

Jatropha Curcas as a Corrosion Inhibitor for API 5L-X65 Steel under Sand Deposit in CO₂ and Aerated Oilfield Environments

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ABSTRACT

Jatropha curcas leaves extract was used in this research as an inhibitor to protect a pipeline steel in static CO₂ and O₂ environments. 100, 200, 300 and 400 ppm of the inhibitor were considered at 298 K and 323 K. The corrosion mechanisms of the API 5L-X65 steel buried under sand deposit were subsequently analysed through potentiodynamic polarization curves and adsorption isotherms. It was found that Jatropha curcas improved the corrosion properties of the alloy at both temperatures significantly at a concentration of 400 ppm reducing the corrosion current density from 103.73 $\mu\text{A}/\text{cm}^2$ to 3.48 $\mu\text{A}/\text{cm}^2$ for the CO₂ saturated environment at 298 K. However, the corrosion inhibition was more significant at 298 K in the CO₂ environment and was more significant at 323 K in the O₂ environment. The result of the adsorption isotherms showed that the adsorption mechanisms of the extracts on the surface of API 5L-X65 were due to Freundlich and Langmuir isotherm.

Introduction

One of the industries where corrosion raises major economic and safety concerns is the oil and gas industry. This is because crude oil and natural gas carry various high-impurity products which are inherently corrosive (Paul *et al.*, 2014). Some of the corrosive species in oil and gas pipelines are CO₂, H₂S, O₂. These impurities enhance most corrosion degradation in oil and gas wells and pipelines (Popoola *et al.*, 2013), hence the continual effort at controlling them effectively.

Under-deposit corrosion occurs when solids like sands and corrosion products (scales) are found at the bottom of pipelines forming layers of solids. Accumulation of such sand deposits in pipelines is a major problem of inhibitor efficiency in oilfield

pipelines (Huang *et al.*, 2010). This is due to the adsorption of the inhibitors by the sand deposits thereby causing a barrier between the inhibitor and the carbon steel. Such sand weathered from reservoir rocks with low strength and the presence of such sand may also result in corrosion under sand beds (Salama, 2000). Under-deposit corrosion causes galvanic corrosion due to the sand deposition on part of the steel surface (Pedersen *et al.*, 2008). The steel area covered with deposit becomes anode and the adjacent area without deposit becomes cathode of the galvanic cell. The potential difference between the anode and the cathode is said to be the driving force for localized corrosion (Han *et al.*, 2008; Nyborg and Foss, 2011).

CO₂ corrosion is a major form of corrosion in the oil and gas industry (Popoola *et al.*, 2013). The corrosive action of CO₂ is assisted by the presence of moisture because water, when mixed with CO₂, forms carbonic acid which is acidic and corrosive (George and Nes, 2007). Among the factors that influence it include temperature, pH value and composition of the aqueous stream, presence of non-aqueous phases, flow condition, and metal characteristics (Popoola *et al.*, 2013). Oxygen ingress takes place in the well fluids through leaking pump seals, casing, and process vents and open hatches (Popoola *et al.*, 2013) causing the inner part of the pipelines to corrode since oxygen is a strong oxidant which reacts with carbon steel. Its continual intake increases the corrosive effects of H₂S and CO₂ gases (Popoola *et al.*, 2013) and this makes its degradation control crucial. Among the methods of corrosion control, use of inhibitors is very popular due to the ease of application (Kamal and Sethuraman, 2012). Mostly, the heterocyclic compounds containing O, S and N as heteroatoms serve as good inhibitors for corrosion (Rani and Basu, 2012) which are usually added in low concentrations (Dariva and Galio, 2014).

Many researchers have worked with both synthetic and natural inhibitors. Kadhum *et al.*, 2014 carried out a research on the inhibitive efficiency of New Coumarin on mild steel corrosion in hydrochloric acid solution (Kadhun *et al.*, 2014). Also, Kamal and Sethuraman (2012) studied the effectiveness of *Spirulina platensis* as a green inhibitor for mild steels. As environmental concerns are paramount in the field of engineering, the use of green inhibitors has been on the rise because of their eco-friendliness (Peter and Sanjay, 2015). They are the most widely used in the petroleum refining processes because of their ability to form a protective layer on the metal surface in media with high hydrocarbon content (Malik *et al.*, 2011) and they are very cheap to come by. Some of the inhibitor extracts that have been used are gotten from plants such as *Mentha pulegium*,

Simmondsia chinensis, *Datura metel*, *Ammi visnaga* and *Jatropha curcas* (*J. curcas*). This research, however, focuses on the use of *J. curcas* because it can grow on almost any terrain (Choksi *et al.*, 2014), hence its ease of access.

