

Chasew (*Anacardium Occidentale*) Leaves Extract as Green Corrosion Inhibitor of API 5L X52 in Acidic Media

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Abstract

Cashew leaves extract has been investigated as green corrosion inhibitor to inhibit the corrosion process of API 5L X52 in acidic media. The inhibition effect of cashew leaves was analyzed using electrochemical measurement such as Tafel Polarization and Electrochemical Impedance Spectroscopy (EIS). FTIR, polyphenol content and phytochemical analysis were also employed to ensure the chemical compound of cashew leaves extract. Concentration of cashew leaves extract was varied for electrochemical measurement i.e. 0, 100, 200, 300, 400 and 500 ppm. Furthermore, immersion time variation (0, 30 and 60 min) was used prior to electrochemical measurement. The electrochemical measurement results indicate that cashew leaves extract work effectively as green corrosion inhibitor of API 5L X52 in acidic media. The performance of this green corrosion inhibitor was optimum at concentration of 500 ppm and 60 min of immersion time. Cashew leaves extract was mixed-type inhibitor because the corrosion potential value shifted less than 85 mV. The Increasing of surface resistance value and the decreasing in the capacitance of double layer due to the adsorption process of cashew leaves extract molecules on the API 5L X52 steel surface.

Keywords: API 5L X52, Cashew leaves, Green corrosion inhibitor, Adsorption, Electrochemical measurement

Introduction

API 5L X52 is one of the most widely pipeline material used in oil and gas industry to transmit petroleum products [1]. Petroleum products contain corrosive agents such as sulfur, carbon dioxide and chloride that can react with water and form acid. However, API 5L X52 is vulnerable to corrosion when exposed to acidic media [2].

Internal corrosion due to corrosive fluid is a challenging and damaging problem for oil and gas industry worldwide [3-6]. Corrosion is a major economic significance because the annual cost of failures due to corrosion in oil and gas industry could be around \$US 2.4 trillion, estimated by World Corrosion Organization (WCO) [7]. Thus, the corrosion controlling method is required to protect pipeline material from internal corrosion. Although there are various methods to control corrosion, the use of corrosion inhibitors is one of the best methods for protecting pipeline material against corrosion [8,9].

Corrosion inhibitors are chemical compounds that are added in small concentration to a fluid to control the dissolution of metals by adsorbing on the metal surface [10-12]. Corrosion inhibitors generally employed in oil and gas companies have been found to be hazardous and toxic to human and the environment. Thus, research are now directed towards of green corrosion inhibitors [13-22].

Efforts have been made to use plant extract as green corrosion inhibitors because they are cheap, readily available, renewable, ecologically acceptable and environmentally friendly [23-28]. Many researchers reported green corrosion inhibitor based on natural products to inhibit corrosion attack of metal in acidic media. For instance, Kaban *et al.* [29] use White tea extract as green corrosion inhibitor. In addition, Mehdipour *et al.* employ Aloe extract [30], Shan Wan *et al.* use Soybean extract [31] and Hassan *et al.* use cashew nut as green corrosion inhibitor [32].

Despite several studies on cashew as green corrosion inhibitor are available [33], there is no literature exploit cashew leaves extract. Cashew is a plant that grows a lot in tropical countries such as Brazil, India and Indonesia. This plant can grow up to 12 m in fertile soil with high humidity (70 - 80 %). Cashews grow well in areas with warm temperature (25 - 35 °C). The harvest period lasts for 4 months, from November to February the following year [34]. Cashew nut is a high-value export crop and used as a source of income by millions of rural households in Indonesia. However, Indonesia has not been able to develop cashew leaves into products that have a higher economic value. Cashew leaves contains polyphenol and flavonoid based on research by Dahlia *et al.* [35]. Polyphenol and flavonoid are reported to be effective as corrosion inhibitor [36,37]. In addition, the number of cashew leaves is much more than the fruit. Hence, the present work is directed at the investigation of extracts from cashew leaves as green corrosion inhibitor for inhibiting the corrosion of API 5L X52 in HCl media highlighted by electrochemical measurement.

Materials and methods

Cashew leaves extract preparation

The cashew leaves (*Anacardium Occidentale*) were collected from the nearby locality and shade dried. The extraction process starts with cleansing cashew leaves with distilled water then drying at room temperature. After drying, cashew leaves were pulverized using blender to become powder. Two hundred fifty g of cashew leaves powder was soaked in ethanol for 3 days. The extract was filtered and concentrated at temperature 100 °C. The resulting extract was analyzed using FTIR, total polyphenol content and phytochemical screening.

