857
	6	16.916	60.000
	12	13.049	69.142
	18	12.566	70.285

Table 3 The relationship between the 0.5 M HCl solution temperatures and the protection efficiency of the green inhibitor

Temperature (K)	C (g/L)	Protection efficiency (%)
303	3	61.111
	6	66.666
	12	72.222
	18	83.333
308	3	60.000
	6	65.000
	12	70.000
	18	80.000
313	3	59.090
	6	63.636
	12	68.181
	18	77.272
318	3	56.000
	6	60.000
	12	68.000
	18	72.000
323	3	51.851
	6	59.259
	12	66.666
	18	70.370

rise in solution temperature. This may be due to the competition existing in between the adsorption and desorption forces.

The significant variation in the protection efficiency values in the temperature range 303–323 K indicates that the mechanism of yellow colour ripe arecanut husk extract adsorption on the Al surface in 0.5 M HCl solution is by physisorption. For physisorption, inhibitor protection efficiency decreases with a rise in solution temperature, while for chemisorption, inhibitor protection efficiency increases with the rise in solution temperature. The decrease in the protective efficiency of the inhibitor at longer contact period may be due to the increase in hydrogen evolution kinetics or cathodic reaction or reducing the adsorption process. The increase in time shifts the adsorption–desorption process towards the desorption side.

Activation energy (E_a), activation entropy (ΔS) and activation enthalpy (ΔH) values give about the organic compound adsorption characteristics. From the slope of the Arrhenius plot (Fig. 3), the activation energy values can be calculated. From the slope and intercept of transition state plot (Fig. 4), the activation enthalpy and entropy values were evaluated.

Activation energy values are directly proportional to yellow colour ripe arecanut husk extract concentration. This is coupled with a reduction in protection efficiency values

Fig. 3 Arrhenius plots for Al in 0.5 M HCl solution in the absence and presence of different concentrations of yellow colour ripe arecanut husk extract

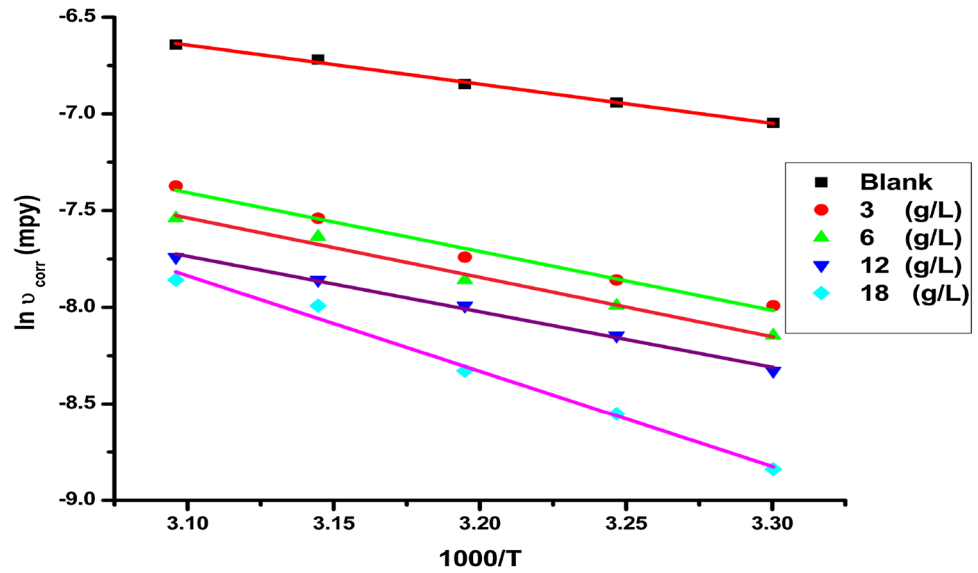
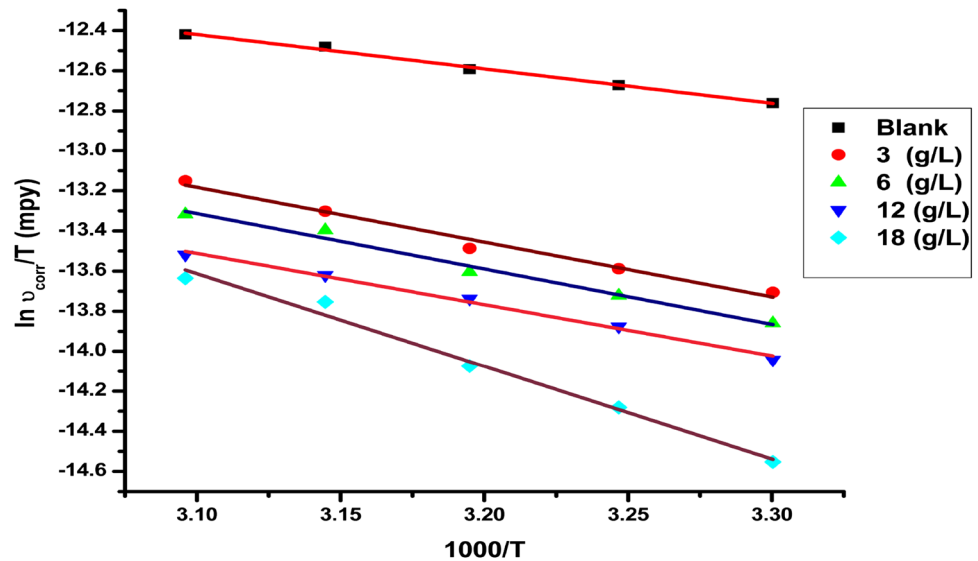


