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Inhibition of Steel Corrosion in 1M HCl by Jatropha Curcas oil

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Abstract

The effects of natural Jatropha curcas oil on the corrosion of steel in molar hydrochloric acid were studied via the measurements of weight loss, electrochemical and EIS polarisation. The results obtained revealed that Jatropha curcas oil reduced the rate of corrosion. The corrosion inhibition efficiency increased with the increase of inhibitor concentration. Potentiodynamic polarisation studies clearly revealed that the presence of the natural Jatropha curcas oil did not alter the mechanism of the hydrogen evolution reaction and acted a mixed inhibitor. The adsorption of Jatropha curcas oil on the steel surface in molar in hydrochloric acid obeys to the Frumkin adsorption isotherm model.

Keywords: Steel, acid, corrosion, inhibition, extraction, natural substance, Jatropha curcas oil.

I. Introduction

It is well-known that acids are used in many industrial operations like cleaning, pickling, descaling, etc. Because of their aggressiveness, inhibitors are used to stop or at least to delay the overall rate of metals dissolution. The most efficient inhibitors are organic compounds having and/or n bonds in their structures [1-5]. The inhibiting effect of an organic molecule, as a successful inhibitor, is mainly dependent on its ability to get on the metal surface which consists of the replacement of water molecules at the corroding interface.

Although the most effective natural inhibitors, the biological toxicity of these products is documented especially about their environmental harmful characteristics [6-9]. From the standpoint of safety, the development of non-toxic and effective inhibitors is of considerable interest. Nowadays, researches seem to retain many attempts and are focused on the use of natural products extracted from aromatic herbs, spices and medicinal plants. Their use as corrosion inhibitors has been preferred because of both economic and environmental goals. A survey in the literature shows that many works were conducted to examine some naturally occurring substances as corrosion inhibitors in various media [10-14].

Jatropha curcas is a non edible oil crop, which supports drought and which is capable to Lop on marginal land unsuitable for dry land agriculture. The plant presented in the picture 1, has many of the characteristics that put it as a potential candidate for the production of bio fuels or green fuel, less polluting than fossil fuels. The interest in this plant increases more and its expansion is accelerating. It was expected that each year during the next five and seven years, about 2 million hectares of Jatropha will be planted around the world. As part of this study on the development of wastewater from the city of Oujda as a source of irrigation and fertilization of Jatropha curcas (plant energy value), we obtained very interesting results, the most important is the improvement of about 30% of the oil content of the seeds of Jatropha curcas .

The oil extracted from its seeds possess several uses (soap production, lubricant, medicinal, etc.), but the most important and it is used as biofuels in diesel engines. The plant produces seeds containing inedible oil that can be converted to biodiesel, which can be used in the transportation and energy sectors. The detoxified cake by-product from oil extraction can be used for fish and animal feed, biogas, or as an organic fertilizer. The crop can be mechanically harvested, and oil yields are comparable or higher than soybean and rape seed without genetic improvement. Jatropha produces renewable energy in the form of biodiesel, which emits 80% less CO_2, 100% lower SO_2, and has a higher flash point than fossil diesel fuel. The Jatropha biodiesel industry currently is relatively minor; therefore, as it grows to a larger scale and the infrastructure is developed, the costs of producing and marketing Jatropha biodiesel may decline in the future.

Photo 1. the pictures of plants and grains of Jatropha curcas (irrigated with wastewater (harvesting December 2012)

The encouraging results obtained by extract of Jatropha curcas leaves and seed [15,16] as corrosion inhibitor of steel have incited us to test its oil toward C38 steel in hydrochloric acid media.

In the present investigation Jatropha curcas oil, a non-edible vegetable oil which has been considered as a potential alternative fuel for C.I. engines has been chosen to find out its suitability for use as fuel oil. Jatropha curcas is a large shrub or tree native to the American tropics but commonly found and utilized throughout most of the tropical and subtropical regions of the world. Several properties of the plant, including its hardness, rapid growth, easy propagation and wide ranging usefulness have resulted in its spread far beyond its original distribution. The Jatropha oil is a slow drying oil which is odourless and colourless when fresh but becomes yellow on standing. The oil content of Jatropha seed ranges from 30 to 50% by weight and the kernel itself ranges from 45 to 60%. The fatty acid composition of Jatropha classifies it as a linoleic or oleic acid type, which are unsaturated fatty acids. The fatty acid composition of Jatropha oil consists of myristic, palmitic, stearic, arachidic, oleic and linoleic acids. The seeds and oil are toxic due to the presence of cursive and curcasive. However, from the properties of this oil it is envisaged that the oil would be suitable as fuel oil. The oil compares well against other vegetable oils and more importantly to diesel itself in terms of its fuel rating per kilogram or hectare of oil produced. But the greatest difference between jatropha oil and diesel oil is viscosity. The high viscosity of curcas oil may contribute to the formation of carbon deposits in the engines, incomplete fuel combustion and results in reducing the life of an engine. But so far very few studies on the effect of Jatropha curcas oil on corrosion of steel have been carried out. Generally, the oil is used for diesel generators. It would be interesting to see its effect on the corrosion of metals such as steel widely used in industry. The study reported here were conducted using gravimetric, polarisation and EIS methods. The effect of concentration on the efficiency of corrosion inhibition was studied. Activation energy in the presence and the absence of inhibitor was also determined.