Specimen and solution preparation

API 5L X52 steel containing C = 0.046 %, Mn = 1.046 %, Si = 0.288 % and Fe = 98.46 % were used in this work. The steel was cut with an area of 1 cm². The specimens were connected to electrical wire, mounted using epoxy resin, and polished with a series of emery paper from 120 to 1,200 grit.

The solutions are prepared by making 1M HCl solution from analytical grade 37 % HCl. Eighty-three mL of 37 % HCl was dissolved with 1,000 mL of double distilled water in the absence and presence of cashew leaves extract in the concentration range from 100 to 500 ppm.

Electrochemical measurement

The electrochemical measurements were conducted using Gamry G750 corrosion measurement system. Tafel polarization and EIS were carried out in a conventional 3-electrode electrochemical cell containing 300 mL of solutions. API 5L X52 specimen was used as working electrode, saturated calomel electrode was utilized as reference electrode, and platinum was employed as counter electrode.

Tafel polarization curves were obtained by shifting the electrode potential automatically from -250 to +250 mV versus corrosion potential (E_{corr}) at a scan rate of 0.01 v/s. By means of Electrochemical Impedance Spectroscopy (EIS), an amplitude of sinusoidal perturbation signal of ± 10 mV, with a frequency range between 10 kHz to 0.2 Hz, was used.

Results and discussion

Chemical analysis of cashew leaves extract

Chemical compound of cashew leaves extract analyzed using FTIR. Total phenol content and phytochemical screening utilized as quantitative and qualitative analysis, respectively. FTIR result of cashew leaves extract is presented in **Figure 1**.

From **Figure 1**, the highest peak at approximately 2,925.17 and 2,854.77 cm⁻¹ can be assigned to C-H stretching vibration. This functional group is non-polar and hydrophobic which can repel water molecule on the steel surface. The broad band at 2,618.48 cm⁻¹ can be attributed to carboxylic acid COOH which is a combination of a carbonyl group (-CO-) and a hydroxyl group (-OH). The peak at 1,700.32 and 1,611.59 cm⁻¹ can be associated with C=O and C=N, respectively. The band at 1,517.08 cm⁻¹ is related to C=C aromatic rings. The absorption peak at 765.77 cm⁻¹ can be attributed to C-H deformations of the aromatic ring. This result show that cashew leaves extract contains O, C and N elements with aromatic rings, which are attracted to the metal surface due to the existence of free electrons.

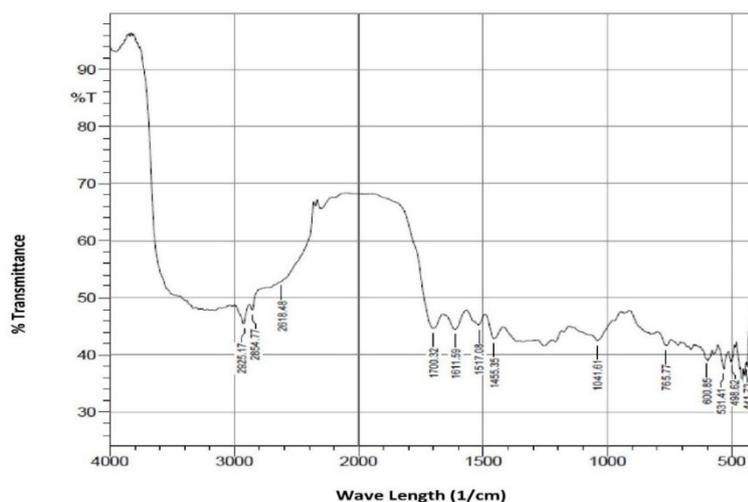


Figure 1 FTIR result of cashew leaves extract.

Table 1 determine the correlation between gallic acid and the absorbance of the molecules at 765 nm in total phenol content (TPC) test. The data in **Table 1** can be plotted as illustrated in **Figure 2**. TPC of cashew leaves extract then calculated through the regression equation of the curve in **Figure 2**. Cashew leaves extract has 27.485 mg of total phenol content according to the calculation result. Based on **Table 1** and **Figure 2**, the absorbance increase with the increasing of gallic acid concentration. This result suggests that the phenolic content enhances the antioxidant properties of the cashew leaves extract and increases the possibility for the cashew leaves to adsorb on the steel surface [29].