Fig. 4 Transition state plots for Al in 0.5 M HCl solution without and with different concentrations of yellow colour ripe arecanut husk extract



with an increase in acid solution temperature, indicating that yellow colour ripe arecanut husk extract did not vary the Al corrosion inhibition mechanism. Rather, inhibition of Al corrosion takes place primarily via blocking effect of the yellow colour ripe arecanut husk extract on the surface of Al, hindering the access of corrosive ions.

In the present case, the activation enthalpy values are positive, which shows that adsorption of yellow colour ripe arecanut husk extract species on the surface of Al is exothermic. The activation entropy values are in a negative mode, which represents the decline in the degree of orderliness of the yellow colour ripe arecanut husk extract molecules (Table 4).

Information about the interaction of plant extract species with the surface of Al can be clearly understood by the

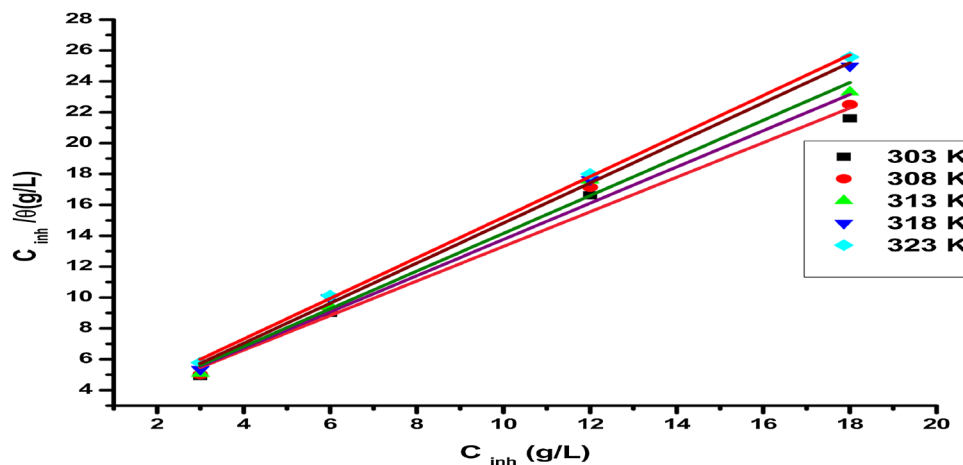
adsorption isotherm models. Hence, adsorption isotherm models are very important in the field of corrosion. In the present case, adsorption characteristics of yellow colour ripe arecanut husk extract on the Al surface in 0.5 M HCl solution were explained by the Langmuir isotherm model. Figure 5 shows the graphical representation of the Langmuir isotherm model (plot of C_{inh} vs C_{inh}/θ). With the help of Langmuir plot, adsorption equilibrium constant (K_{ads}) and free energy of adsorption (ΔG_{ads}°) values were obtained and are listed in Table 5.