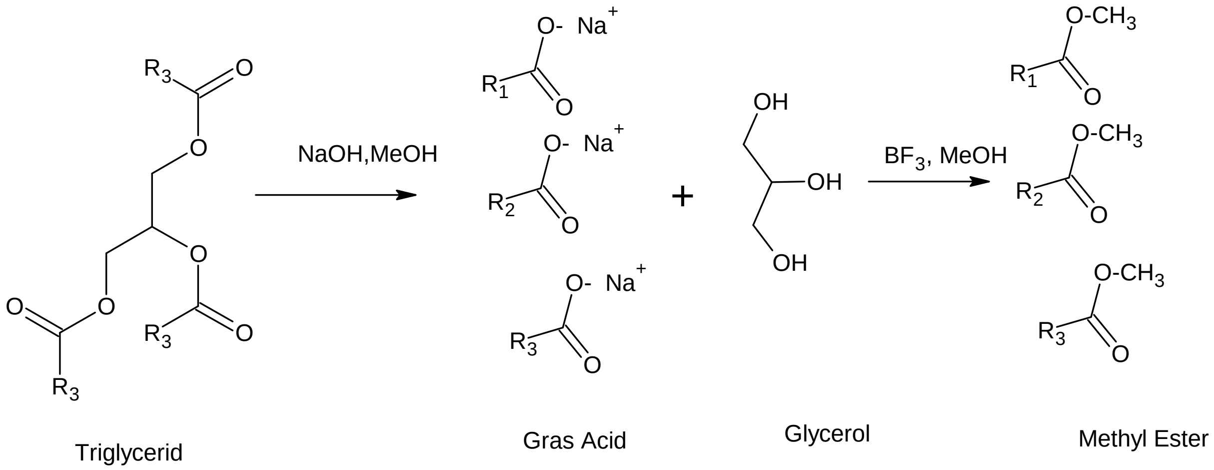
II. Experimental

II.1 Materials

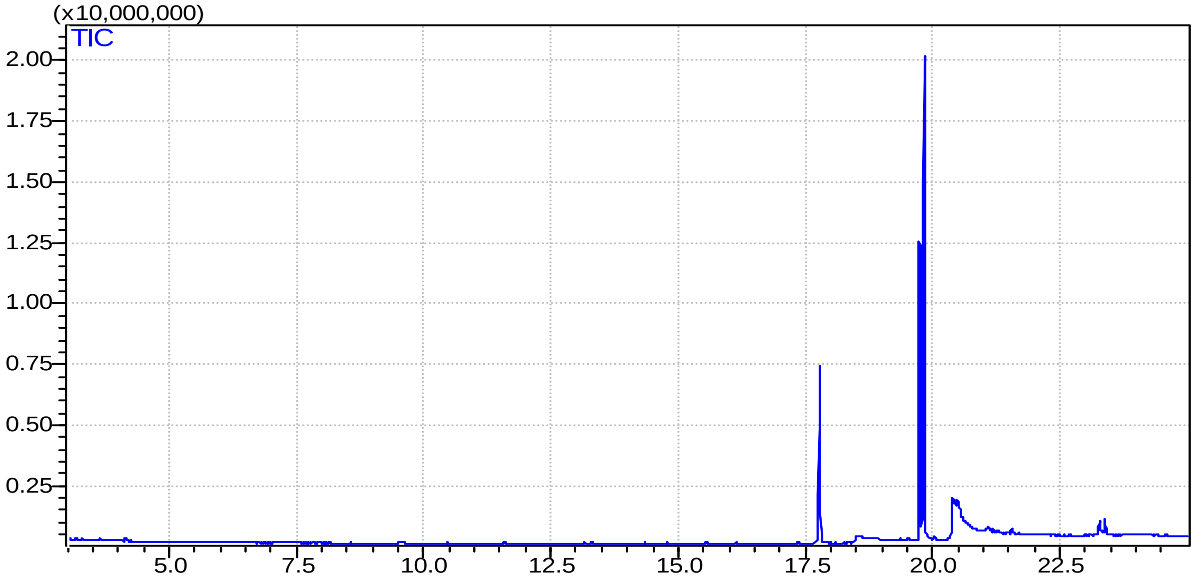
Steel panels used were composed of 0.09% P, 0.38% Si, 0.01% Al, 0.05% Mn, 0.21% C, 0.05% S and iron. Prior to experimental investigations, the steel panels were polished using different emery papers up to 1200 grade, washed thoroughly with bi-distilled water degreased with acetone and dried. The molar hydrochloric acid solution was prepared by dilution of Analytical Grade 37% HCl with bi-distilled water