The oil from the seeds of *J. curcas* is used to make biodiesel and jet fuels (Ofor and Nwufu, 2011). This is the major reason for its cultivation, but its use as a corrosion inhibitor is on the rise. Its corrosion inhibition efficiency has been researched on metals such as copper and its alloys (Parameswaren *et al.*, 2013; Rani and Selvaraj, 2011), and mild steel (Omotoyinbo *et al.*, 2013; Odusote and Ajayi, 2013) in different environments. It has been proven to be a good corrosion inhibitor in the researched environments and temperatures. However, the aim of this study is to produce corrosion inhibitor from *J. curcas* leaves extract with a view of determining the effectiveness of its corrosion inhibition on mild steel under sand deposit in sweet and aerated oilfield environments at temperatures of 298 K and 323 K.

2. Experimental Procedure

2.1. Preparation of *Jatropha Curcas* sample

The *Jatropha curcas* leaves were sourced from the Federal University of Technology, Akure, Nigeria. The chemical composition is based on phytoconstituents screening as follows: a class of glycosides comprising saponnins in the majority, followed by tannins, cardioactive and flavonoids groups in company of small amounts of alkanoids, calcium oxalate, and terpenoids among others (Ahirrao *et al.*, 2011; Jide-Ojo *et al.*, 2013). The leaves were washed, cut into pieces and dried in air for few days and then grinded and sieved into powdery form. 10 g each of the powder was put into flat bottom flask containing 100 ml of ethanol. The resulting solution was left for 72 hours before it was carefully filtered. The stock solution was prepared from the filtrate and then used to make solutions of 100, 200, 300, and 400 ppm concentrations of the green inhibitor.

2.2. Specimen preparation

Mild steel X65 specimens with compositions C = 0.154%, Si = 0.45%, Mn = 1.357%, P = 0.023%, S = 0.014%, Cr = 0.061, Cu = 0.001, Ni = 0.022, and the remaining % of Fe were chosen for this research. A 25 mm diameter specimen was used for the electrochemical studies. An electrical wire was glued to one face of each specimen with conductive aluminium tape and then mounted into epoxy resin. Prior to measurements, the steel samples were wet-ground with SiC grit sizes ranging from 240 to 1200. This was followed by polishing using diamond suspensions of 15 μm . The sample was then degreased with acetone, washed in a stream of water and dried with air.

2.3 Silica sand deposit

Silica sand used was sourced from seashore, washed with water to remove the chloride content and then dried. Samples were buried under the sand particles to simulate sand at the 6 O'clock of a pipeline.

2.4 Brine used for the research

All the experiments in this study were simulated in standard brine solutions comprising of 3.5 wt. % sodium chloride with corrosion inhibitor added in required concentrations. For the sweet environment, the test solutions were saturated with CO₂ at atmospheric pressure for 2 hours prior to the experiments. The measured initial pH was approximately 4.7. This CO₂-saturated test solution was then transferred to the test cell and was continuously purged throughout the test period with CO₂. This procedure ensured the removal of oxygen and to sustain dissolved CO₂ concentration in the test solution. There was no significant change in final pH value measured at the end of the experiments. For the aerated solution the 3.5 wt. % sodium chloride solution was left as prepared without any seal to ensure ingress of oxygen. The pH of the aerated solution was 6.5. In this work, tests were conducted at two temperatures of 298 and 323 K with a view to

preventing scales (FeCO₃) formation which can obstruct the corrosion measurements.

2.5 Electrochemical Measurement

Tafel polarisation tests were conducted using AUTOLAB PGSTAT 204N with Ag/AgCl reference electrode and a platinum counter electrode. The working electrode was buried under the silica sand and the tip of the counter electrode was also buried at the same depth. The surfaces of the specimen was polarized from -250 mV below OCP to +250 mV above OCP at a scan rate of 0.25 mV/s.

3. Results and discussion

Results in this study are presented and discussed in this order: Tafel plots measurements to analyse the corrosion behaviour in blank and inhibited environments, open circuit potential was employed to evaluate the inhibition mode, the inhibitor performance in the different environments were evaluated by inhibition efficiency. Lastly, the mechanisms governing the inhibition were determined by adsorption isotherm fittings while possible inhibition mechanism was proposed.