On the present investigation, ΔG_{ads}° values are in between the range of -32 to -35 kJ/mol. It is well known that ΔG_{ads}° values in between -40 and -20 kJ/mol indicate the mixed-type interaction. Hence, obtained values propose that adsorption of yellow colour ripe arecanut

Table 4 Kinetic parameters for the yellow colour ripe arecanut husk extract on the surface of Al in 0.5 M HCl solution

Inhibitor	C (g L ⁻¹)	E_a (kJ mol ⁻¹)	ΔH (kJ mol ⁻¹)	ΔS (J mol ⁻¹ K ⁻¹)
Yellow colour ripe arecanut husk extract	Blank	16.830	14.230	- 313.901
	3	25.286	22.685	- 294.034
	6	25.534	22.933	- 294.352
	12	23.846	21.246	- 301.229
	18	41.071	38.471	- 248.672

C concentration

Fig. 5 Langmuir isotherm plot for Al in acid solution in the presence of different concentrations of yellow colour ripe arecanut husk extract**Table 5** Thermodynamic parameters for Al in 0.5 M HCl solution with different concentrations of yellow colour ripe arecanut husk extract

Inhibitor	Temperature (K)	K_{ads} (L g ⁻¹)	ΔG_{ads}^0 (kJ mol ⁻¹)
Yellow colour ripe arecanut husk extract	303	473.983	- 32.928
	308	494.268	- 33.579
	313	515.312	- 34.232
	318	541.535	- 34.910
	323	486.442	- 35.171

husk extract species on the Al surface in 0.5 M HCl solution takes place spontaneously and it involves two types of interaction, i.e. physical adsorption and chemical adsorption, with predominant physical interaction. K_{ads} values signify the strength between the adsorbent and adsorbate. Large K_{ads} values represent that inhibitory species are strongly adsorbed on the surface of metal and therefore superior protection efficiency. In the present case, K_{ads} values are high, which clearly shows that adsorption of yellow colour ripe arecanut husk extract species on the surface of Al is easy and effective. As a result of effective adsorption of plant extract species on Al surface, the yellow colour ripe arecanut husk extract species shows the good inhibition or protection efficiency property.

3.2 Electrochemical Measurements

3.2.1 Tafel Plot Studies

Tafel studies were performed in the absence and presence of yellow colour ripe arecanut husk extract of different concentrations in order to understand the process of cathodic and anodic reactions of Al in the acid medium. The Tafel curves of Al metal without and with plant extracts are shown in Fig. 6. The Tafel parameters such as corrosion current density (i_{corr}), corrosion potential (E_{corr}) and cathodic and anodic Tafel slopes (β_c , β_a) are obtained from the Tafel plots and are included in Table 6.

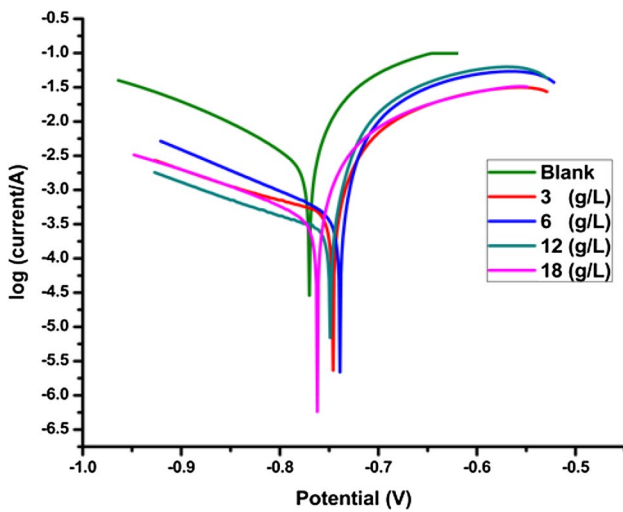


Fig. 6 Tafel curves for Al metal in 0.5 M HCl solution without and with four different concentrations of yellow colour ripe arecanut husk extract

The protection efficiency of the yellow colour ripe arecanut husk extract for each concentration was derived from the equation below,

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

where i_{corr} = corrosion current density without yellow colour ripe arecanut husk extract, and i'_{corr} = corrosion current density with yellow colour ripe arecanut husk extract.

