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Opuntia Ficus Indica Extract as a Corrosion Inhibitor for 304 Stainless Steel in Sulfuric Acid Solution

Latifa Hamadi[1], Soumia Benbouta[1], Abdelhak Kareche[2], Salah Mansouri[3]

[1]Laboratory of Structural Mechanics and materials, Faculty of Technology, University of Batna 2, Algeria
[2]Department of Mechanic, Faculty of Technology, University of Batna 2, Algeria
[3]Laboratory of Innovation in Construction, Eco-design and Seismic Engineering, Faculty of Technology, University of Batna 2, Algeria

Abstract: ***The addition of the extract of cladodes of Opuntia Ficus Indica (OFI) in an H_2SO_4 solution to toward the corrosion of 304 stainless steel was studied using weight loss measurement and potentiodynamic polarization curves. A maximum inhibition efficiency of about 91.23% was achieved at 300mg/L. Results reveal that the inhibition efficiency has increased with an increase in extract concentration. By analysis of polarization curves, it is found that OFI acts as a mixed-type inhibitor. The adsorption of OFI is found to obey the Langmuir isotherm. The adsorption free energy for OFI on stainless steel surface reveals a physical adsorption of the inhibitor on the metal surface.***

Keywords: Opuntia Ficus Indica; 304 stainless steel; Corrosion inhibition; Sulfuric acid

1. Introduction

304 stainless steel (304 SS) has excellent resistance to a large range of atmospheric environments and numerous corrosive medium. It is frequently used in chemical processing equipment, heat exchangers, and food, dairy, and beverage industries **[1]** due to its different properties. 304 SS is subject to different forms of corrosion such as pitting in chloride environments **[2, 3]** and to stress corrosion **[4, 5]** at high temperature. Metal corrosion is a great industrial problem, and it has attracted the attention of researcher's worldwide **[6]**. The prevention of metal against this problem is obligatory. Among various corrosion prevention methods, corrosion inhibition presents advantages of economy, high-efficiency, and facile-feasibility, and has been widely applied in different domains **[7]**.

Recently, usage of toxic materials as inhibitors, especially chromates and organo-phosphates, has been limited because of their environmentally harmful characteristics. Several studies have been indicated that non-toxic and organic inhibitors have again become important because of their environmentally friendly, readily and availability and renewable sources **[8, 9]**. These compounds are called environmentally friendly, eco-friendly, nature-friendly or green inhibitors

The initial report of corrosion inhibition by organic compounds is attributed to Speller **[10]**. In 1981 Sanyal, reported that the extract of Chelidonium majus was used as the first organic inhibitor in the pickling process using sulfuric acid **[11]**.

Numerous natural products, for example, Silybum marianum **[12]**, red pepper seed **[13]**, Thymus Vulgaris **[14]** and inorganic compounds such as butan-1-ol **[15]** and molybdate **[16]**, all have been reported to be effective in reducing the corrosion rate of 304 SS in acidic media. Recently and with the new topics on the corrosion protection field, polymers or inorganic coating have been used for corrosion protection of 304 SS **[1, 17]**. In another hand, the self-assembled monolayer of red pepper seed oil was prepared on 304 SS surface and tested its ability to control the corrosion rate **[18]**.

OFI is a dicotyledonous angiosperm plant. It belongs to the Cactaceae family and is characterized by its remarkable adaptation to arid and semi-arid climates in tropical and subtropical regions of the globe. OFI has large cladodes covered with hairs and thorns. There are also varieties without thorns. The cladodes of OFI are very fibrous, retain water and allow the plant to resist heat and drought. Its rich composition in polyphenols, vitamins, polyunsaturated fatty acids and amino acids has been highlighted through the use of a large panel of extraction methods. El-Mostafa in his review has presented the distribution and contents of phenols and flavonoids in the various parts of OFI **[19]**. The property of antioxidant activity of OFI is attributed to the presence of the phenolic compounds in its different parts **[20, 21]**. Ayadi et al in his work has shown that spiny and spineless cladodes are rich in bio-molecule and dietary fiber **[22]**. F.A. Goycoolea and A. Cardenas indicated in their review that the chemical and industrial analyses of the OFI liquid elucidate that it consists of a large amount of pectin and many sugars **[23]**.

