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Extracted of Fresh Olive Leaves for Corrosion Steel Inhibitor

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Abstract

The corrosion problem is one of the most important problems in our days, because it causes very series economic problems for construction and plant. Therefore, many research are carried out in various fields in order to reduce loses specially in oil extraction method because the equipment used in this field are very expensive and the corrosion of it causes a serious problem. In this study fresh olive leaves extraction used as corrosion natural inhibitor for protection steel pipe and equipment used in field of oil extraction, at temperatures (25°C and 45°C), in medium solution that was prepared in the following concentration to stimulate the field environment for oil extracting. (19.2% of sodium chloride (NaCl), 8% of calcium chloride (CaCl), 1.08% of magnesium chloride (MgCl) and saturated with CO₂ gas, the corrosion rate of steel sample were measured be using a potentiostat device with reference of calomel electrode. This project is to simulate and evaluate the corrosion behaviours and the extent of inhibition of fresh olive leaves extraction. The result was, the fresh olive leaves extract is an effective inhibitor material. Fresh olive leaves extraction inhibitor has a low corrosion rate at 25°C and 45°C and the Corrosion rate increase with increasing temperature for oil extraction solution. These results are induct to use the fresh olive leaves extraction as green inhibitor for steel pipe and structures.

Keywords

Fresh Olive Leaves, Potentiostat, Inhibitor Efficiency, Electro-Chemical Polarization

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1. Introduction

The technology of today has a wide kind of engineering material (metal and it's alloys, rubber, plastics, ceramics, wood, composites, etc.) and the choosing an appropriate one for the specific application is the most important job for the design engineer. There are many factors according to it, the choice will be done, one of it. The corrosion of material in the work environment so it's very important to studying the corrosion and it's kind and mechanism and to study the way of protection from it. [1]

Corrosion is the detrimental attack of the material by the effect of its environment. The serious result of the corrosion

becomes a real problem of the world. In addition to our always facing with the problem of destroying the material. there are many effects of corrosion problems from these problems are:-

- 1. Plant shutdowns.
- 2. The valuable sources are wasting. Loss of product or maybe cause contamination of it.
- 3. The efficiency is reduced.
- 4. Need high cost maintenance.
- 5. To eliminate the corrosion there will be over designing

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cost.

It also threatens safety and impairs technological progress. The multidisciplinary side of corrosion problems combined with Distributing the responsibilities associated with such problems only Increased the hardness of the subject. Material losses and corrosion consequences are priced so high that in some countries like the U.S. and England these factors have been estimated from 3 to 4% of the GDP.[2] Corrosion not only has economic implications, but also social and these engage the safety and health of people either working in industries or living in nearby towns. The oil industry in Mexico is one of the most affected by corrosion because this phenomenon exerts its effects from the very moment of oil extraction on, causing a constant struggle against it [3]. The use of corrosion inhibitors (CIs) constitutes one of the most economical ways to mitigate the corrosion rate, protect metal surfaces against corrosion and preserve industrial facilities [4].

The purpose of inhibitors is to form a barrier of one or several molecular films against the attack of acid. This protective action associated with chemical and/or physical adsorption containing a variant charge of the adsorbed substance and transfer the charge from one phase to another, Because of the toxic nature and high cost of some chemicals currently are used as inhibitors [5]. So the practical criteria for selection of the corrosion inhibitors from the variety compounds of inorganic and organic substances with sufficient inhibiting properties are not only their inhibition

efficiency but it must be the safety of use, compatibility with other chemicals in the structure, economic constraints and environmental concerns [6]. There are many studied effects of temperature on the inhibitors efficiency [7], it is seen that Inhibition efficiency improved with concentration but decreased with rise in temperature [8]. Therefore, plants and natural product extracts have been posed to achieve the target products which are used as co-friendly corrosion inhibitors for various metals and alloys in different acid media [1, 10].

2. Experimental

Electrochemical Tafel test method, Scanning Electron Microscopy, EDS detection, optical microscope examination, and Fourier Transform Infrared-spectroscopy analysis applying, in order to detect the effect of fresh olive leaves extraction with different temperature on mild steel samples corroded in crude oil extraction.

2.1. Materials

2.1.1. The Mild Steel Samples

A cylindrical mild steel sample which has a threaded hole in the centre of it total surface area of the sample will be 5.3 cm² in order to be fitted in the holder of the working electrode of the potentiostat device to be immersed in the prepared media solution. The chemical composition of the steel specimen is list in Table 1.