The corrosion current values are inversely proportional to the yellow colour ripe arecanut husk extract concentration, which indicates that increasing the plant extract concentration greatly reduces the both anodic and cathodic aluminium reactions in acid solution. Lower corrosion current density values in the presence of inhibitor are an indication of higher protection efficiency of the yellow colour ripe arecanut husk extract for Al metal in 0.5 M hydrochloric acid solution. On the present investigation, the corrosion potential values do not move towards any side. Further, both β_c and β_a values are equally affected by the yellow colour ripe arecanut husk extract species. These indicate

Table 6 Tafel plot parameters

Inhibitor	Concentration (g L ⁻¹)	E_{corr} (mV)	$i_{\text{corr}} \times 10^{-3}$ (A)	β_c (V dec ⁻¹)	β_a (V dec ⁻¹)	Protection efficiency (%)
Yellow colour ripe arecanut husk extract	Blank	- 770	8.916	6.679	6.031	
	3	- 746	2.277	4.311	5.946	74.461
	6	- 739	2.171	6.005	6.031	75.650
	12	- 749	1.635	4.779	6.638	81.662
	18	- 762	1.629	5.197	6.399	81.729

Fig. 7 Nyquist plots for Al metal in the acid system without and with different concentrations of yellow colour ripe arecanut husk extract

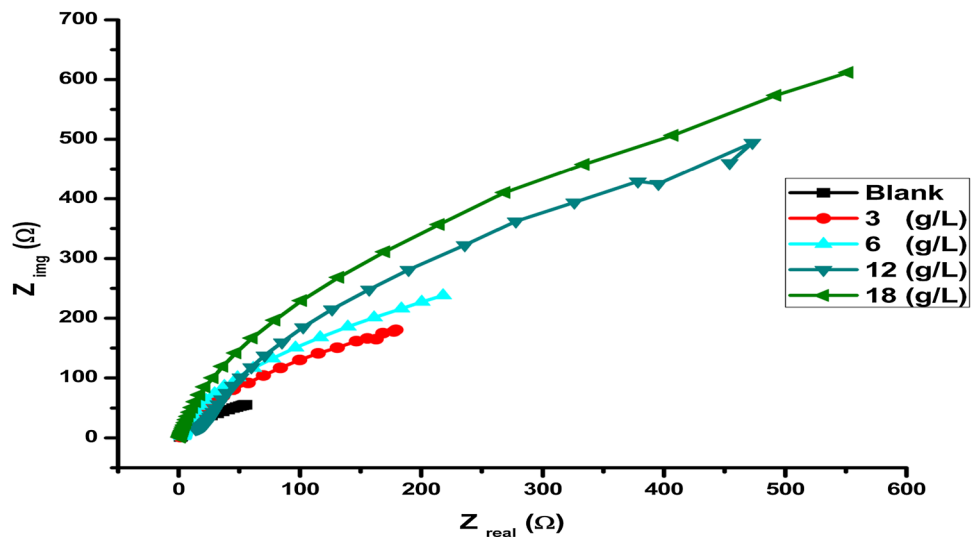


Table 7 Impedance results for Al in 0.5 M HCl in the absence and presence of yellow colour ripe arecanut husk extract of different concentrations

Inhibitor	Concentration (g L ⁻¹)	<i>n</i>	<i>R</i> _{ct} (Ω)	<i>C</i> _{dl} (μF)	Protection efficiency (%)
Yellow colour ripe arecanut husk extract	Blank	1	55.94	291.355	
	3	0.5696	284.8	219.260	80.358
	6	0.5502	535	96.322	89.543
	12	0.8064	743.9	123.020	92.480
	18	0.6338	1179	114.457	95.255