3.1 Tafel plot results for CO₂ environment

From the Tafel plots of the mild steel in a CO₂ environment at 298 K and 323 K shown in Figures 1(a) and (b), it could be seen that the anodic inhibition is predominant at 298 K and 323 K. The anodic inhibition experienced signifies that the inhibition process is through adsorption as corroborated by the ΔG_{ads} values (Tables 4).

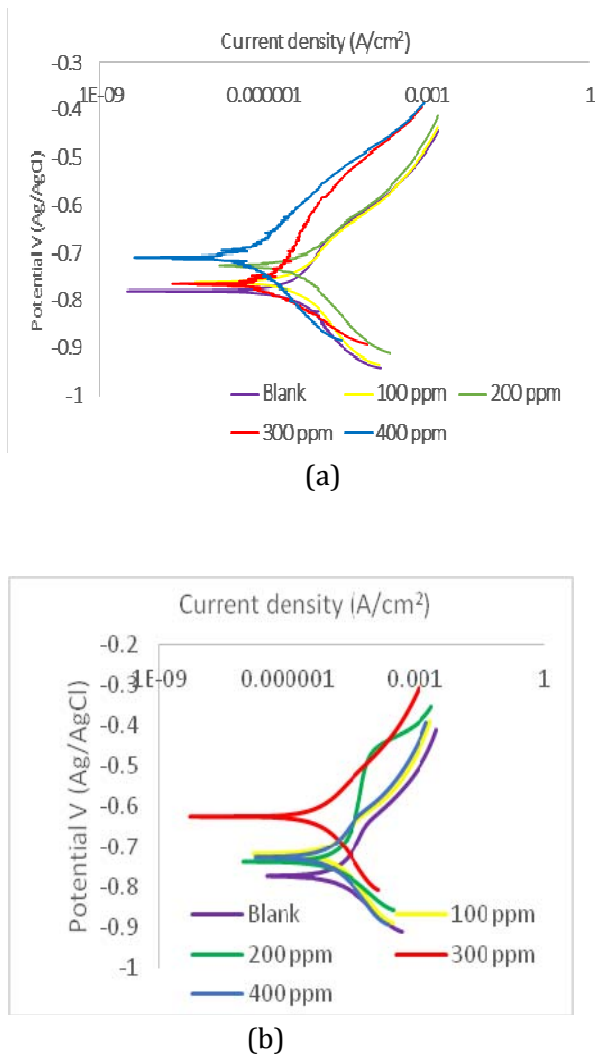


Figure 1: Tafel plot for the mild steel (buried under silica sand) at different concentrations of *J. curcas* in CO₂ at 298 K (a) and at 323 K (b)

3.2 Tafel plot results for O₂ environment

From the Tafel plots of the mild steel in aerated (O₂) environments at 298 K and 323 K shown in Figure 2, it could be seen that the anodic inhibition is also predominant in the O₂ environment at 298 K (Figures 2a) and a mixed mode inhibition is seen in the O₂ environment at 323 K (Figure 2b). The mixed mode process (Figure 2b) probably indicates that the inhibitor merely blocks the reaction sites of the mild steel surface and changes the mechanism of metal dissolution (anodic) and/or oxygen reduction/hydrogen evolution/carbonic reduction (cathodic) reaction (Behahmed *et al.*, 2012). It is expected that at 323 K less oxygen is dissolved in

the solution (Figure 2b). The cathodic reduction of oxygen is thus suppressed and hence a lower corrosion current is recorded compared to 298 K in the aerated salt solution.