Table 1. The Chemical Analysis of Mild Steel Sample.

Element %	С%	Si%	S%	P%	Mn%	Cr%	Ni%	Mo%	Cu%	Fe%
Result	0.21	0.12	0.02	0.03	0.6	0.07	0.07	0.1	0.03	Rem.

Cylindrical samples are used for electrochemical tests. Extraction of fresh olive leaves is added to crude oil extraction solution at different temperatures.

2.1.2. Green leaf Extraction

The olive leaves are collected from the tree and washed from any dirt then blend, Mixing 100 gm. Of blended olive leaves with 1000 ml of ethanol and made the fractional distillation for (1 hr) to get 450 ml the extraction and finally the solution is Filtered.

2.1.3. The Solution Preparation

The medium used in testing is the solution that was prepared in the following concentration to stimulate the field environment for crude oil extracting. (19.2% of sodium chloride (NaCl), 8% of calcium chloride (CaCl), 1.08% of magnesium chloride (MgCl) and saturated with CO₂ gas, this solution is the same as the solution used in the extraction process of crude oil, at temperatures (25°C and 45°C) [11].

2.2. Electrochemicel Measurements

A Gamry Serise G300 Potentiostat built in a computer used for electrochemical corrosion measurement (Tafel test). The test was of three electrodes connection (reference electrode, counter electrode and working electrode). Saturated Calomel reference electrode was the reference electrode and graphite rod was the auxiliary electrode. By using Gamry workframe software to record test data, the experiments were carried at room temperature. Saving data in Data Gamry folder and using Gamry Echem Analyst software [12]. The inhibitor efficiency percent (ŋ%) has been calculated for each olive leave extraction at each test condition by using formula (1) [13].

$$\eta\% = \frac{W_{corr.} - W_H}{W_{corr.}} \times 100 \tag{1}$$

Where: $\eta\%$ = inhibitor efficiency percent,

W_{corr} = Corrosion rate in mils per year (mpy), and

 W_H = Corrosion rate in (mpy) with inhibitor.

2.3. Fourier Transform Infrared Spectroscopy Analysis (FT-IR test)

This test has been done for identify the chemical compounds that compose with the fresh olive leafs, which acts as organic inhibitor.

2.4. Microscopic Test

Using optical microscope for examine the specimen surfaces. The images were taken for the specimen after test.

2.5. Scanning Electron Microscopy

Uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens.

2.6. Energy Dispersive X-ray Spectroscopy Detection

This test has been done to improve the elements that deposit on the sample surface as organic compounds help to acts as inhibitor from the fresh olive leaves extraction.

3. Results

The result found in this research and experimental work are shows and discussed to evaluate its effect.

Figure 1 show the fractional distillation unit used for extract the inhibitor of fresh olive leaves. While figure 2 shows the Gamry Serise G300 Potentiostat connected with corrosion cell. The tests

have been done in water bath at 25°C and 45°C.



Figure 1. Show the distillation unit.



Figure 2. Show the potentiostat with corrosion cell.

3.1. Electrochemical Polarization Test

3.1.1. Electrochemical Polarization Test at (25°C)

This test is done at room temperature 25°C. Figure 3 show tafel test for steel specimen without inhibitor, and with fresh inhibitor

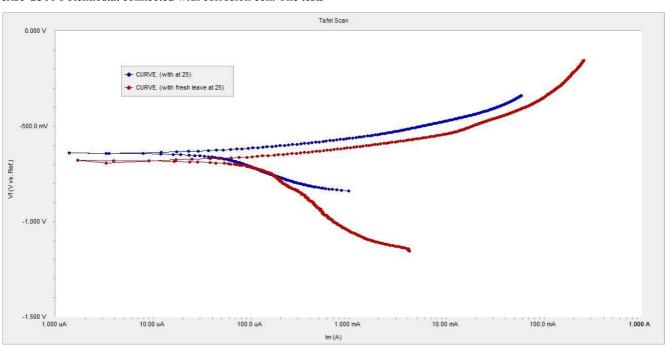


Figure 3. Electrochemical polarization test at (25°C).