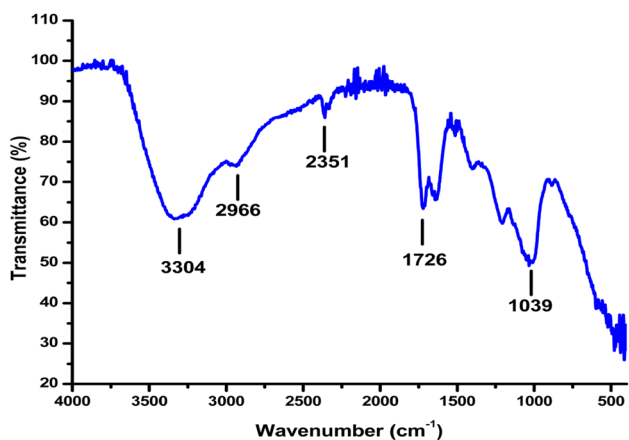


Fig. 8 IR spectrum of yellow colour ripe arecanut husk extract

that investigated inhibitor (yellow colour ripe arecanut husk extract) acts as a mixed type of inhibitor on the Al surface in studying medium.

3.2.2 AC Impedance Spectroscopy

Nyquist curves (Fig. 7) were obtained for Al metal in 0.5 M HCl solution in order to understand the Al corrosion behaviour in the absence and presence of the yellow colour ripe arecanut husk extract. From the Nyquist plot, surface heterogeneity factor (*n*), double-layer capacitance (*C*_{dl}) and charge transfer resistance (*R*_{ct}) values were obtained.

The protection efficiency of the inhibitor can be calculated by using *R*_{ct} values as follows,

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

where *R*_{ct} = charge transfer resistance without green inhibitor, and *R*_{ct(inh)} = charge transfer resistance with green inhibitor.

All these values are placed in Table 7. The increase in the amounts of yellow colour ripe arecanut husk extract in acid solution enhances the *R*_{ct} values and reduces the *C*_{dl} values, showing that inhibition of the Al corrosion process takes place via charge transfer phenomenon. The increase in *R*_{ct}