Table 1 Calibration data of TPC test.

Concentration (μmL)	Absorbance		
	A1	A2	Average
0	0.0478	0.0495	0.0487
5	0.3594	0.3948	0.3771
10	0.7203	0.7193	0.7193
20	1.4164	1.4378	1.4378
30	2.1401	2.1723	2.1723
40	2.9095	2.9267	2.9267

Chemical compound of cashew leaves extract also was analyzed qualitatively using phytochemical screening. **Table 2** summarizes the phytochemical screening result of cashew leaves extract. According to **Table 2**, cashew leaves extract contain tannin, saponin, terpenoid/steroid, flavonoid and alkaloid. Tannins are polyphenolic compounds with O-H groups that can increase the formation of passive layer so that corrosion process hampered. Flavonoid act as antioxidant that can inhibit oxidation reaction of steel. Alkaloid have nitrogen element which is attracted to the steel surface due to the existence of free electrons. Tannins, alkaloids and flavonoid are antioxidants because they act as free radicals scavengers by removing a hydrogen atom from its hydroxyl group. The hydrogen atom that released can bind to free radicals, to a neutral charge. They loses a hydrogen atom then undergoes resonance of the hydroxyl group which causes its activity energy decreases and remains stable [38].

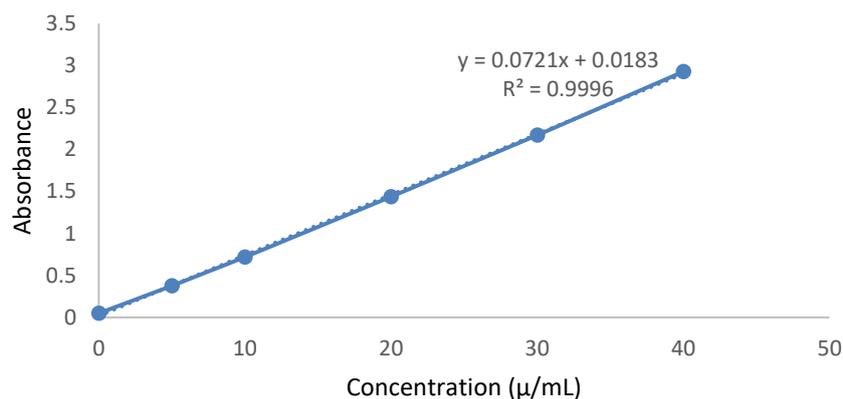


Figure 2 Linear fitting calibration data of TPC test.

Table 2 Phytochemical screening of cashew leaves extract.

No	Phytochemical	Reactor	Result
1	Tannin	FeCl ₃	+
2	Saponin	Aquadest + HCl	+
3	Flavonoid	Zn + HCl	+
4	Terpenoid/ Saponin	Chloroform + acetate anhydrate + H ₂ SO ₄	+
5	Alkaloid	Bouchardat	+
		Dragendorf	+

Tafel polarization analysis

Tafel polarization curves of API 5L X52 in HCl with various cashew leaves inhibitor concentration are presented in **Figure 3**. It is observed that the presence of cashew leaves inhibitor retarded both anodic and cathodic reactions. Both anodic and cathodic current densities with 0 min of immersion time (**Figure 3(a)**), 30 min (**Figure 3(b)**) and 60 min (**Figure 3(c)**) were shifted reduced in the presence of inhibitor [23].

The inhibition performance of cashew leaves extract was more pronounced at higher concentration. Moreover, the Tafel polarization curves displace toward positive corrosion potential (E_{corr}) and less corrosion current densities (I_{corr}) in the presence of cashew leaves extract. The shiftment of the curves was more pronounced in the anodic curves, which corresponds to the dominant anodic inhibition mechanism of the cashew leaves extract. However, the corrosion potential (E_{corr}) showed no significant change (under 85 mV) in the presence of inhibitor which suggest a mixed type inhibitor.