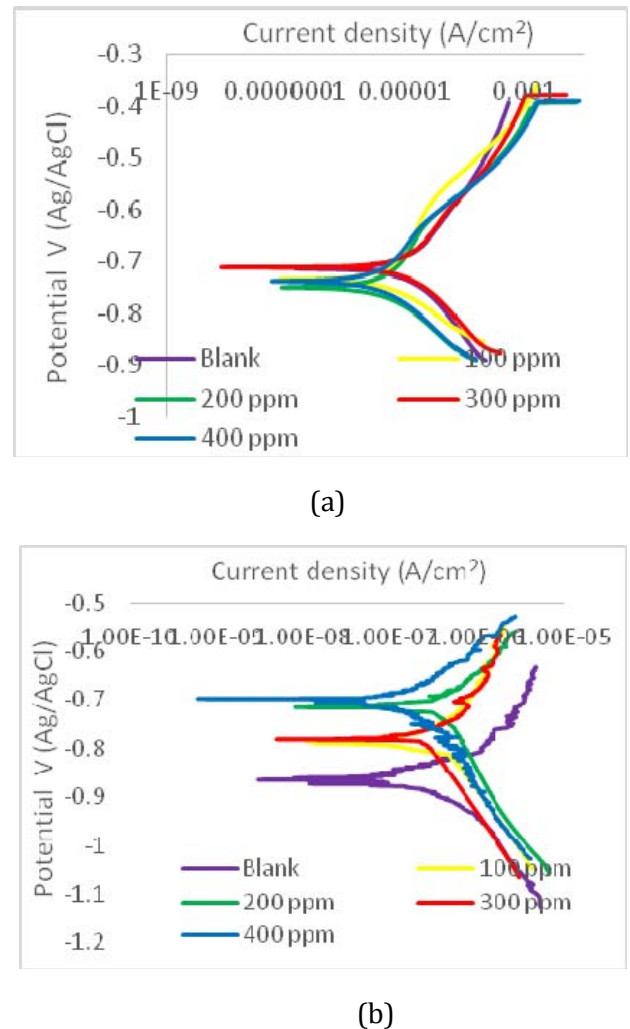
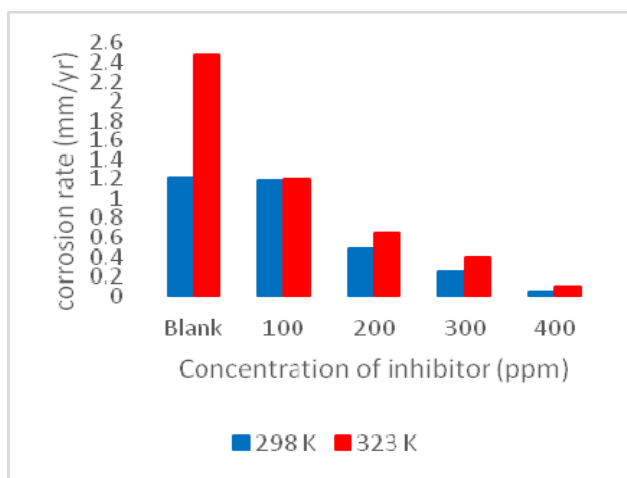


Figure 2: Tafel plot for the mild steel (buried under silica sand) at different concentrations of *J. curcas* in O₂ at 298 K (a) and at 323 K (b)

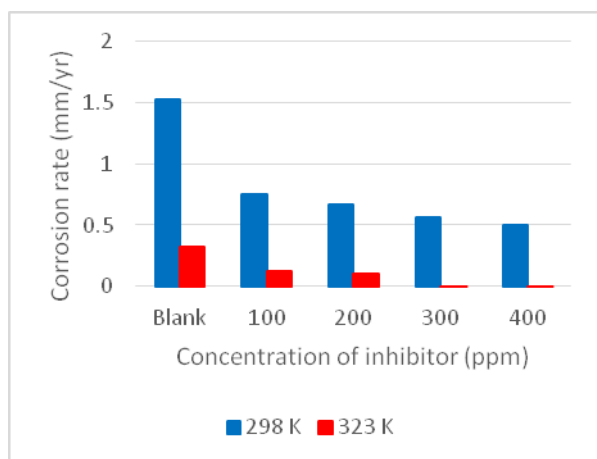
3.3 Corrosion rate of the mild steel (buried under silica sand) in both environments

Values of corrosion current density and hence corrosion rate in the aerated salt solution is lower at higher temperatures as against the values for the CO₂ saturated salt solution (Table 1). In essence, as expected of oxygen corrosion that is driven mostly by oxygen reduction; higher temperature favours lower corrosion rate as

lower oxygen is available at elevated temperature (Figure 1b and Table 1). Initial corrosion susceptibility of the alloy is higher in the aerated solution at 298 K (Figure 3b) as expected. So also, the protection offer by the inhibitor is not as effective as what is obtainable in the CO₂ environment at the same temperature. Also, though the corrosion rate of the alloy at high temperature in aerated solution is low, the inhibition protection offer is also more effective compared to 298 K.