There are reports on the use of OFI plant extract for corrosion protection for different metals in various acid solutions. For example, the effect of the methanolic extract of OFI flowers as a green corrosion inhibitor for mild steel in 1MHCl solution has been investigated by using weight loss tests, potentiodynamic polarization curves, and electrochemical impedance spectroscopy measurements. Results indicate that inhibition efficiency has increased with an increase in extract concentration. Polarization curves reveal that the investigated plant extract is a cathodic behavior. It was found that the adsorption of the inhibitor on mild steel surface obeys the Langmuir's adsorption isotherm **[16]**. Similarly, Flores et al used weight loss tests and electrochemical techniques to investigate the protective ability of OFI on carbon steel in 1M HCl. The inhibition efficiency was found to increase with an increase in inhibitor concentration but decreased with rising in temperature. Polarization curves reveal that the investigated extract is a cathodic behavior inhibitor that is physically adsorbed onto the steel surface. It was found also that adsorption of OFI on carbon steel surface follows Langmuir adsorption isotherm **[24]**. In another published paper, the inhibitive action of OFI toward the corrosion of steel is tested using electrochemical measurements. It was found that the dehydrated OFI acts as a good corrosion inhibitor for steel in chloride contaminated alkaline solution. The microscopic evaluation indicated that the addition of OFI led to the formation of a denser layer on the steel surface that decreased corrosion activity **[25]**. In another study, the adsorptive and inhibitive action of mucilage extracted from the modified stems of prickly pears towards corrosion of aluminum in HCl solution was investigated using weight loss, thermometry, hydrogen evolution, and polarization. It was found that the OFI extract acts as an effective corrosion inhibitor for aluminum corrosion in HCl solution **[26]**.

In this work, inhibition effect of the extract of OFI as a green inhibitor on corrosion of 304 SS in 0.5M H_2SO_4 solution was studied. Weight loss and potentiodynamic polarization tests were employed to investigate the effect of inhibitor concentration on corrosion process.

2. Experimental Procedures

2.1. Material

304 SS was used as the substrate with the chemical composition given in **Table 1**

Table 1: The chemical composition of 304 SS

Element	C	Mn	Si	Ni	Cr	P	S	Mg	Fe
Wt(%)	0.05	1.2	0.4	8.01	17.59	0	0.02	-	Bal

2.1.1. Samples preparation

From a 14mm diameter SS 304 bar, samples of 8mm in length were cut using a power saw under lubricant. Samples are prepared using conventional polishing methods. They are mechanically polished with different grades Silicon Carbide papers (180, 400, 600, 1000, 1200, 2000 and 2400 finish). The operation was controlled with an inverted Metallographic microscope, Nikon EPIPHOT (×400). Finally, the samples were washed with water, rinsed with acetone, washed again thoroughly with doubly distilled water for several times, dried and then stored in a desiccator.

2.2. Test media

0.5M of sulfuric acid was prepared from the H_2SO_4 mother solution of analytical grade by dilution with distilled water.

2.3. Inhibitor

The OFI was collected from the region of Ngaous (Batna-Algeria). Slices of cladodes of OFI were ground into a fine powder which was then passed through different sieves. Next, 500g of powder was blended with 1L of ethanol for 24h. The resulting mélange was filtered with a filter paper placed on a glass funnel to obtain the homogeneous solution that was concentrated after in a rotary evaporator followed by drying in a vacuum oven for one night. As a result of the process, extract of OFI was obtained.

2.4. Techniques employed

2.4.1. Weight loss test

The purpose of corrosion studies is to determine quantitatively the degradation of a material over time. The simplest method and the least expensive is that of mass loss. The principle of this technique is the immersion of the sample to be studied after it's weighing, during a definite time in the corrosive medium considered without and with inhibitor. Then the sample is removed from the test solution and reweighed. The utility of measuring lost mass is the evaluation of the rate of corrosion, which is defined as a weight loss per unit area and time and is expressed in (g/cm^2.year).

2.4.2. Potentiodynamic polarization test

The experiments were performed in an electrochemical cell with saturated calomel electrode (SCE) as a reference and a platinum electrode as a counter electrode. The 304 SS was used as a working electrode specimen. potentiodynamic experiments were conducted using a Voltalab PGP201 Potentiostat/Galvanostat that interfaced to a computer that was used for data acquisition. The electrodes were immersed in solutions consisting of sulfuric acid (0.5M) with and without organic inhibitors (OFI) at concentrations ranging from (100, 200, 300 mg/L). The potential variation is recorded for 6 hours, followed by potentiodynamic polarization, which was performed by scanning the applied potential from -1000 mV/SCE to +1000 mV/SCE, at 5 mV/min scanning rate. The measurements were made under aerated conditions without agitation. Tafel plot was applied to the current potential curve, in order to extract some electrochemical parameters such as the corrosion potential, the corrosion current, polarization resistance and to calculate inhibition efficiencies.