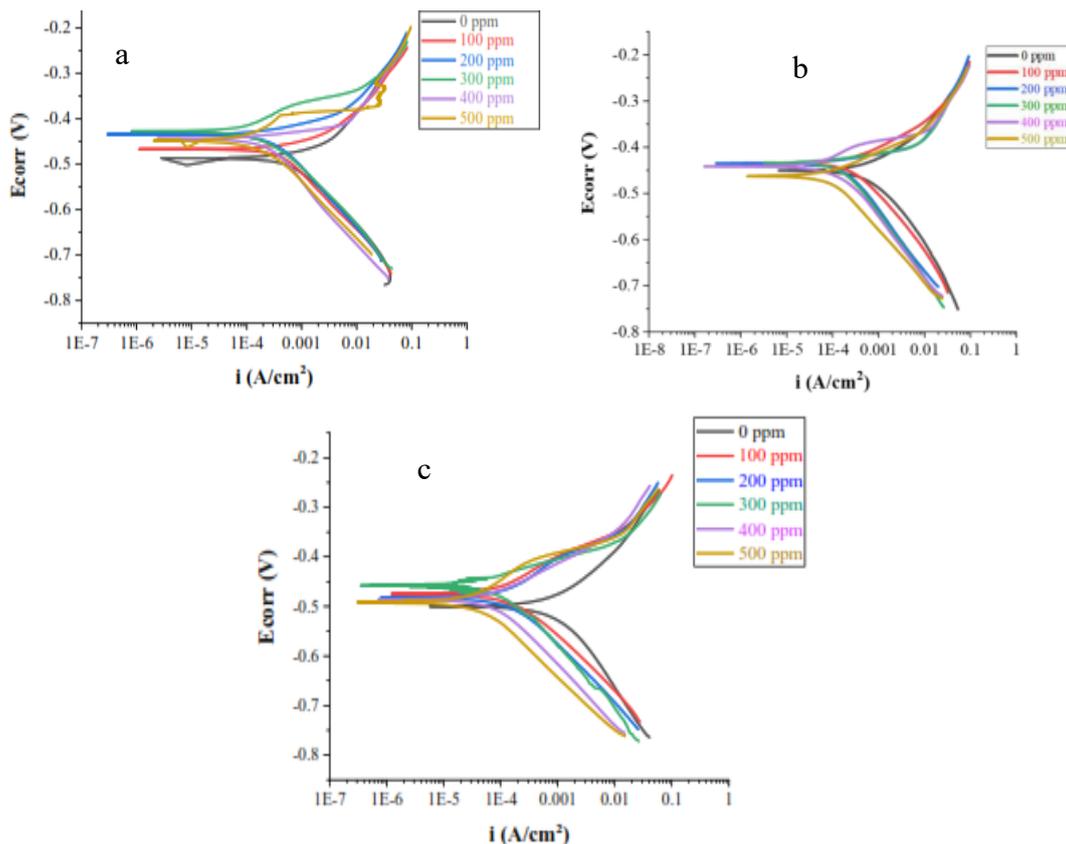


Figure 3 Tafel polarization curves of API 5L X52 in acidic media and various concentration of extract with (a) 0 min, (b) 30 min, and (c) 60 min of immersion time.

The corrosion rate obtained from the extrapolation of linear Tafel curves are given in **Figure 4**. From **Figure 4**, it is observed that corrosion rate decrease with the increase of inhibitor concentration and immersion time. The decreasing of corrosion rate due to the stronger of adsorption process on the steel surface, so that a protective film is formed at the interface of the steel surface with the acidic media [39].

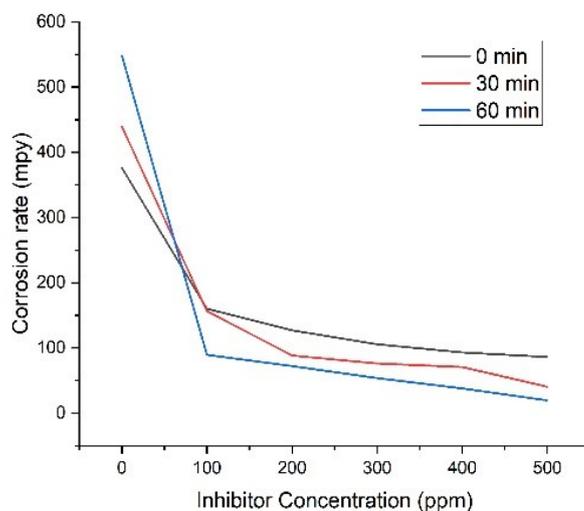


Figure 4 Corrosion rate vs inhibitor concentration plot.