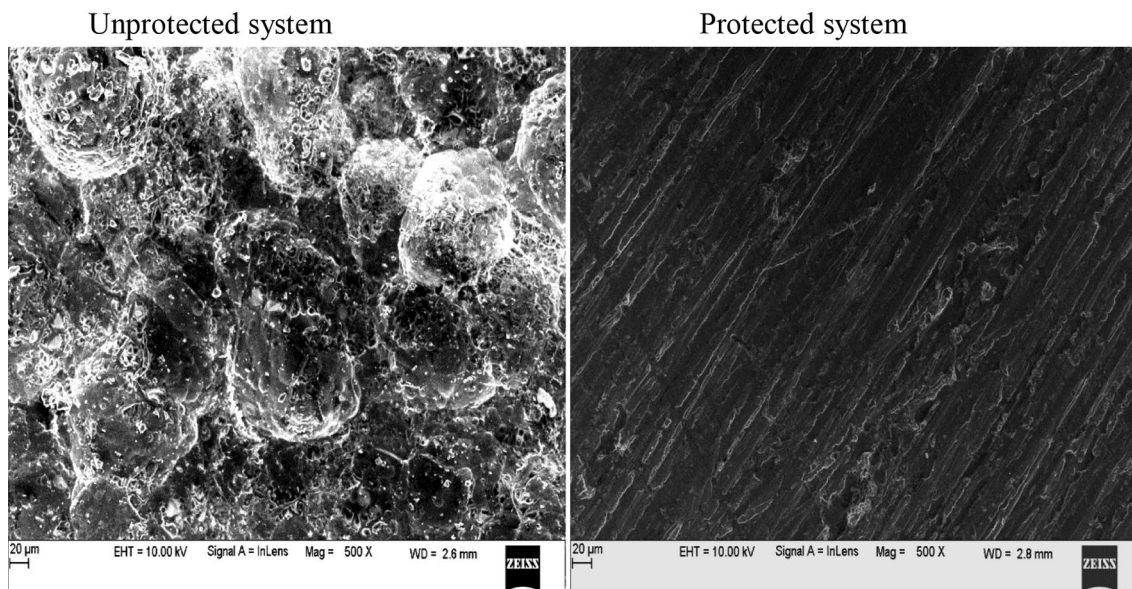


Fig. 9 SEM photographs of Al specimens without and with inhibitor

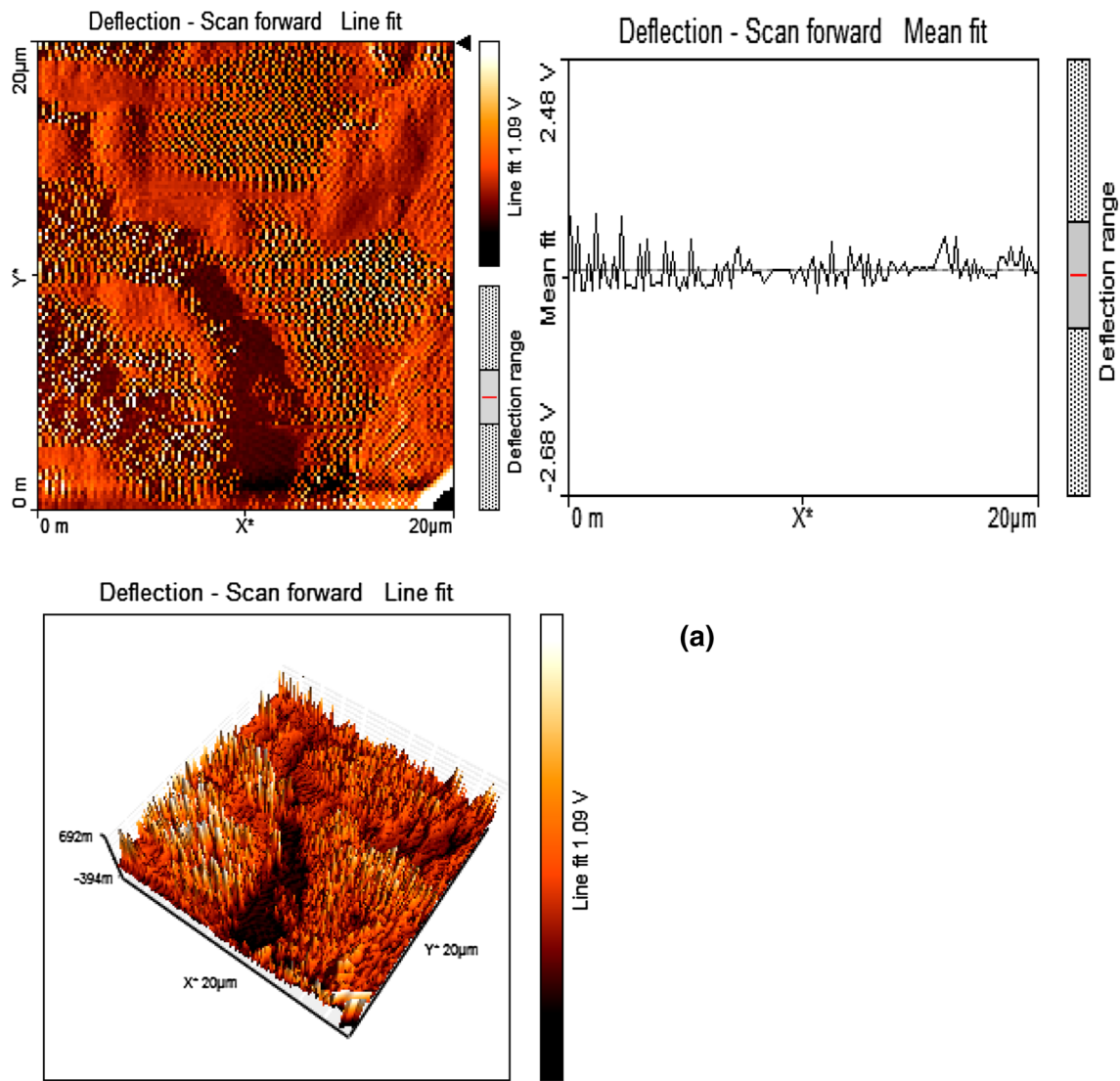


Fig. 10 (a, b) AFM images of Al specimen in 0.5 M hydrochloric acid solution, **a** without yellow colour ripe arecanut husk extract, **b** with 18 g/L of yellow colour ripe arecanut husk extract