(a)



(b)

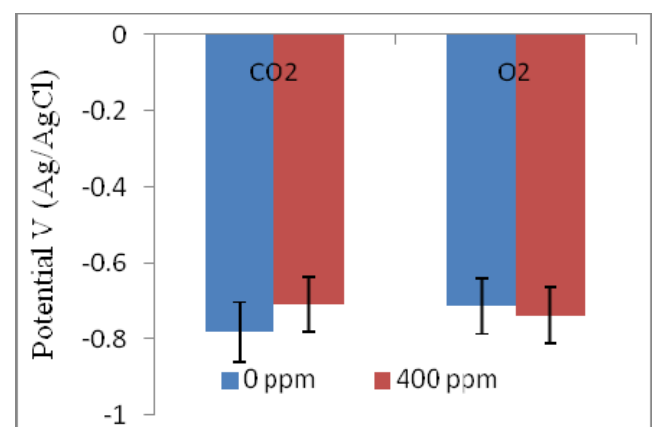
Figure 3: Corrosion rate on the mild steel (buried under silica sand) at different inhibitor dosing in a) CO₂ and b) aerated (O₂) environments

Table 1: Corrosion current density of the mild steel (buried under silica sand), i ($\mu\text{A}/\text{cm}^2$) values for CO₂ and aerated (O₂) environments

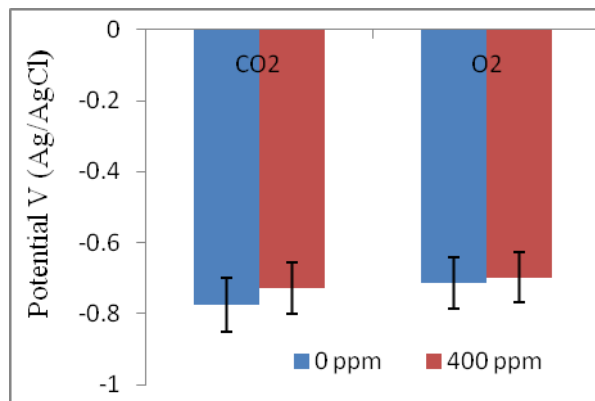
S/ N	Concentration of inhibitor (ppm)	Current density ($\mu\text{A}/\text{cm}^2$)			
		CO ₂		O ₂	
		298K	323K	298K	323K
1	0	103.72	210.76	129.78	27.36
2	100	100.76	101.82	63.72	10.36
3	200	41.56	55.04	57.26	9.13
4	300	21.57	33.97	47.99	1.52
5	400	3.48	8.14	42.34	0.27

3.4 Open circuit potential (OCP)

The open circuit potential elucidates the inhibitor characteristics vis-a-vis the types and it must be appreciated that this particular methodology does provide a conclusive inhibition mechanism like electrochemical impedance spectroscopy. In this study, the open circuit potential of the mild steel buried under silica sand without the addition of inhibitor (blank) and 400 ppm inhibited solutions were evaluated as these were the two extremes having the highest and lowest corrosion rates respectively, with the results as presented in Figure 4.



(a)



(b)

Figure 4: The open circuit potential for the mild steel (buried under silica sand) in both sweet (CO₂) and aerated (O₂) environments (a) 298 K and (b) 323 K

The process fluid saturated with CO₂ at 298 K have OCP of -780 mV (blank) and -709 mV (400 ppm); it is assumed that the *J. curcas* inhibits the mild steel in CO₂ fluid (-709 mV) more by suppressing the anodic dissolution probably through formation of passivating films. The other process stream exposed to oxygen at same temperature (298 K) on the other hand behaves cathodically by either slowing down the cathodic reactions or preventing the corrosion reactions by forming selective precipitates on the cathodic areas. The former trend was also observed for both media (CO₂ and O₂) at 323 K whereby according to Pourbaix diagram, the OCP measurement moved in a more noble direction denoting suppression of anodic reactions as the governing mechanisms. For example, in an aerated system the OCP for the mild steel buried under silica sand was -714 mV while the samples inhibited with 400 ppm has -698 mV. Notwithstanding the operating temperature and the environment, it seems that the *J. curcas* is a mixed-type inhibitor in that the maximum shift in the E_{corr} with and without the inhibitor was -71 mV for sweet system at 298 K and the least occurred for aerated environment having -16 mV at 323 K. This assertion was in line with previous studies that referred to all inhibitors when added into a system having less than ± 85 mV in both

directions as mixed-type inhibitors (Li *et al.*, 2008; Ferreira *et al.*, 2004). Notably, based on the chemical composition characterization, it is known that the inhibitor is a class of organic compounds which are often adjudged as mixed or adsorption inhibitors. According to literature, these types of inhibitors confer protection on the mild steel via three possible ways: physisorption, chemisorption, and film formations (Ebenso *et al.*, 2008; Ahamad *et al.*, 2010). The mode of inhibition protection mechanism of *Jatropha Curcas* in the studied environments will be further probed using adsorption isotherms and the resultant thermodynamic parameters.