Inhibitor efficiency (η) can be calculated based on corrosion rate with (CR_{in}) and without inhibitor (CR) according to Eq. (1) [40].

$$\eta (\%) = (CR - CR_{in})/CR \times 100 \% \tag{1}$$

Figure 5 depicts the inhibitor efficiency at each inhibitor concentration. As the increasing of inhibitor concentration, the ability of the inhibitor molecule to be adsorbed on the steel surface increases. Therefore, the value of the inhibitor efficiency also increases as shown in **Figure 5**. The significantly increased of inhibitor efficiency value has a low corrosion rate. The increase in inhibitor concentration and immersion time causes a higher of surface coverage. The efficiency inhibitor enhances with the inhibitor concentration and immersion time. The highest of inhibitor efficiency (96.42 %) was 500 ppm of inhibitor concentration and 60 min of immersion time. All polarization parameters are listed on **Table 3**.

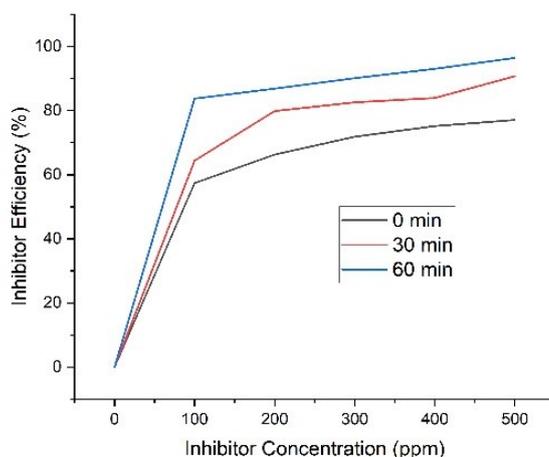


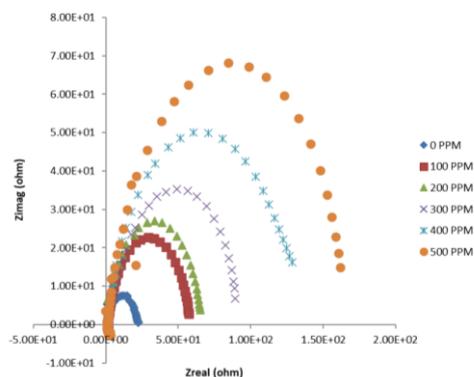
Figure 5 Inhibitor efficiency vs inhibitor concentration plot.

Table 3 Polarization parameter.

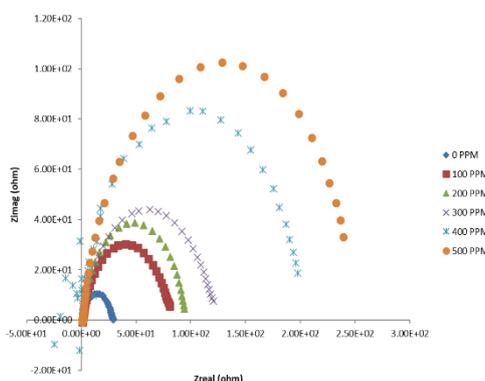
Time (min)	$C_{inhibitor}$ (ppm)	E_{corr} (mV)	I_{corr} (A/cm ²)	CR (mpy)	η (%)
0	0	-486.3	820.9×10^{-6}	376.1	0
	100	-467	349.9×10^{-6}	160.3	57.38
	200	-434.3	277.0×10^{-6}	126.9	66.26
	300	-426.8	231.0×10^{-6}	105.8	71.87
	400	-443.9	203.8×10^{-6}	93.36	75.18
	500	-447.6	188.4×10^{-6}	86.32	77.05
30	0	-450.8	958.8×10^{-6}	439.2	0
	100	-434.3	341.5×10^{-6}	156.4	64.39
	200	-434.7	192.8×10^{-6}	88.31	79.89
	300	-433.8	166.9×10^{-6}	76.45	82.59
	400	-441.7	154.3×10^{-6}	70.67	83.91
	500	-462.9	88.56×10^{-6}	40.57	90.76
60	0	-499.4	1.197×10^{-3}	548.4	0
	100	-474.6	195.1×10^{-6}	89.36	83.71
	200	-482.4	157.7×10^{-6}	72.24	86.83
	300	-457.3	117.9×10^{-6}	54.00	90.15
	400	-485.6	83.24×10^{-6}	38.13	93.05
	500	-491.4	42.88×10^{-6}	19.64	96.42