3.1.2. Electrochemical Polarization Test at (45°C).

This test is done at 45°C. Figure 4 show tafel test for steel specimen without inhibitor, and with fresh inhibitor

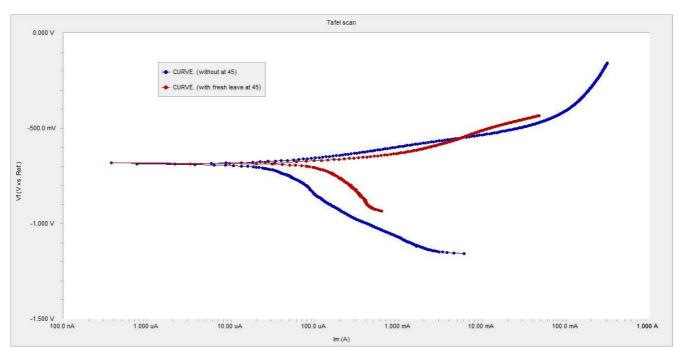


Figure 4. Electrochemical polarization test at (45°C).

3.1.3. Electrochemical Polarization Test Without Inhibitor at (25 and 45°C)

Figure 5 show tafel test for steel specimen without inhibitor at (25°C and 45°C),

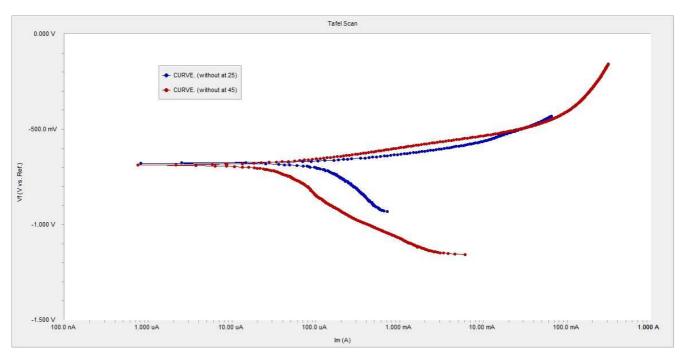


Figure 5. Electrochemical polarization test without inhibitor at (25 and 45°C).

3.1.4. Electrochemical Polarization Test with Adding Fresh Inhibitor at (25, 45°C)

Figure 6 show tafel test for steel specimen with fresh inhibitor at (25, 45°C).

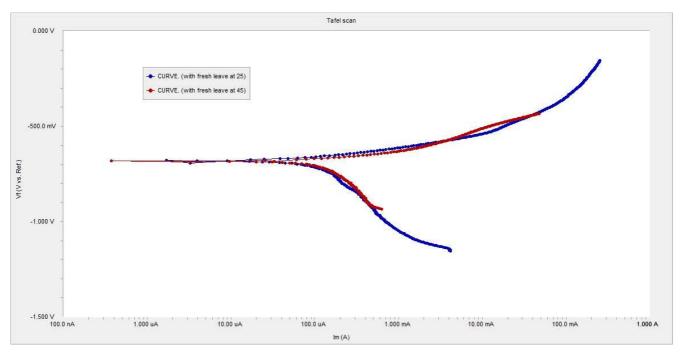


Figure 6. Electrochemical polarization test with fresh inhibitor at (25 and 45°C).

3.1.5. Corrosion Rates and Efficiencies

According to tafel tests the corrosion rates and the inhibitor efficiency have been recorded in table 2 and table 3 respectively.

Corrosion case E corr I corr β_c β_a Corrosion rate, mpy *10-3 *10-3 mV μA Without inhibitor at 25°C 125 -619 32.54 1.871 6.32 Without inhibitor at 45°C 140.44 -688 64.4 263.6 7.101 Fresh olive leave extraction at 25°C 10.1 -641 23.3 42.9 0.867Fresh olive leave extraction at 45°C 10.15 -675 7.1 12.8 0.872

Table 2. The results of the Electrochemical Polarization Tests at 25°C and 45°C.

Table 3. The efficiency of inhibitor for green olive leaves extraction at (25 and 45°C).

Temperature	25°C	45°C
Efficiency of Fresh olive leave extraction %	86.2	87.7

For figure 7 show the comparison between corrosion rates without and with fresh leave extraction inhibitors at 25 and 45°C. temperatures.