In general, the amount of oil obtained from the seeds varies between 28 and 32% for Jatropha plants irrigated with wastewater and between 23-25% for controls whereas for almonds, the oil content is much more important than it is in irrigated with wastewater or in control plants. Indeed, the lipid yield almonds is 46 to 47% for plants irrigated with Jatropha wastewater, and 42% for controls. gifts is at the kernel that is the high oil content The oil extracted by Soxhlet solvent (hexane at 69 ° C) obtained in the second part of our study. Different oil samples will be used to determine the chemical composition of fatty acids

We chose that is corresponds to the oil extracted from the seed kernels of Jatropha curcas witnesses irrigated with fresh water (harvesting December 2012). The oils obtained can not be analyzed directly, they must be derived (derivatization reaction). The oil sample is firstly saponified by the action of a basic catalyst (NaOH), and then esterified with a solution of BF3 in excess methanol presented in the Schema 1. The fatty acid composition of the oils was determined by analysis of their methyl esters



Schema1: Derivatization reaction for the oil obtained by extraction of almond cookies Jatropha seeds irrigated with wastewater collected late 2012.



Spectre 1: The Chromatogram spectrum of the sample (almond cookies Jatropha seeds irrigated with wastewater collected late 2012).

The fatty acid composition of the oils was determined by analysis of their methyl esters. Quantification was made by gas chromatography coupled with mass spectrometry (GC / MS: shimadzu Japan QP 2010) equipped with a DB5 capillary column 30 m long and 0.25 m internal diameter, the film thickness is 0.25 micro meter. The column temperature is maintained at 80 ° C for 1 min, the temperature program is 10 ° C / min to 280 ° C held for 5 min. The temperature of the injector and the interface of the mass spectrometer are fixed at 250 ° C and that of the voltage detector with a SM 1.7 kV, the carrier gas is helium. The analysis was conducted in Electronic impact SCAN mode and the ion source temperature was maintained at 200 °C (spectre 1).

The oil used is extracted from the seed kernels of Jatropha curcas compound of saturated acids (palmitic acid (13.68%), stearic (22,06)) and unsaturated acids (oleic acid (20.46) and linoleic (43,80) whose structure is presented respectively in figure (1) and (2). The fatty acid composition is given in Table (1).