3. Results and discussion

The effect of the presence of the extract of OFI near the surface of the SS 304 on weight loss, and the potentiodynamic polarization curves response is thoroughly examined. The inhibitor concentration is the important factor in this work which affects more the behavior of the ionic transport to the 304 SS surface.

3.1. Weight loss test

The inhibition efficiency E(%) corrosion in the case of the gravimetric method was calculated from the following equation[12]:

$$E(\%) = \frac{V_{0corr} - V_{corr}}{V_{0corr}} \times 100 \quad (1)$$

Where V_{0corr} and V_{corr} are the corrosion rates of 304 SS due to the dissolution in 0.5 M H_2SO_4 in the absence and the presence of definite concentrations of the OFI, respectively.

The corrosion rate of tested specimens in 0.5M H_2SO_4 solution at different concentrations of OFI was determined after 3 days of immersion time at 298K. The obtained corrosion rates (Vcorr) and the inhibition efficiency are represented in **Table 2**.

Results obtained from this method show that the corrosion rates values decrease when the concentration of the OFI increases due to the performance of the extract of a natural plant that affected by the adsorption of organic molecules of the extract on the metal surface. From the analysis of **Table 2**, it is very clear that protection efficiency increased with the increase in concentration from 100 to 300 mg/L for the extract studied. We noted that OFI used in this work showed very excellent corrosion inhibitor for 304 SS in 0.5 M H_2SO_4. A remarkably high maximum inhibition efficiency of (88.23%) was obtained at the highest OFI concentration applied (300 mg/L).

Table 2: Weight loss results of 304SS in 0.5 M H_2SO_4 with addition of various concentrations of OFI

C of OFI (mg/l)	Δm (mg)	V (g/cm^2.year)	E (%)
0	0.0112	0.4922	---
100	0.0035	0.1548	68.54
200	0.0023	0.1010	79.48
300	0.0013	0.0579	88.23

3.2. Potentiodynamic polarization test

The effect of OFI concentration on the corrosion behavior of stainless steel electrode in 0.5M sulfuric acid solution has been studied by polarization measurements and the recorded Tafel plots are shown in **Figure 1**. From polarization curves, the electrochemical parameters as obtained are shown in **Table 3**. These include electrochemical corrosion kinetic parameters such as corrosion potential (Ecorr), current density (Icorr), corrosion rate (mm/ year), cathodic and anodic Tafel slopes (Bc and Ba), polarization resistance were determined by the analysis of Tafel plot. The inhibition efficiency E (%) and surface coverage (θ) were also listed in **Table 3**.

The percentage inhibition efficiency E(%) and the degree of surface coverage (θ) were calculated using the following equations **[12, 13, 16, 21, 24]**.

$$E(\%) = \frac{I_{0corr} - I_{corrinhi}}{I_{0corr}} \times 100 \quad (2)$$

$$\theta = \frac{I_{0corr} - I_{corrinhi}}{I_{0corr}} \quad (3)$$

Where (I_{corr} and $I_{corrinhi}$ are the corrosion current density in the absence and the presence of an inhibitor respectively.

A maximum inhibition efficiency of about 91.23% for OFI was achieved at 300 mg/L. It is clearly observed that OFI reduces the corrosion current density and the suppression in current increases as the inhibitor concentration increases. In the presence of OFI corrosion potential is shifted to positive values. Ba and Bc Tafel slope values are not significantly affected in an inhibited solution as compared to an uninhibited solution. This observation shows that the inhibitor molecules are adsorbed onto the metallic surface by blocking the active sites on the surface. Depending on the nature of corrosive environment and nature of metal, inhibitor behaves as a different type of inhibitor, namely cathodic, anodic or mixed type. According to Riggs **[12, 27]**, the inhibitor can be seen as of the cathodic or anodic type if the displacement in the corrosion potential (E_{corr}) for the uninhibited system is higher than 85 mV with respect to (E_{corr}) in the inhibited system. If this displacement is less than 85 mV the inhibitor can be seen as of the mixed type.