value with the concentration of the inhibitor is the result of enhancing in the surface coverage by the plant extract species which leads to increase in protection efficiency values. The reduction in the C_{dl} values with an increase in the concentration of yellow colour ripe arecanut husk extract may be due to the adsorption of plant extract species by replacing the H_2O molecules at aluminium–hydrochloric acid interface, which increase the double-layer thickness. Hence, the Al dissolution rate decreases with an increase in the natural extract concentration. The n values also support the corrosion inhibition role of yellow colour ripe arecanut husk extract on the Al surface in 0.5 M hydrochloric acid solution.

3.3 Functional Group Analysis by FT-IR Spectroscopy Technique

FT-IR spectroscopy technique is used to determine the electron-rich elements in the extract of yellow colour ripe arecanut husk. Figure 8 clearly shows the yellow colour ripe arecanut husk extract that consists of hydroxyl, carbonyl and other electron-rich centres in their moieties. These species cover the Al surface in 0.5 M HCl solution and retard the corrosion process.

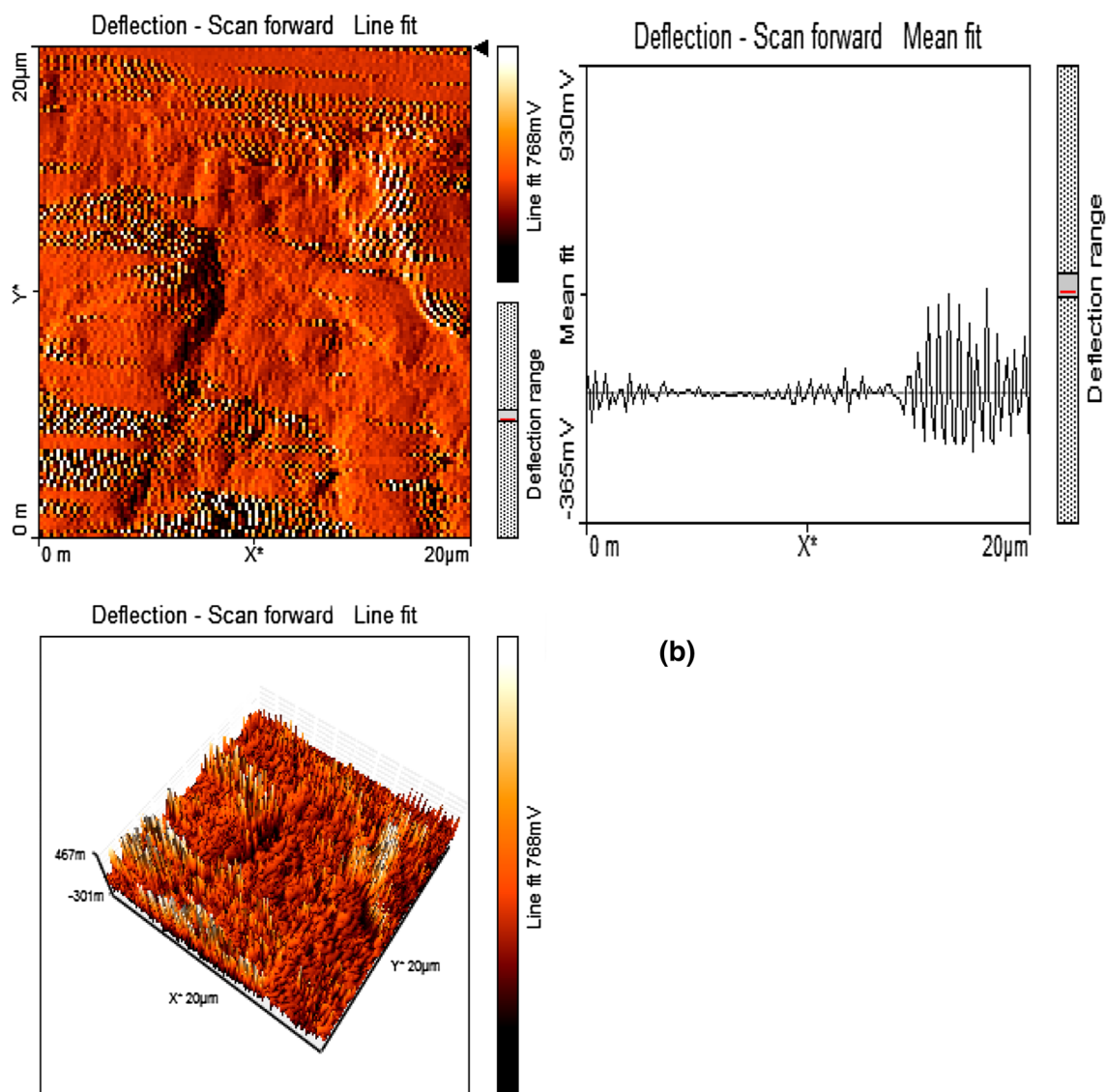


Fig. 10 (continued)

3.4 Surface Examination

3.4.1 Scanning Electron Microscopy

The Al surface analysis was carried out by SEM technique. Figure 9a, b represents the micrographs of Al sample submerged in 0.5 M HCl solution without and with 18 g/L of yellow colour ripe arecanut husk extract. In the absence of yellow colour ripe arecanut husk extract, the Al surface is corroded due to attack of free 0.5 M HCl solution ions. As a result of number of scratches and cavities on the Al surface which is clearly seen from the resulted SEM photograph, in the presence of green inhibitor (yellow colour ripe arecanut husk extract), the Al surface is covered by the plant extract

species, which controls the Al dissolution process in acid medium. Hence, smooth surface is observed in the presence of yellow colour ripe arecanut husk extract.

3.4.2 Atomic Force Microscopy Technique