EIS analysis

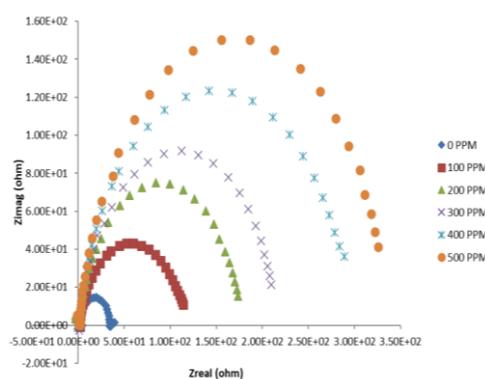
EIS measurement was used to characterize the kinetics of the electrochemical processes and capacitive behaviors at acidic/steel interface in the presence and absence of cashew leaves inhibitor. **Figure 6** shows Nyquist plot of API 5L X52 in acidic media with various inhibitor concentration and immersion time.



(a)



(b)



(c)

Figure 6 Nyquist plot of API 5L X52 in acidic media with various inhibitor concentration and (a) 0 min, (b) 30 min, and (c) 60 min of immersion time.

As shown in **Figure 6**, the shape of the Nyquist plot remains the same in spite of addition of different concentrations of cashew leaves inhibitor which indicates similar corrosion mechanism of the API 5L X52 in acidic media in the absence and presence of the inhibitor [23]. Furthermore, the diameter of the semi-circle in the Nyquist plots showed an ascending trend as the inhibitor concentration and immersion time

increase. This means that cashew leaves inhibit the corrosion process of API 5L X52 in acidic media by boosting the impedance.

Figure 7 displays an equivalent circuit that fits the impedance data obtained. The equivalent circuit in Figure 7 includes solution resistance (R_u), polarization resistance (R_p) and the constant phase element (CPE). Element R_u represents to HCl solution, while element R_p represents to charge transfer resistance in the phase interface and is inversely proportional to the corrosion rate and surface area undergoing corrosion. The CPE corresponds to characterize “capacitance dispersion” related to the capacity of the surface area of a material with complex surface roughness, inhomogeneous reaction rate, nonuniform current distribution and corrosion products formed [41]. The Eq. (2) was represented the CPE impedance. All impedance parameters which corresponds to Figure 7 are listed in Table 4.

$$Z_{CPE}(\omega) = Q^{-1}(j\omega)^{-n} \tag{2}$$

where Q : Pseudocapacitance (Ωcm^{-2}), ω : the angular frequency ($\text{rad}\cdot\text{s}^{-1}$) and n : the CPE exponent ($-1 < n < 1$) [42].

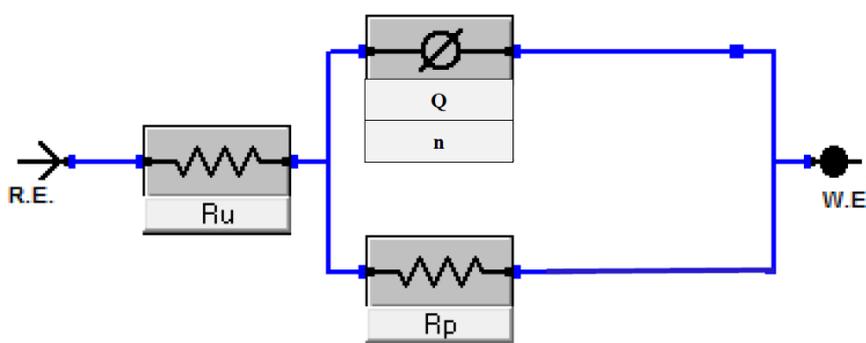


Figure 7 Equivalent circuit of API 5L X52 in absence and presence of inhibitor plot of API 5L X52 in acidic media with various inhibitor concentration and immersion time.

Table 4 EIS parameters.