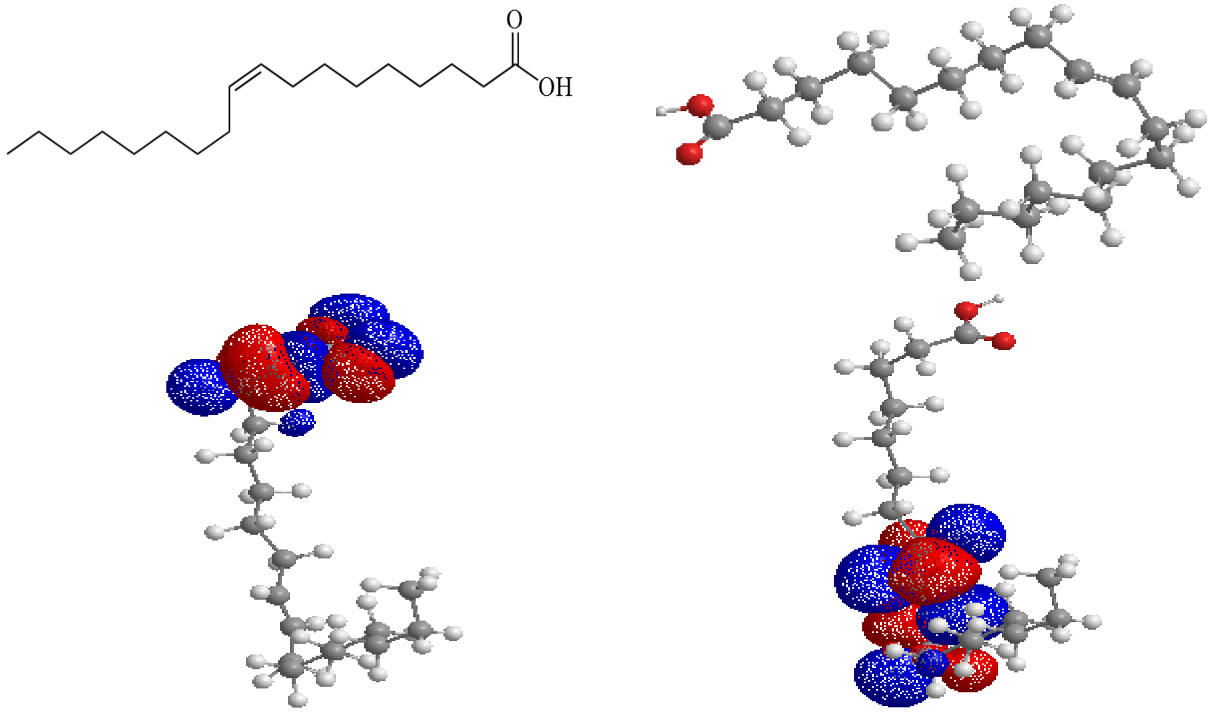


Figure 1. Oleic acid structure

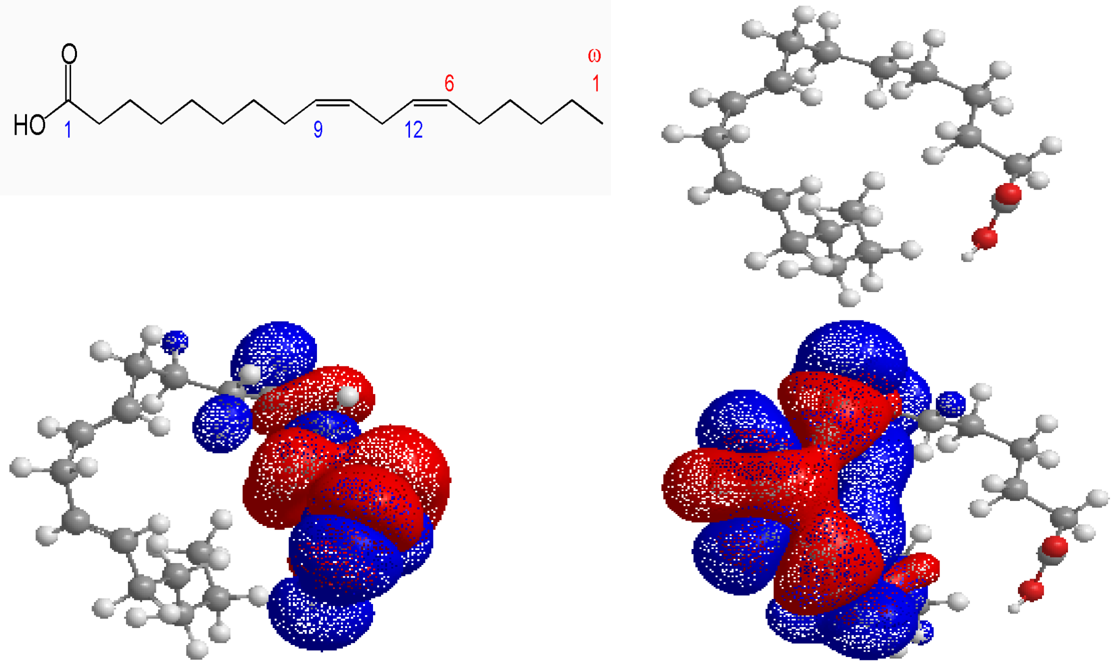


Figure 2. Linoleic acid structure

Table 1. Chemical composition of Jatropha curcas oil

Acid grass	Jatropha curcas oil (The weight of the seed kernels)
Ac. Palmitic (17,774 min)	13,68 %
Ac. Stearic (19,744 min)	22,06 %
Ac. Linolic (19,834 min)	43,80 %
Ac. Oleic (20,385 min)	20, 46 %

The essential oil used was obtained by steam distillation in water (DW) of the aerial part (stem and leaves) of Jatropha curcas, freshly cultivated from Oujda, Morocco. The oil obtained, Jatropha curcas oil also known as essence, was a pale Jatropha curcas liquid. Distillation converted the volatile liquid (the essential oils) into vapour which was condensed back into a liquid. This was the most popular and effective method in use today in producing essential oils.

In the process of manufacturing the essential oils using the method of water distillation, the botanic material was completely immersed in water and the still was brought to the boil. This method protected the oils extracted from overheating to a certain degree since the surrounding water acted as a barrier to excessive heat. It was clear from Figure 1 that Jatropha curcas oil is constituted by four products as majority components.

III.Methods

III.1. Gravimetric measurements

Gravimetric measurements were carried out in double walled glass cell equipped with a thermostatic cooling condenser. The solution volume was 100 cm^3. The steel panels used had a rectangular shape (2 cm × 2 cm × 0.05 cm: length × width × thickness).

III.2. Electrochemical and EIS measurements

Electrochemical measurements were carried out in a conventional three - electrode electrolysis cylindrical Pyrex glass cell. The temperature of the cell was controlled at 308 ± 0.5K. The working electrode (WE), in the form of disc cut from the steel panel, had a geometric area of 1 cm^2 and was embedded in polytetrafluoroethylene (PTFE). A saturated calomel electrode (SCE) and a platinum electrode were used as reference and auxiliary electrodes, respectively. The scan rate of the potentiostat model Voltalab PGZ 100 computer is a 20 mV/min. Before recording the polarisation curves, the steel electrode was polarised at -800 mV vs. SCE for 10 min. Potentials were scanned from –800 mV to anodic values. The test solution was de-aerated using pure nitrogen. Gas bubbling was maintained throughout the experiments.

