Corrosion Inhibition of Organic Oil Extract of Leaves Of *Lanvandula Stoekas* on Stainless Steel in Concentrated Phosphoric Acid Solution

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The Corrosion inhibition effect of organic oil of leaves of *Lanvandula stoekas* [L] on corrosion of alloy UB6 stainless steel in 5.5 M H_3PO_4 solution was carried out using electrochemical polarisation and EIS methods. Experiments are performed by varying concentration of the inhibitor and temperature. The inhibition efficiency increases with increased organic oil concentration to attain a maximum value of 87.3 % at 1.2 g/L. The study reveals that [L] acts as a mixed inhibitor and showed a result of the growth in the compactness of the protecting film dressing. The inhibition efficiency of organic oil is temperature-dependent in the range 293 - 353 K, the associated activation energy has been determined. The organic oil [L] adsorbs on the stainless steel surface according to a Langmuir isotherm adsorption model.

Keywords: Corrosion, stainless steel, phosphoric acid, the electrochemical study, inhibitor

1. INTRODUCTION

Phosphoric acid in pure state is not very corrosive compared to nitric or sulphuric acids. Ninety-five percent of phosphoric acid is obtained by the wet process. The main stages in this process

involve the attack of phosphate ore by concentrated sulphuric acid, filtration and concentration of acid. This technique generates severe corrosion problems due to the presence of impurities such as chlorides, fluorides and sulphides [1–4] in concentric acid (5,5M). Many authors address the influence of these factors on the corrosion resistance of alloys [1-5, 15]. However, depending on the nature of phosphates and the type of phosphoric acid manufacturing process used, the equipment (reactors, agitators, pumps, drain, etc.) are subjected to slower or faster deterioration. In our laboratory, several works studied the corrosion of various materials in the phosphoric acid with addition of chemical ions [3, 17-19] and solid particles [14] and sometimes measurements were performed in the industrial phosphoric acid to study the behaviour in a real but complex medium [15,17,20].

The acids damage the materials under treatment and in this kind of situation; organic and inorganic inhibitors are used to reduce the corrosive property of acids [21-23]. Some of organic inhibitors available are considered to be very toxic and expensive. Therefore, attention has been focused on 'environment friendly green inhibitors' of plant origin, which are inexpensive, readily available and are renewable sources of materials [24-25].The protection of metals against corrosion in phosphoric acid has been the subject of much interest since it has been used in many industrial processes, especially in fertilizer production. Some investigations have also been done on the inhibition in phosphoric acid using synthetic inhibitors [26-28], but little works appear to have been done using naturally occurring substances in phosphoric acid as corrosion inhibitors [26, 29]. In previous work, lavender oil acted as a good ecofriendly inhibitor of corrosion mild steel in molar HCl [30].

The inhibition of corrosion in acid solutions can be secured by the addition of a variety of organic compounds and has been investigated by several studies [31-36], we propose in this work, the influence of addition of oil extracted from *Lanvandula stoekas* on inhibition of the corrosion of UB6 stainless steel in 5.5 M H_3PO_4 evaluated at various temperatures. Study of the kinetics of the corrosion of UB6 stainless steel in phosphoric acid was achieved by potentiodynamic polarization and electrochemical impedance spectroscopy methods.

2. MATERIALS AND METHODS

Preparation of specimen:

Ub6 stainless steel (C:0.0013 ;Ni:25.09 ;Cr: 20.77 ;Mo:4.39 ;Fe:43.97 ;Mn:1.84 ;Si:1.45 ;S;0.007 ;P:0.0029 others:2.36 Cu), The sample was polished with different emery papers up to 400-800 grades, then washed with distilled water and dried with blowing warm air, Electrode have circular shaped and the exposed area of the specimen to the solution was 0.6 cm^2 .The electrolyte used in this study was the phosphoric acid 5.5M H_3PO_4 with addition of 4% of H_2SO_4 and 400 ppm of chloride ions. In this last case, the solution was called polluted H_3PO_4 solution. Electrochemical measurements were carried out in a standard three-electrode cell with a platinum counter electrode and a saturated calomel reference electrode and electrode work. All potentials were given versus saturated calomel electrode (SCE). Potentiodynamic curves of alloy specimens in concentrated phosphoric acid in the

absence and in the presence of lavandula stoekas extract were obtained in the potential range from -0.6 to +1.3 V/SCE.

The experimental apparatus used for electrochemical studies is the PGZ 100 potentiostat, monitored by a PC computer and Voltamaster 4.0 software. Graphs were obtained in potentiodynamic mode with 5.10^{-4} V/s as a scanning rate of potential. The electrochemical impedance spectroscopy (EIS) were conducted in the frequency range of 100 kHz-100 mHz, with an amplitude signal of 10 mV peak to peak, The impedance diagrams are given in the Nyquist representation. Samples were tested at the temperatures of 20 to 80 °C. During each experiment, the test solution was mixed with a magnetic stirrer.

Preparation of plant extract

The leaves of *Lanvandula stoekas* were collected in the around region of Agadir. This plant extracted with hydrodistillation method to obtain an essential oil, which was subsequently used to study the corrosion inhibition properties of stainless steel in polluted phosphoric acid solution. The extracted oil was analyzed using Ultra Trace GC series gas chromatography and a Thermo-Fisher Scientific mass spectrometer.VP-5 capillary fused silica column (30m x 0.25mm, 0.25 µm film thickness) was used. Oven temperature was kept at 60°C for 2 min and programmed to 280°C at a rate of 16 °C/min and kept constant at 300°C for 20 min. The carrier gas was He (99.99%) at a rate of 1.4mL/min. The injection temperature and detector temperature were 220°C and 300°C respectively. The injection volume was 1mL with a split ration 1:25. EI/MS were taken at 70eV ionisation energy.