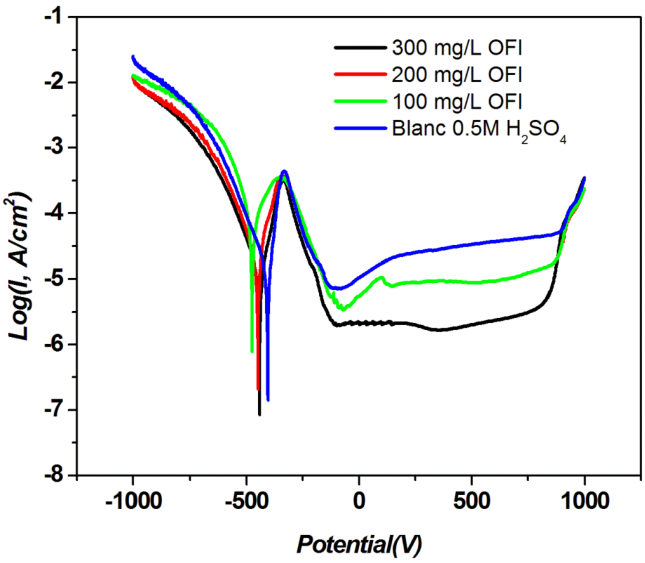

Figure 1: Tafel polarization curves for 304SS in 0.5M H_2SO_4 with various concentrations of OFI at 298 K.

The results in Table 3 reveal that the differences Ecorr values between inhibited and uninhibited systems are generally lower than 85 mV, approximately 68 mV, which implies that the studied OFI is a mixed-type inhibitor. Organic inhibitors cannot be specifically designed as anodic or cathodic inhibitors. They are mixed inhibitors that inhibit both anodic and cathodic reactions. The effectiveness of organic compounds is allied to the extent to which they are adsorbed and coat the metal surface. Corrosion inhibitors effectively block metal oxidation and eliminate the unwanted destructive effect.

Table 3: Tafel polarization parameters obtained at different concentrations of OFI for 304 SS in 0.5 M H_2SO_4 at 298K.

Inhibitor OFI (mg/l)	Ecorr (mV)	Icorr (μA cm^{-2})	Ba (mV)	Bc (mV)	Rp (Ω cm^{-2})	CR (mm/y)	θ	E (%)
blank	-473.4	74.7	150.4	95.5	237.32	0.874	---	---
100	-447.6	21.4	76.2	-121.1	795.85	0.250	0.7135	71.35
200	-440.6	8.3	58.5	-103.9	1230	0.0097	0.8888	88.88
300	-404.6	6.5	32.8	-104.1	1000	0.0075	0.9129	91.29

3.3. Comparison of inhibition efficiency obtained by weight loss test and polarization measurement

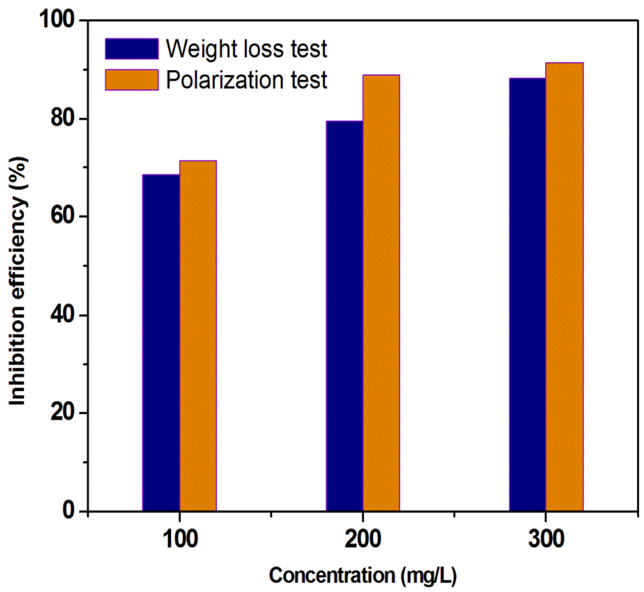

Figure 2: The comparative graph for weight loss test and polarization test

Figure 2 presents the bar graph that obtained using inhibition efficiency obtained by weight loss test and polarization method. It's quite clear that the results are almost approximated. However, the highest inhibition efficiency was obtained by polarization method (91.29%).