To determine the roughness value of Al surface in protected and unprotected systems, the treated Al sample is submitted to AFM surface test. The resulted image is shown in Fig. 10a, b. The roughness values of Al sample obtained from AFM photograph are shown in Table 8. This table shows that the roughness value of Al surface is low in the presence of yellow colour ripe arecanut husk extract compared to blank solution, which shows that the protective film

Table 8 AFM results

Concentration (g/L)	Average roughness (Sa) (nm)	Root mean square roughness (Sq) (nm)
0	437.65	660.81
18	299.75	374.34

formed by yellow colour ripe arecanut husk extract species decreases the Al dissolution rate in 0.5 M HCl solution.

4 Conclusion

Yellow colour ripe arecanut husk extract was found to be the potential eco-friendly corrosion inhibitor for Al metal in 0.5 M HCl solution, particularly at 303 K. The protection efficiency of the plant extract may be due to the presence of cellulose, hemicelluloses, pectin, protopectin, furfuraldehyde, lignins and traces of tannins in the extract. Weight loss study shows that the inhibition behaviour of yellow colour ripe arecanut husk extract on the Al surface in hydrochloric acid solution is basically controlled by immersion time, temperature and concentration of the inhibitor. The obtained standard free energy of adsorption (ΔG_{ads}^0) values indicates that the adsorption of yellow ripe arecanut husk extract on the surface of the working electrode (Al) is spontaneous. Tafel curves show that yellow colour ripe arecanut husk extract species suppress the both anodic and cathodic reactions. Nyquist plots reflect that the introduction of different concentration of yellow colour ripe arecanut husk extract greatly affects the charge transfer resistance (R_{ct}) values. SEM and AFM photographs further support that the yellow colour ripe arecanut husk extract species significantly protect the Al surface in acid medium.

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Compliance with Ethical Standards

Conflict of interest The authors declare no conflict of interest.

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