3.5 Inhibition Efficiency for the mild steel buried under silica sand

The inhibition efficiency was obtained from the corrosion current density at different concentrations of inhibitor at temperatures 298 K and 323 K in CO₂ and O₂ environments for the mild steel buried under silica sand. The variation in inhibition efficiency with concentration of the *J. curcas* is shown in Table 2.

Table 2: Effect of *Jatropha curcas* on mild steel buried under silica sand in both CO₂ and aerated media

S / N	Concentration of inhibitor (ppm)	Inhibitor Efficiency %			
		(CO ₂ environment)		(O ₂ environment)	
		298 K	323 K	298 K	323 K
1	100	44.85	51.68	50.90	62.13
2	200	59.93	73.88	55.87	66.64
3	300	79.20	83.88	63.02	88.36
4	400	96.64	96.14	67.37	97.90

The inhibitor efficiencies (IE) were calculated from the corrosion current density according to equation (1)

$$IE (\%) = \frac{i_o - i_{inh}}{i_o} \times 100 \quad (1)$$

Where *i_o* and *i_{inh}* are the corrosion current density of the mild steel in the presence and absence of inhibitor, respectively.

From the values of Table 2, it is clear that the *J. curcas* effectively inhibited the corrosion rate of the steel in CO₂ at 298 K and 323 K at a concentration of 400 ppm in CO₂ environment. In O₂ environment, the green inhibitor performed excellently at 323 K with the same concentration. It performed well at a concentration of 300 ppm at both temperatures in CO₂ but only at the higher temperature in O₂. Overall, it could be said that the green inhibitor performed better in CO₂ environment than in O₂ because the efficiencies in CO₂ were better at lower concentrations of the inhibitor. This is thought to be due to the higher aggressiveness of the oxygen environment compared with CO₂ environment.

Inhibition efficiency was found to increase with the concentration of inhibitor (Table 2), with maximum IE at a concentration of 400 ppm in both environments. The increase in inhibition efficiency is due to the increase in the number of constituent molecules of *J. curcas* adsorbed on the metal surface at higher concentrations (Rani and Basu, 2012). It is suspected that as the inhibitor concentration increases, there is higher tendency for the active sites of the metal to be protected by the inhibitor molecules. Correlating the open circuit potential with the inhibition efficiency showed clearly that the only instance where the inhibitor suppresses the cathodic reactions was the condition at which it has the least IE% of about 67%. This happens to be the only environment that *Jatropha curcas* extracts cannot be recommended for industrial applications as it does not meet with the 90% as advocated by the oil and gas industry (NACE, 1995).

3.6 Adsorption Isotherms

According to Durnie *et al.*, (2005) the structural-chemical properties and adsorption characteristics of inhibitor molecule is pre-eminent in selecting an appropriate corrosion inhibitor for a given environment. In spite of the recent upsurge in the study relating theory development that affects design and selection of corrosion inhibitor (Durnie *et al.*, 2001). The

adsorption isotherms and models were powerful tools for determining inhibitor performance on the basis of thermodynamic parameters and calculations (Barker, 2012). In this study, the corrosion data revealed that increasing the inhibitor concentration, increases the IE% and also the surface coverage (θ).

The *Jatropha curcas* inhibitor adsorbs pretty well on the metal surface based on the data obtained and after evaluating numerous isotherms; it was observed that the experimental data fits well with Freundlich isotherm at 298 K for CO₂ environment (Figure 5a). The import of this is that the plant extracts and the metal substrate adsorption is regarded as non-ideal, reversible, and that it is not restricted to monolayer formation. This implies that it can form multilayer adsorption with non-uniform distribution and it is characterized with low inhibitor concentration (Ige, 2013).

Meanwhile it was Langmuir isotherm that gives the best surface coverage with samples exposed to CO₂ environment at 323 K (Figure 5b). Also, theoretical fitting of Langmuir isotherm were observed to describe the nature of adsorption for aerated conditions at both temperatures of 287 and 323 K (Figure 6). The linearized equation for Langmuir isotherm is given as:

$$\frac{C}{\theta} = \frac{1}{K} + C \quad (3)$$

where, C = plant extract concentration (mg/L), θ = surface coverage, and K_{ads} = equilibrium constant of the adsorption process (l/mg).