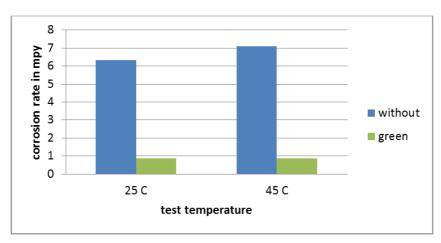


Figure 7. Comparison between corrosion rates without and with fresh leave extraction inhibitors at 25 and 45°C temperatures.

3.2. Fourier Transform Infrared Spectroscopy Analysis (FT-IR test)

Figure 8 show the analyzing of the olive leaves extraction that Deposition on the surface of sample at 25°C for fresh leaves, FT-IR identifies chemical bonds in molecule by producing an infrared absorption spectrum. the spectra

produce a profile of sample, a distinctive molecular fingerprint that can be used to screen and scan samples for many different components. these can be identified from the histogram. Each peak at a range gives a chemical compound. table 4 show the chemical compounds and their bonds of fresh olive leaves [14-15].

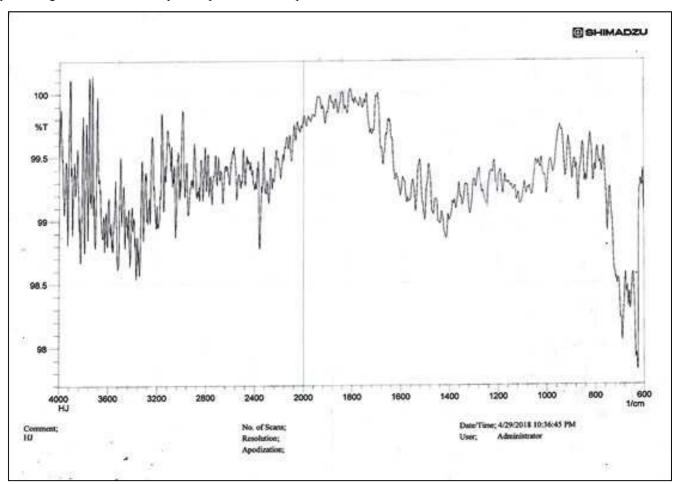


Figure 8. FT-IR diagram for deposits on the surface of low carbon steel specimen with green olive leaves extraction.

Table 4. The major Important frequencies' in the green olive leaf extraction deposit on the surface of the sample by FT-IR Test.

Stretching frequency cm ⁻¹	Major Functional group	Absorption Intensity	Compound Type	
634	C-Cl	strong	Halocompound	
679	C=C	strong	Alkene	
700	C=C	strong	Alkene	
791	C-Cl	strong	Halocompound	
976	C=C	strong	Alkene	
994	C=C	strong	Alkene	
1173	C-O	strong	Tertiary alcohol	
1250	C-O-C	strong	Ester	
1297	C-O	strong	Aromatic ester	
1430	С-Н	medium	Alkane	
1686	C-C	weak	Alkene	
1816	С-Н	medium	Alkane	
2827	С-Н	medium	Alkane	
2998	N-H	strong	Amine salt	
3096	С-Н	medium	Alkene	
3224	О-Н	strong	Alcohol	
3432	О-Н	strong	Carboxylic acid	
3891	О-Н	strong	Carboxylic acid	

3.3. Optical Microscopic Examination

From figure 9 which show the optical image for the mild steel specmine after electro-chemical test carried out, it clear that is a deposition of a layer of fresh olive extraction inhibitors. Figure 10 show the same specmine but after rubbing the deposite layer from the surface,

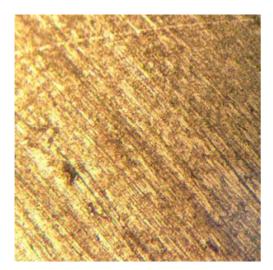


Figure 9. Deposit layer.

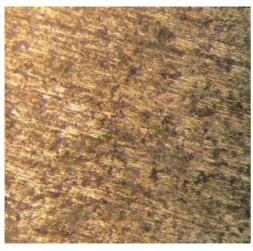
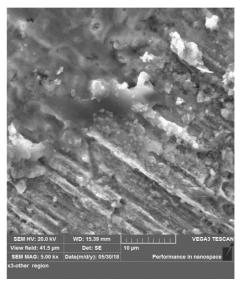


Figure 10. Rubbing the deposit layer.

From the two images its clear that the inhibition caused with deposit layer of component of the fresh olive extracted inhibitor on the surface of the specimen leading to insulate the parent metal from the corrosive medium, helping to reduce the corrosion of the steel.