Time (min)	$C_{inhibitor}$ (ppm)	R_u (Ω)	R_p (Ω)	Q (Ωcm^2)	n	X2	η (%)
0	0	1.191	18.16	63.88×10^{-6}	0.89	6.72×10^{-2}	-
	100	1.222	49.71	73.91×10^{-6}	0.85	2.23×10^{-2}	63.5
	200	1.262	57.89	80.84×10^{-6}	0.85	2.09×10^{-2}	68.6
	300	1.174	71.75	84.50×10^{-6}	0.79	3.41×10^{-2}	74.7
	400	1.185	106.1	48.92×10^{-6}	0.75	3.97×10^{-2}	82.9
	500	1.116	139.3	55.48×10^{-6}	0.71	2.68×10^{-1}	86.9
30	0	1.230	24.57	52.37×10^{-6}	0.87	9.60×10^{-2}	-
	100	1.322	67.41	50.37×10^{-6}	0.85	2.13×10^{-2}	63.5
	200	1.243	98.66	51.38×10^{-6}	0.83	6.52×10^{-2}	75.1
	300	0.899	148.4	48.70×10^{-6}	0.83	1.38×10^{-2}	83.4
	400	0.911	156.8	48.23×10^{-6}	0.79	2.57×10^{-1}	84.3
	500	1.096	207.7	44.51×10^{-6}	0.69	2.03×10^{-1}	88.2
60	0	1.019	31.45	184.7×10^{-6}	0.86	6.13×10^{-2}	-
	100	1.242	94.42	47.53×10^{-6}	0.85	3.87×10^{-1}	66.7
	200	0.593	153.2	46.53×10^{-6}	0.81	1.73×10^{-1}	79.5

Time (min)	$C_{\text{inhibitor}}$ (ppm)	R_u (Ω)	R_p (Ω)	Q (Ωcm^2)	n	X_2	η (%)
	300	0.657	188.7	44.74×10^{-6}	0.80	1.21×10^{-1}	83.3
	400	0.849	253.2	33.96×10^{-6}	0.80	6.37×10^{-2}	87.6
	500	1.066	298.5	31.40×10^{-6}	0.71	2.18×10^{-1}	89.5

According to **Table 4**, the polarization resistance (R_p) increases as the concentration of the inhibitor increased. On the other hand, the pseudocapacitance (Q) decreases with increasing the concentration of inhibitor. The downward trend of pseudocapacitance (Q) as an increase in inhibitor concentration could also be correlated to the reduce in local dielectric constant arising from the replacement of water molecules on the sample surface by active component of cashew leaves extract [30]. The CPE exponent (n) also related to the corrosion products formed on the API 5L surface in presence of HCl that can be caused by the formation of iron oxides with a porous nature and non-protective properties. Therefore “ n ” value in HCl without cashew leaves extract is greater than with the presence of cashew leaves extract.

The average value of solution resistance (R_u) with 0, 30 and 60 min of immersion time are 1.191, 1.116 and 0.9 ohm, respectively. This result indicated that ionic capacitance of the different solutions is very similar. Thus, the solution resistance does not depend on variations in inhibitor concentration and immersion time. The polarization resistance values (R_p) attained for the different concentration and immersion time contribute to determine the aggressiveness of the acid solutions. The inhibition behavior of cashew leaves extract is observed (increasing the R_p value) which can be related to competition between the electroactive species and which can be related to competition between the electroactive species and the cashew leaves extract adsorption at the active sites of the API 5L surface [43]. This EIS result is in agreement with the Tafel polarization result that cashew leaves extract adsorb to the steel surface to form the passive layer to inhibit the corrosion process of API 5L X52 in acidic media.

Adsorption mechanism of cashew leaves inhibitor

The adsorption mechanism of cashew leaves inhibitor may be explained on the basis of isothermal adsorption. Isothermal adsorption determine the interactions that occur between the inhibitor molecule and the steel surface [7]. Surface coverage (θ) data obtained from the inhibitor efficiency were used to characterize the adsorption process of cashew leaves inhibitor. The data were fitted to Langmuir adsorption isotherm by making a graph as displayed on **Figure 8**.