The values for ΔH and K_{ads} which were deduced from the graph were used to get the Gibb's free energies of the adsorption process, ΔG_{ads} (Table 4) at both temperatures in each environment through equation (Rani and Basu, 2012) and the ΔS was calculated using equation 5:

$$\Delta G_{ads} = -RT \ln(55.5K_{ads}) \quad (4)$$

$$\Delta S^\circ = \frac{\Delta H^\circ - \Delta G^\circ}{T} \quad (5)$$

The Langmuir isotherm theorises that there is no interaction between the adsorbed inhibitor constituents and the steel substrate. Equally, it assumes that each site of metal surface holds one adsorbed species of the plant extracts; hence, adsorbed water molecule on the metal surface is replaced with one inhibitor adsorbate via substitution process according to equation 6:

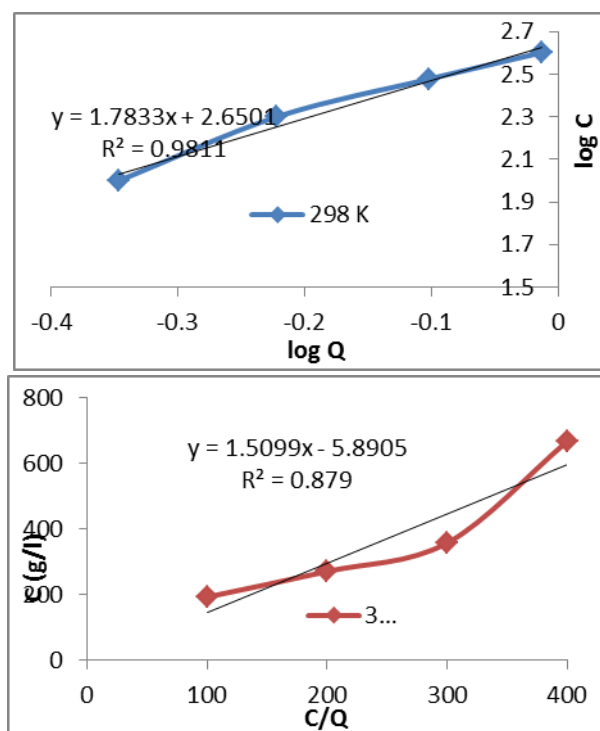
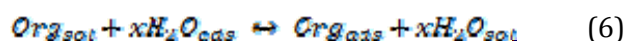


Fig 5: Freundlich isotherm of *J. curcas* in CO₂ environment at 298 K (a) and Langmuir isotherm of *J. curcas* in CO₂ environment at 323 K (b)

The thermodynamic data associated with the plant extract in the studied environments are presented in Table 3. The negativity associated with ΔH obtained in this study connotes exothermic reactions that suggest with the exception of samples exposed to CO₂ environment at 323 K (3.90 KJ/mol) which might favour endothermic reaction to a little extent due to the considerably low positive values. All these advocate that the reactions are physisorption or chemisorption in nature. An explanation was earlier offered that the presence of both heteroatoms (nitrogen and oxygen atoms) which

lead to coordinate bonds and aromatic rings suggested physisorption has been the prevailing adsorption mode (Ebenso *et al.*, 2008).

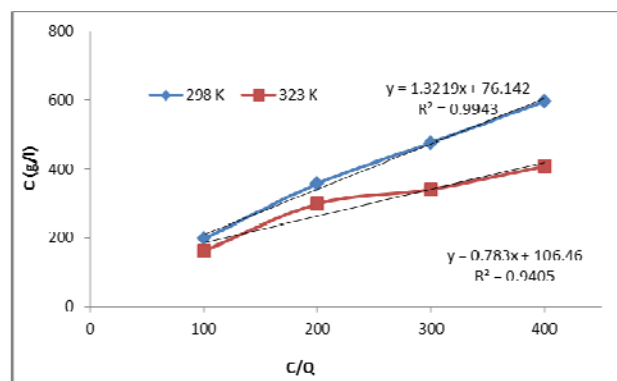


Figure 6: Langmuir isotherm of *J. curcas* in O₂ environment at 298 K and 323 K.

The adsorption strength of the *Jatropha curcas* leaves extract on the mild steel in sweet environments were not robust considering the equilibrium of adsorption process (ΔK_{ads}) which are closer to zero. Contrary behaviour was noticed in the aerated solutions, whereby the ΔK_{ads} were on the higher side and increases as the temperature increased.