4. Adsorption Isotherm

In aggressive solution, the action of an inhibitor is assumed to be due to its adsorption at the metal/solution surface. The adsorption process is influenced by many factors, such as the metal (surface state, chemical nature), the medium (concentration, pH, and temperature), inhibitor (concentration, structure molecular, solubility, the inhibitor-metal surface bonding). Really, the solvent H_2O molecules could also be adsorbed at metal/solution interface. Therefore, the adsorption of organic inhibitor molecules from the aqueous solution can be considered as a quasi-substitution process between the organic compounds in the aqueous phase $Org_{(sol)}$ and water molecules at the metal surface $H_2O(ads)$ **[12]**.

The adsorption of the organic substance (OFI extract) at the metal/solution surface may be written according to the following reaction:

$$\text{Organic}_{(sol)} + n\, H_2O_{(ads)} \longrightarrow \text{Organic}_{(ads)} + n\, H_2O_{(sol)}$$

Where n is the number of water molecules replaced by one organic inhibitor. Distinctly, the value of n depends on how the molecule arranges itself on the surface grossness, and on the interaction among molecules. The adsorption isotherm provides us up the basic information on the interaction between the inhibitor molecules and the metal surface. To obtain more information on the interaction between OFI and the electrode surface, the Langmuir isotherm was investigated. The degree of surface coverage values (θ) for different concentrations of the tested inhibitor in H_2SO_4 solution, have been calculated from Tafel polarization data according to equation (3) and listed in **Table 3**. The obtained values of θ are fitted to Langmuir isotherm. The Langmuir isotherm is verified in the case of OFI **[12, 16]**. Langmuir adsorption isotherm is expressed by:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (4)$$

Where C_{inh} is the inhibitor concentration, K_{ads} is the adsorption equilibrium constant and θ is the surface coverage.

Figure 3 presents the best fitted straight line which is obtained for the plot of C_{inh}/θ versus C_{inh}. This indicates that the adsorption of OFI onto the surface of stainless steel surface follows the Langmuir isotherm. K_{ads} value is calculated from the intercepts of the straight line on the C_{inh}/θ-axis. The free energy of adsorption (ΔG°_{ads}) is the most important thermodynamic adsorption parameter. The K_{ads} was related to the standard free energy of adsorption (ΔG°_{ads}) with the following equation:

$$K_{ads} = \frac{1}{C_{sol}} \exp \frac{-\Delta G^0_{ads}}{RT} \quad (5)$$

Where R is the gas constant (8.314 $J.K^{-1}mol^{-1}$), T is the absolute temperature (K) and C_{sol} is the molar concentration of the solvent which is 55.5 mol. L^{-1} in the case of water. In our study, the unit ok K_{ads} is L g^{-1}, which in turn implies that the unit of C_{sol} is g/L with the value of approximate 1.0X 10^3 **[28]**. The thermodynamic parameters of OFI adsorption on 304 SS in 0.5M H_2SO_4 at 298K are calculated and they are given in **Table 4.**

Table 4: Thermodynamic parameters of OFI adsorption on 304 SS at 298K and in 0.5M H_2SO_4

System	Langmuir isotherm		
	K_{ads} L/g	R^2	$\Delta G°_{ads}$(KJ mol^{-1})
OFI	15.58	0.9967	-23.9

Generally, the values of $\Delta G°_{ads}$ up to -20 KJ mol^{-1} are consistent with the electrostatic interaction between the charged molecules and the charged metal (physical adsorption) while those more negative than -40 KJ mol^{-1} involve sharing or transfer of electrons from the inhibitor molecules to the electrode surface to form a coordinate type of bond (chemisorption).

In our work, the value of free energy of adsorption of OFI on 304 SS surface, $\Delta G°_{ads}$, is found to be -23.9 KJ mol^{-1}. The calculated value for the adsorption of OFI on the electrode 304 SS suggests that the adsorption is made in a physical way. In this case, a coordinate type of bond was formed onto the surface of 304 SS. The adsorption of OFI obeys the Langmuir isotherm, and the negative sign of $\Delta G°_{ads}$ indicate the strong nature of interactions between the inhibitor molecules and the metal surface and the high efficient adsorption. The character of metal corrosion inhibition by organic inhibitors depends on the capacity of the inhibitor to get adsorbed and form a protective coherent film on the metal surface.