Electrochemical impedance spectroscopy (EIS) was carried out with a Tacussel electrochemical system which included a digital potentiostat model Voltalab PGZ 100 computer at E_{corr} after immersion in solution without bubbling, the circular surface of steel exposing of 1cm^2 to the solution were used as WE. After the determination of steady-state current at a given potential, sine wave voltage (10 mV) peak to peak, at frequencies between 100 kHz and 10 mHz were superimposed on the rest potential. Computer programs automatically controlled the measurements performed at rest potentials after 30 min of exposure. The impedance diagrams are given in the Nyquist representation.

4. Results and discussion

4.1. Inhibition efficiency based on Weight loss

Table 2 shows the corrosion rates of steel panels in 1M HCl without Jatropha curcas oil (W^0_{corr}) and with various concentrations of Jatropha curcas oil (W_{corr}), determined at 308 K during 6 hours of immersion. The inhibition efficiency, E_w, was determined using the relation represented by Equation 1.

$$E_w\% = 100 \times \left(1 - \frac{W_{Corr}}{W^{\circ}_{Corr}}\right) \qquad (1)$$

In Equation 1, W_{corr} and $W°_{corr}$ are the corrosion rates of steel in 1M HCl solution with and without Jatropha curcas oil, respectively. The values of the degree of surface coverage (θ) have been obtained from Inhibition efficiency based on Weight loss for various concentrations of inhibitors. Here, θ can be given as equation .2.

$$\theta = \frac{\%E}{100} \qquad (2)$$

Table 2. Gravimetric results of the mild steel corrosion with and without addition of inhibitors studied at 308 K after 6 h of immersion in 1 M HCl

Inhibitor	Concentration g L^{-1}	W mg cm^{-2} h^{-1}	E_W %	θ
Blank	1M	0.1740	--	--
Inhibitors	0.125	0.1033	41	0.41
	0. 25	0.0859	51	0.51
	0.5	0.0149	91	0.91
	1	0.0009	99	0.99

It could be seen, from data given in Table 2, that the presence of Jatropha curcas oil reduced the corrosion rate of steel in HCl solution. Indeed, the inhibitory effect increased with the increase of inhibitor concentrations.

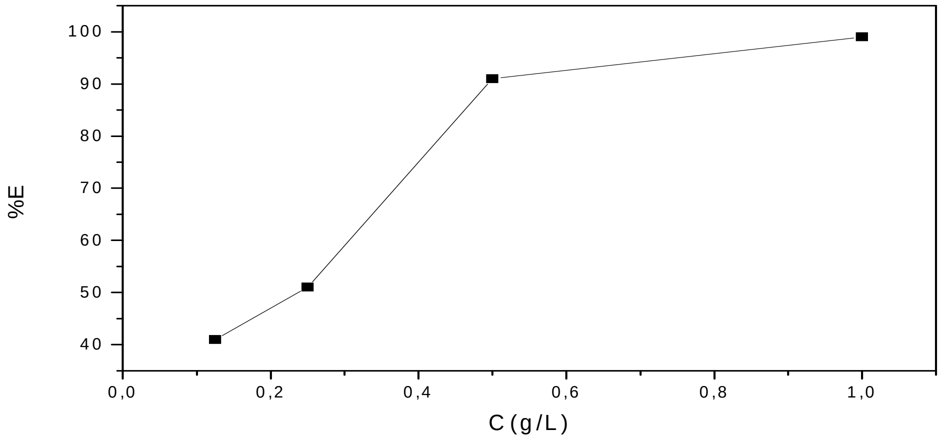


Figure 3. Variation of the of Jatropha curcas concentration with %E

The inhibition efficiency, E%, reached a maximum of 99% at 1 g/L of Jatropha curcas. The increase in inhibition efficiency observed at higher inhibitor concentration indicates that more inhibitor molecules are adsorbed on the metal surface thus providing wider surface coverage and the essential oil is acting as adsorption inhibitor figure 3. It could be concluded that Jatropha curcas oil was a good inhibitor of corrosion of steel in 1M HCl solution.

4.2. Inhibition efficiency based on electrochemical measurements

Polarisation behaviour of steel in 1M HCl in the presence and the absence of Jatropha curcas oil are shown in Figure 4. Table 3 gives the values of corrosion current (I_{corr}), corrosion potential (E_{corr}) and cathodic Tafel sJatropha curcas pe (b_c). The inhibition efficiency (E_I%) was calculated using the relation shown in Equation 3.

$$E_I(\%) = (1 - \frac{I_{corr}}{I^0_{corr}}).100 \qquad (3)$$

In Equation 2, I^0_{corr} and I_{corr} are the uninhibited and inhibited corrosion current densities, respectively, determined by extrapolation of the cathodic Tafel lines to corrosion potential.

The examination of Figure 4 and results reported in Table 3 shows that the addition of the natural substance decreases only cathodic current density. The decrease is more pronounced with the increase of the inhibitor concentration.