The negative values of ΔG_{ads} in both the environments reflect a spontaneous adsorption of phytoconstituents of *J. curcas* on the surface of the mild steel (Rani and Basu, 2012; Ebenso *et al.*, 2008). Generally, the consensus was that the threshold value between chemical and physical adsorption process is -40 KJmol^{-1} ; and that values up to -20 KJmol^{-1} indicating physisorption and above -40 KJmol^{-1} suggesting chemisorption. From Table 4, it is obvious that the system under study is a physisorption process in that the ΔG is not up to -20 KJmol^{-1} which signified that the molecules of the green inhibitor are physisorbed through the electrostatic interactions between the charged molecules. The exemption is aerated environment at a temperature of 323 K having free energy value (-23.32 KJ/mol) which is characterised with mixed physisorption or chemisorption process. The values for ΔG_{ads} could also mean that the $-NH_2$ group of amino acids in *J. curcas* might

be protonated in the CO₂ and O₂ environments and be adsorbed over the metal surface through negatively charged acid anions (Ahamad *et al.*, 2010).

The entropy value as obtained from Table 3 revealed that oxygenated environment at 323 K temperature have the uppermost value (-0.35 J/molK)). On closer scrutiny of the entropy data, it was indicated that the formation of the *Jatropha curcas* adsorbed layer on the metal surface is favoured at 323 K compared with 298 K for oxygen-laden environment. This can be attributed to increase in the disorderliness of the system. Literature revealed that less than zero value of ΔS for a system is considered as an activation complex controlled corrosion process (Rani and Basu, 2012) and it can be further argued that the formed inhibitor layer hinders the release of hydrogen ions at the metal surface (Rani and Basu, 2012; Gunasekaran and Chauhan, 2004).

Table 3: Thermodynamic data for the adsorption process in CO₂ environment (a) and O₂ environment (b)

S/N	T (K)	ΔH (kJ/mol)	K _{ads} (mg ⁻¹)	ΔG _{ads} (kJ/mol)	ΔS _{ads} (J/mol/K)
1	298	-1.49	2.65	-12.37	0.04
2	323	3.90	-5.89	-15.55	0.06

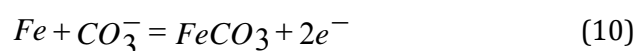
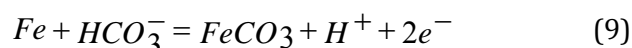
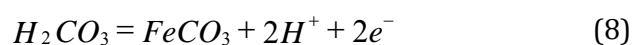
(a)

S/N	T (K)	ΔH (kJ/mol)	K _{ads} (mg ⁻¹)	ΔG _{ads} (kJ/mol)	ΔS _{ads} (J/mol/K)
1	298	-55.60	76.14	-19.44	-0.13
2	323	-135.96	106.46	-23.32	-0.35

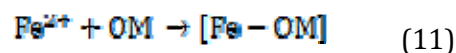
(b)

3.7 Inhibition mechanism

The governing mechanism for the inhibitor employed in this study is as proposed, wherein based on the fact that the *Jatropha curcas* leaves extract is suspected to have plant phytoconstituents having heteroatoms such as O, N, and S. This implied that it is organic in nature and as such will either adsorb or form film on the mild steel. Arising from OCP and IE data, it can be stated that the inhibitor though mixed-type, is expected to block the anodic areas more effectively by forming film. In the absence of comprehensive understanding of the principle of under-deposit corrosion (Huang *et al.*, 2010), the anodic electrochemical reactions for mild steel in CO₂ - containing solution are as follows (Linter and Burstein, 1999).



The dissolution of mild steel during the initial stage is the most prevalent and it causes the formation of Fe²⁺ ions. The plant extracts react with these ions to form organo-metal complex (Fe-OM) layer on the metal surface to suppress the iron dissolution according to the following equation (Gunasekaran and Chauhan, 2004; Victoria *et al.*, 2015).



This complex again reacts with carbonate ions in the process fluid to form equation 10 which is catalysed by the Fe-OM layer. Whereas in the presence of oxygen, it is suspected that there is formation of galvanic cells. Finally, despite the assertions that inhibition performance is problematic in under deposit corrosion, this study has provided a leeway in understanding the inhibitory behaviour of *Jatropha curcas* in the studied environments.

4. Conclusions

1. The efficiency of *Jatropha curcas* leave inhibitor extract increased with increase in concentration.
2. Inhibitor efficiencies of 96.64% and 96.10% for CO₂ and 67.37% and 97.89% for aerated environments were achieved at 25 °C and 50 °C respectively.
3. *Jatropha curcas* leaves extract was found to be effective as an inhibitor and can serve as corrosion inhibitor for mild steel under sand bed in CO₂ and O₂ environments.
4. *Jatropha curcas* leaves extract behaved more like a mixed-type inhibitor.
5. Adsorption of the extracts on the surface of API 5L-X65 is physisorption and consistent with both Freundlich and Langmuir isotherms.

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