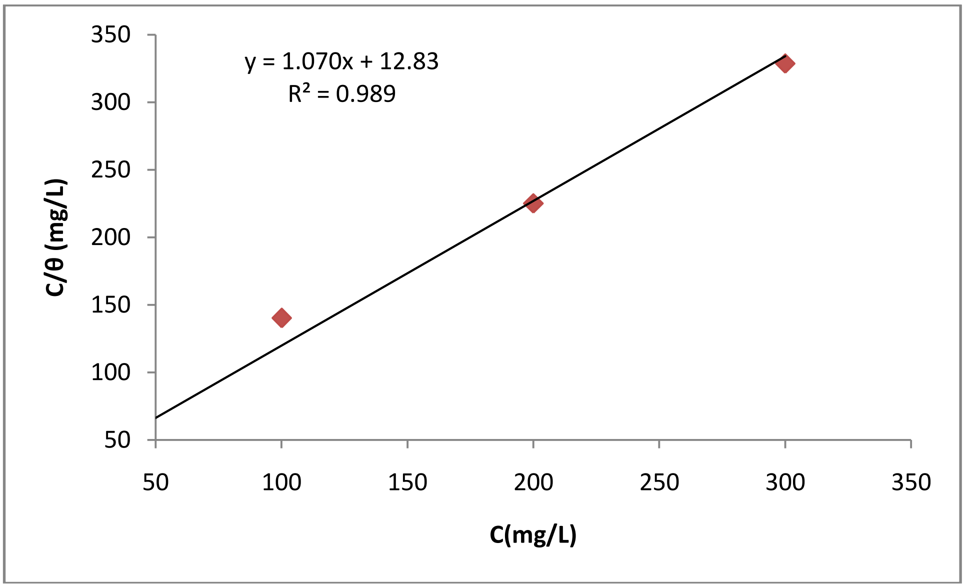

Figure 3: Langmuir adsorption isotherm for OFI on 304 SS obtained by Tafel data at T= 298K in H_2SO_4 solution (0.25M).

As well we presented (in the introduction) that OFI contains various bioactive molecules; such as, phenols and flavonoids and amino acids **[19]** that have a particular importance to cite it as green corrosion inhibitors, due to their nontoxicity and biodegradability. These properties would justify their use as corrosion inhibitors. The corrosion inhibition effect of some amino acids was evaluated by several researchers worldwide **[29-31]**. **Figure 4** presents the chemical structure of flavonoids. It's quite clear that flavonoids molecule contains benzene rings (source of ∏-electrons) and oxygen atom (no bonding electrons) in its structure. Flavonoids could be the responsible for the formation of chemical bond on to the surface of stainless steel. However, it was found through the extraction methods that polymer such as pectin **[23]** lignin**,** polysaccharide and cellulose **[32,33]** have existed in the OFI extract. The use of polymers as corrosion inhibitors has attracted considerable attention recently, because, through their functional groups, they form complexes with metal ions and on metal surfaces, thereby blanketing the surface and protecting the metal from corrosive agents present in the solution **[34]**.

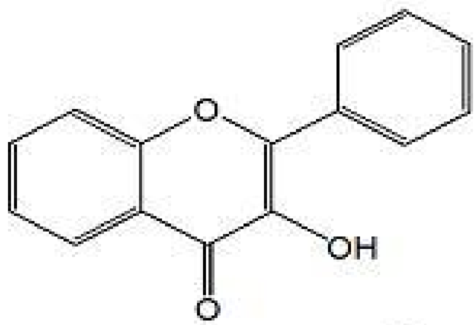

Figure 4: Chemical structure of flavonoids

5. Conclusion

In the present work, weight loss and potentiodynamic polarization test were used to investigate the ability of Opuntia Ficus Indica to inhibit the corrosion of 304 stainless steel in sulfuric acid solution. The principal conclusions are the following:

- The OFI acts as a good inhibitor for corrosion of 304 SS in 0.5M H_2SO_4. The inhibition efficiency increases with increasing extract concentration.
- OFI acts as a mixed-type inhibitor influencing both cathodic hydrogen evolution and anodic stainless steel

dissolution reactions as revealed by the polarization measurements.

- An increase in the concentration of the inhibitor leads to the decrease of the current and of the corrosion rate. It was found also that the corrosion current density obtained by the Tafel plot of the polarization curve of 304 SS IN 0.5M H_2SO_4 is 74.7 μA cm^{-2}, while the current density is 21.4 μA cm^{-2} when 100mg/L of OFI are added. In addition, it was found also that the polarization resistance was to be important when the concentration of inhibitor was increased.
- The extract of OFI was found to be physically adsorbed on the 304 SS surface following the Langmuir isotherm model.
- Weight loss test and potentiodynamic curves are almost in agreement, however, the high inhibition efficiency value (91.23%) was obtained by polarization technique.

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