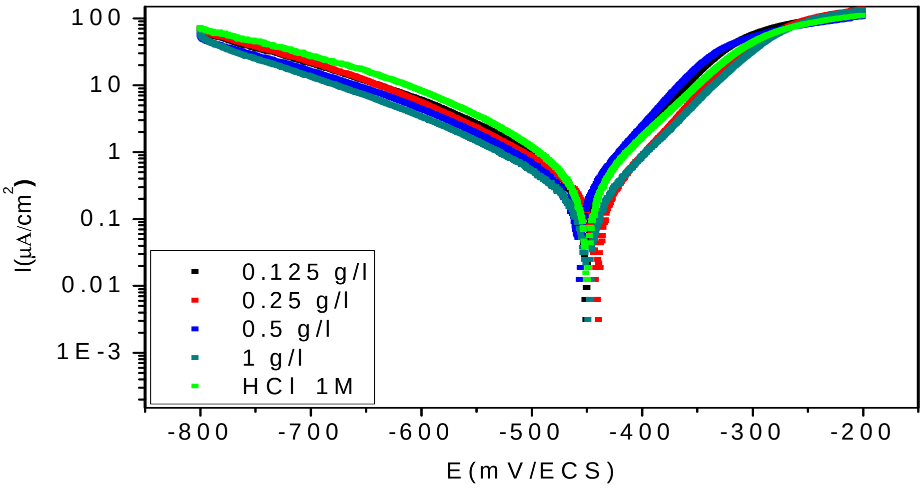


Figure 4. Polarisation Jatropha curcas of mild steel in 1M HCl for various concentrations of Jatropha curcas inhibitor

Table 3: Electrochemical parameters for mild steel in 1M HCl at various concentrations of different Jatropha curcas

Inhibitor	Concentration g L^{-1}	$-E_{cor}$ (V)	β_C (V/dec)	I_{cor} (mA/cm^2)	E %
Blank		0,439	0,138	1.1951	-
Inhibitors	0.125	0,461	0,189	0.5305	56
	0. 25	0,442	0,136	0.3654	69
	0.5	0,437	0,109	0.3465	71
	1	0,449	0,138	0.2778	77

Figure 4 indicates that the cathodic current-potential curves give rise to parallel Tafel lines, indicating that the hydrogen evolution reaction is under activation control. The addition of tested oil does not affect the cathodic Tafel slope ($|\beta_c|$ remains constant) indicating that the mechanism of H^+ reduction is not modified with the oil concentration. Also the corrosion potential is almost constant in the presence of oil inhibitor.

The anodic curves with and without Jatropha curcas oil show that the inhibition mode depended upon electrode potential (figure 4). In the case where the corrosion inhibition depends on the potential of the electrode, the observed phenomena is generally described as corrosion inhibition of the interface associated with the formation of a protective layer of adsorbed inhibition species at the electrode surface [15-23]. We conclude that the Jatropha curcas oil has effect on the cathodic and anodic behaviour of steel and acts as a mixed inhibitor.

The inhibition efficiency reaches 77 % at 1 g L^{-1}. The increase in inhibition efficiency observed at higher inhibitor concentration indicates that more inhibitor molecules are adsorbed on the metal surface thus providing wider surface coverage and the essential oil is acting as adsorption inhibitor. The values of i_{corr} decrease dramatically with the increase of Jatropha curcas oil concentrations.

IV.4. Inhibition efficiency based on EIS measurements

A better understanding of the mechanism taking place at the electrode surface was attained though EIS measurements. The EIS impedance was performed under potentiostatic conditions at E_{corr} and 308 K in the uninhibited and inhibited acidic solution containing various concentrations of Jatropha curcas. Before each measurement, the electrode was left at the open circuit conditions during 30 min. The electrode system did not evolve significantly during the impedance measurements. The impedance diagrams obtained are characterized by a capacitive behaviour.

A depressed semicircle, as often obtained in acidic media can be seen. The difference from theoretical results is generally attributed to the generation of microscopic roughness at the surface during the corrosion process [20,21]. The existence of single semicircle relates the presence of single charge-transfer process, which is unaffected by the presence of Jatropha curcas.

The electrolyte resistance R_s determined between reference and working electrodes can be obtained from the abscissa axis intercept of the semicircle at $f \to \infty$, in all solutions studied. Whereas R_t is the charge-transfer resistance and C_d the double layer capacitance. The charge-transfer resistance R_t values are calculated from the difference in impedance at Jatropha curcas were and higher frequencies, i.e. the diameter of the semicircle. In first approximation, C_d and the frequency f_{max} at which the imaginary component of the impedance is maximal $(-Z''_{max})$ are found as represented in equation 4:

$$f(-Z''_{max}) = \frac{1}{2\pi\tau_t}, \text{ where } \tau_t = R_t C_d \tag{4}$$

It may be assumed, as an approximation that, either $(R_t)^{-1}$ [22] or $(C_d)^{-1}$ [23] parameters are directly related with the corrosion rate. The inhibiting efficiency got from the charge-transfer resistance is calculated as Jatropha curcas, equation. (5):

$$E_{R_t}\% = \frac{R_{t/inh} - R_t}{R_{t/inh}} \times 100 \tag{5}$$

Where R_t and $R_{t/inh}$ are the charge-transfer resistance values without and with inhibitor, respectively.