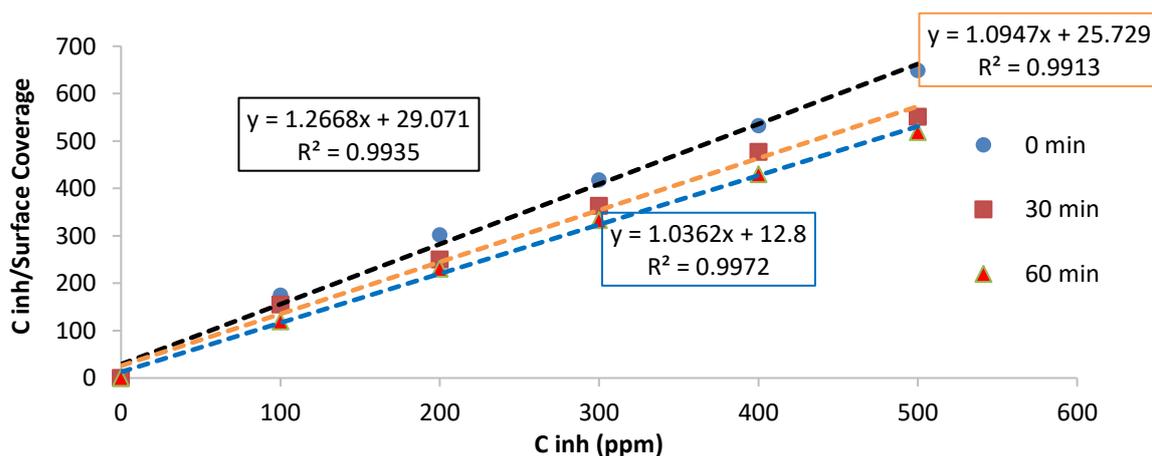


Figure 8 Langmuir adsorption isotherm of cashew leaves inhibitor fitting calibration data of TPC test.

Most calculations of R^2 are close to 1 according to **Figure 8**. This behavior indicate that the adsorption of cashew leaves extract inhibitors will undergo in a monolayer in accordance with the Langmuir adsorption process [2]. Thermodynamic parameters of cashew leaves inhibitor at ambient temperatures are presented in **Table 5**. K_{ads} value in **Table 5** obtained from intercept of linear equation in **Figure 8** according to Langmuir Eq. (3). $\Delta G^{\circ}_{\text{ads}}$ calculated from Eq. (4) [2].

$$\frac{c}{\theta} = y C + \frac{1}{K_{ads}} \quad (3)$$

$$\Delta G_{ads}^0 = -RT \ln(10^6 K_{ads}) \quad (4)$$

where R = 8.314, T = 298 K and K_{ads} is the equilibrium constant of the adsorption-desorption process.

Table 5 Thermodynamic parameters of cashew leaves inhibitor.

Time (min)	T (K)	K_{ads} (L/mol)	ΔG_{ads}^0 (kJ/mol)
0	298	0.0344	-25.88
30	298	0.0389	-26.18
60	298	0.0781	-27.91

It is clear from **Table 5**, K_{ads} value increase with the time because adsorption of cashew leaves inhibitor gets bigger with increasing the immersion time. Moreover, ΔG_{ads}^0 have negative value. The negative value of ΔG_{ads}^0 attribute to the spontaneous reaction of the adsorption process. Generally, ΔG_{ads}^0 value around -20 kJ/mol indicates physisorption process. While, ΔG_{ads}^0 value equal or less than -40 kJ/mol indicates charge sharing of organic species on the metal surface to form a coordinate covalent bond, termed as chemisorption sprcess [32]. In this present work, the values of ΔG_{ads}^0 are purely physisorption. Hence, cashew leaves inhibitor may be adsorbed through the donor (inhibitor) - acceptor (steel) interactions between the p electrons and the unshared electron pairs of the heteroatoms to make a bond with vacant d-orbitals of the metal surface [44].

Cashew leaves extract have the potential to be used as an eco-friendly inhibitor because they have a negative ΔG_{ads}^0 value that indicates the feasibility of adsorption and the stability of the passive film such as White tea extract [15]. Furthermore, the inhibitor efficiency of cashew leaves extract (89.5 %) are greater than white tea extract (85 %) and cashew extract (80.5 %) [33].

Conclusions

On considering the data obtained using classical electrochemical measurements, FTIR, TPC test, phytochemical screening and complementary adsorption studies, it can be concluded that cashew leaves extract was a very good green corrosion inhibitor for API 5L X52 in acidic medium. The corrosion rates of API 5L X52 decreased with increasing concentrations of cashew leaves extract and retention time. Cashew leaves inhibitor has an optimum efficiency of 89.5 %. The cashew leaves extract contain polyphenol, tannin, flavonoid and alkaloid which are the active corrosion inhibiting agents. Furthermore, the adsorption mechanism of cashew leaves inhibitor obeys the Langmuir adsorption isotherm. Regarding to the Langmuir isotherm, the cashew leaves extract will adsorp on the steel surface based on physisorption and form a monolayer of protective film that hind the interaction between steel surface and acidic medium.

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