3.4. Scanning Electron Microscopy

The scan is applied on two spacemen which are examined by green olive leaves extraction at 45°C to clarify what happened on the surface of each one and the result is shown in the figure 11 below. The images show that there are constitutes layer on face of the sample.



(a)

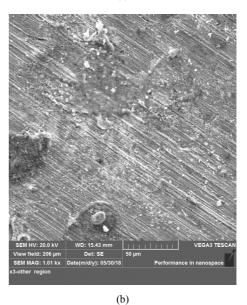


Figure 11 The SEM photo for steel sample at 45°C immersed in oil extraction solution with green olive leaves inhibitor (a) 1000X, (b) 5000X.

3.5. Energy Dispersive X-ray Spectroscopy Detection

The EDS is applied on the same sample to clarify the elements that present on the surface in order to have a good imaging of what element responsible for the inhibition process and to know what element is deposited on the surface of sample the result is shown in figure 12. It is clear that the precipitated layer like fibres surface. EDX test of the layer as shown in figure 13 present of 31.2%wt of carbon and 9.5%wt of Oxygen the phenolic compound of the olive leaves reacts with the medium, soften it and the acid activity is weakened. At the same time, the precipitated layer acts as a hinder to medium solution, which causes the inhibitor behaviour for the period of its endurance. [15-18]

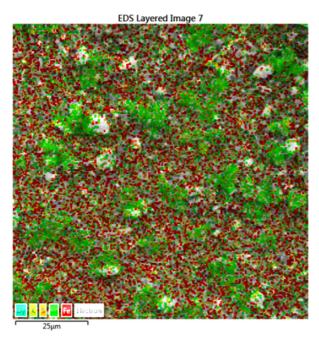


Figure 12. EDS layers photo for each element on the surface of sample at 25°C with fresh olive leaves extraction percentage.

4. Discussion

According to the table 4 for FT-IR Test its clear that there are many compounds can be act as inhibitor to many solutions media, it is an anodic adsorption inhibiter, because the anodic reactions are reduced. The microstructure for tested specimens in optical microscope (figures 9 & 10) and in SEM (figure 11) shows that a layer precipitate on the surface, which help to obstruct the metal surface from the corrosion media leading to decrease the corrosion rate of the metal. This layer is the organic compounds, which are detected with the FT-IR. This phenomenon is confirm with the EDS test (figures 12 and 13), where figure 12 show the map image of the sample surface, where's figure 13 show the chart of the elements of the precipitate layers. From the electrochemical polarization test without and with fresh olive leaves extraction inhibitor at (25 and 45°C), for the figures (3, 4, 5 and 6), its clear that a reduction in corrosion rate of the specimens, with an efficiency about 86% at 25°C, and 88% at 45°C. As mention before this is because of the inhibition layer that insulate metal surface from the medium

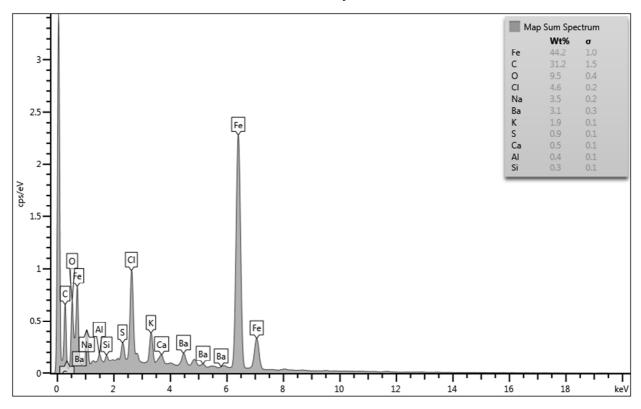


Figure 13. EDS chart shows the maps of elements on the surface of sample at 25°C with fresh olive leaves extraction and it's percentage.

5. Conclusion

Fresh olive leaves extraction could work as commercial inhibitor. It is safe, cheap and available. Corrosion rate decrease with increasing temperature for medium used in

crude oil extraction. This phenomena help to use it at relatively high temperature. Fresh olive leaves extraction has 86% efficiency at 25°C, and efficiency increase with increasing temperature. From the EDS test found that the phenolic compound of the olive leaves extraction is responsible of inhabitation effect of the olive leaves [15].

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