It can be expected that the R_t values enhanced with Jatropha curcas concentrations and consequently the inhibition efficiency increases. This indicate that the charge-transfer process mainly control the corrosion of mild steel. The values of double layer capacitance are brought down to the maximum extend in the presence of Jatropha curcas oil and the decrease of C_d of Jatropha curcas ws the order similar to that obtained for i_{corr} in this study. This result is in favour of selectively adsorption of Jatropha curcas oil in specific places [24] and/or formation of complex onto the metal surface [14]. According to this inhibition mechanism inhibitor could be adsorbed on active points, thus causing the corrosion rate to drop. We note also that values of inhibiting efficiency obtained from the three methods used in the present study are in a quite reasonable agreement.

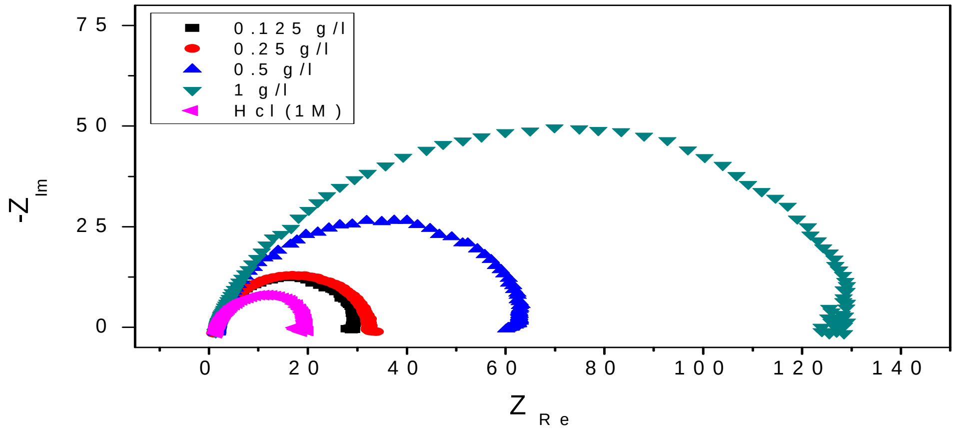


Figure 5: Nyquist plots of mild steel in 1M HCl containing various concentrations of inhibitors

Electrochemical impedance spectroscopy method is carried out to get more information about the corrosion behaviour of steel/acid interface. Figure 5 shows the diagrams of steel in 1M HCl in the presence and absence of Jatropha curcas oil. EIS parameters were given in Table 4. Inhibition efficiency (E_R%) was obtained by the Jatropha curcas inhibitor wing equation 6 :

$$E_R(\%) = (1 - \frac{R_t}{R_t^{'}}).100 \qquad (6)$$

where R_t' and R_t were the charge transfer resistance of steel in acid with and without inhibitor.

Table 4: Impedance parameters for Mild steel in 1M HCl for various Jatropha curcas concentrations

Inhibitor	Concentration g L^{-1}	R_{ct} (ohm.cm^2)	f_{max} (Hz)	C_{dl} (µF/cm2)	E (%)
Blank		16.96	111.61	8.41224 10^{-5}	-
Inhibitors	0.125	28.54	100	5.57939 10^{-5}	41
	0. 25	38.43	79.365	5.22085 10^{-5}	56
	0.5	57.13	56.18	4.96129 10^{-5}	70
	1	113.7	31.646	4.42549 10^{-5}	85

It was clear that the addition of inhibitor increased R_t value and decreased C_d value. This phenomenon is explained by the adsorption of inhibitor on the steel surface. Inhibition efficiency obtained is in significantly with those obtained by Weight loss and polarisation methods.

IV.5. Adsorption isotherm

Adhesion of molecules dissolved to a surface is called adsorption. In this process creates a film of the adsorbate– the molecules being accumulated, on the surface of the adsorbent. The molecules of liquids or the solutes in solutions get adhered to the surface of the solids. In adsorption process two substances are involved. One is the solid on which adsorption occurs and it is called adsorbent. The second is the adsorbate, which are the inhibitors or the solute from a solution which gets adsorbed on the surface.

Depending upon the nature of forces existing between adsorbate molecules and adsorbent, the adsorption can be classified into two types: Physical adsorption (physisorption): If the force of attraction existing between adsorbate and adsorbent are Vander Waal's forces, the adsorption is called physical adsorption. It is also known as Vander Waal's adsorption. In physical adsorption the force of attraction between the adsorbate and adsorbent are very weak, therefore this type of adsorption can be easily reversed by heating or by decreasing the pressure.

The Jatropha curcas oil may be adsorbed on the metal surface in the form of neutral molecules involving the replacement of water molecules from the metal surface and sharing of electrons between the oxygen atoms and

the metal surface. These heteroatoms compounds may also be absorbed through electrostatic interactions between the positively charged atom and the negatively charged metal surface [24]. It has been observed that the adsorption of the inhibitor can be influenced by the nature of anions in acidic solution [25]. The specific adsorption of anions having a smaller degree of hydration, such as chloride ions, is expected to be more pronounced. Being specifically adsorbed, they create a layer of excess negative charge directed towards the solution and favour more adsorption of the cations. Strong adsorption of organic molecules forming the inhibitor oils is not always a direct binding of the molecule with the metal surface. In some cases, the adsorption can occur through the already adsorbed chloride ions which interact with the subsequently adsorbed organic molecules forming the Jatropha curcas composition. The inhibition is to be expected for an anion–cation pair in which both ions have appreciable tendencies toward covalent binding [26].
The percentage inhibition efficiencies calculated from the weight loss data presented in the figure 6. Consistent with an S-shaped adsorbed isotherm for Jatropha curcas showing an adsorption on the steel surface according to the Frumkin isotherm, equation 7 and 8:

$$\left(\frac{\theta}{1-\theta}\right)\exp\left(-\,f\theta\right)=K.C \qquad (7)$$

with

$$K=\left(\frac{1}{55.5}\right)\exp\left(-\frac{\Delta G_{ads}}{RT}\right) \qquad (8)$$

ΔG_{ads} is the free enthalpy and f is a function of adsorption energy. The adsorption indicating major components have more active sites in the Jatropha curcas oil is strongly adsorbed on the metal surface and indicating that it is strongly adsorbed on the metal surface and accompanied by mutual attraction of the molecules. In general, when the value of the ΔG_{ads} energy is negative indicate that the adsorption of inhibitors on the steel surface is a spontaneous process. And this means that the adsorption process takes place easily and the adsorption layer on mild steel is stable.

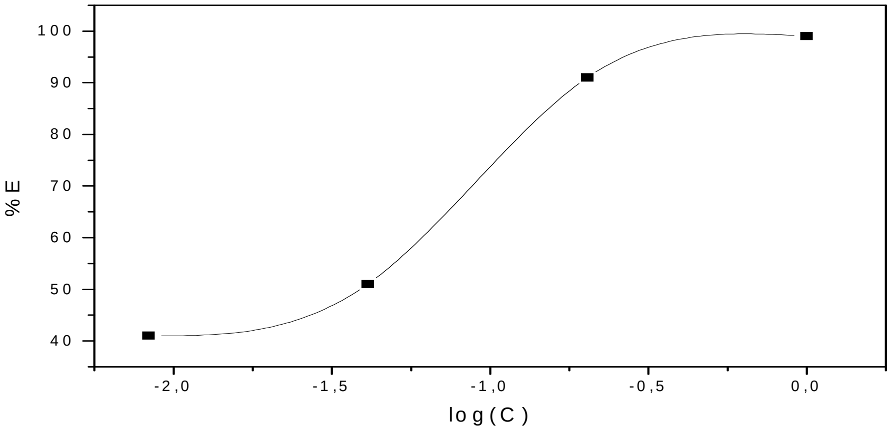


Figure 6. Frumkin isotherm adsorption model of Jatropha curcas oil on steel in 1MHCl

This is in fact possible in view of the presence of unshared electron pairs in the organic compounds molecules forming the oil inhibitor and taking into consideration the behaviour of steel as electrons acceptor as its d-sub monolayer is incomplete. The inhibitors studied may then be adsorbed via donor–acceptor interactions between the π-electrons of the systems and the unshared electrons pairs of the heteroatom's (-O) to form a bond with the vacant d orbital of the mild steel atom on the metal surface, which act as a Lewis acid, leading to the formation of a protective film. According to that the adsorption model is favoured by establishing electrostatic interaction between the inhibitor molecule and alloy surface. it can be seen that inhibitors which contains group (which is release electrons) in the inhibitor molecule will increase the electron density on the active centre on the molecule and good protection will occur. It is safe to not determineΔG_{ads} values in mixtures components as the extracts and oils of natural plants, the concentration notion losses its meaning and extracts and oils contents are expressed by ppm or mL/L [27-29].

Conclusion

The principal finding of present work can be summarised as follows:

1. A correlation was obtained between the percentage inhibition efficiencies calculated from the polarisation and weight loss data
2. Steady state electrochemical measurements have shown that the Jatropha curcas oil acts mixed inhibitor for the corrosion of steel in 1M HCL without modifying the mechanism of hydrogen evolution reaction.
3. The adsorption of Jatropha curcas oil on the steel surface in molar in hydrochloric acid obeys to the Frumkin adsorption isotherm model.
4. The inhibitory efficiency is dependent of the concentration of the Jatropha curcas oil and the efficiency reaches 99% at 1g/L of Jatropha curcas oil concentration.

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