The qualitative and quantitative analysis by GC-MS identified almost more than 20 compounds, which are divided into groups following chemicals:

> Monoterpenes (α-pinene, sabinene, carene, ocimene ...)
> Sesquiterpenes (cadinene γ, caryophyllene oxide, 3.9 Cardina diene);
> Terpene alcohols (Linalool, Borneol ....);

Essential oils of lavender are characterized by major compounds namely:

> L-fenchone: 26, 05%.
> (+)-Epi-bicyclosesquiphellandrene: 24, 6%.
> Camphore: 15, 92%.
> Naphthalene, 1,2,3,4,4 a-hexahydro-1 0.7 0.6-dimethyl-4-(1methylethyl): 12.34%.

Following this analysis; the gas chromatography (GC)-mass spectrometry (MS) plant extract showed that the majority compound in the oil extracted is : L-fenchone.
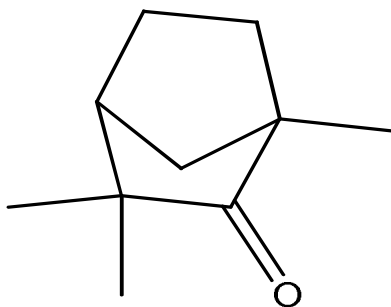
Figure 1. Chemical structure of L-fenchone

3-RESULTS AND DISCUSSION

3.1. Effect of inhibitor concentration

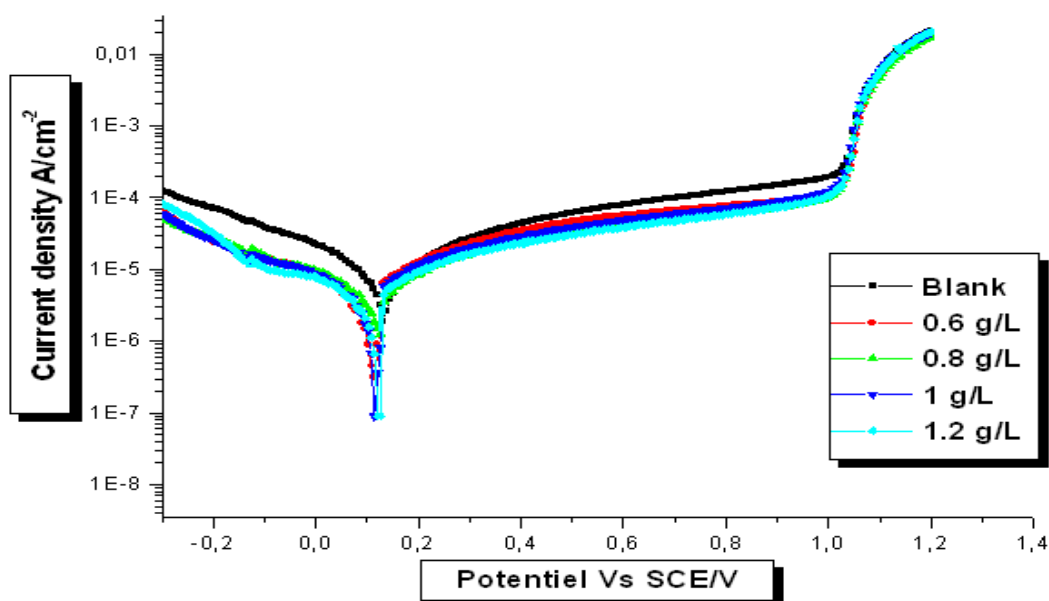### 3.1.1. Potentiodynamic Polarization Study

Figure 2. Polarisation curves of stainless steel in concentrated H3PO4 at different concentrations of organic oil [L]

The effect of organic oil concentration on the potentiodynamic anodic and cathodic polarization curves of stainless steel has been studied in 5.5M concentrated H_3PO_4 solution.

Electrochemical parameters such as corrosion current density (Icorr), corrosion potential (Ecorr), Tafel slope constants calculated from Tafel plots (β_c and β_a) and the inhibition efficiency (E %) are given in figure 2, Table 2.

It is obvious from the Fig.2, that in the presence of the inhibitor the curves are shifted towards lower current regions, showing the inhibition tendency of inhibitor that the cathodic current potentiel curves gave rise to parallel Tafel lines, which indicate that the hydrogen evolution reaction is activation controlled and that the addition of the [L] does not modify the mechanism of this process. Fig.2 also reveals that the presence of the inhibitor affects the anodic dissolution of stainless steel as well as the cathodic reduction of hydrogen ions,

For electrochemical measurements, the inhibition efficiency is calculated by using corrosion current density as follows:

$$E = \left(1 - \frac{i_{corr}}{i_{corr}^{\circ}}\right).100 \quad (1)$$

i_{corr} and i°_{corr} are the corrosion current density value with and without inhibitor, respectively, determined by extrapolation of cathodic Tafel lines to the corrosion potential.

Table 1. Electrochemical parameters of stainless steel in 5.5M H3PO4 in the presence of different concentrations of [F] at 298 K

Alloy	Blank	0.6g.L^{-1}	0.8g.L^{-1}	1g.L^{-1}	1.2g.L^{-1}
I(uA /cm2)	41.7	10.3	9.2	7	5.3
E(mV/SCE)	132	127.6	129	109.6	119
β_a (mV)	396.6	256.9	180.5	194.5	188.6
β_c(mV)	-360	-190.4	-231.1	164.9	-154.1
%E	-	75.2	77.9	83.2	87.3

The Potentiodynamic polarization parameters and inhibition efficiency are presented in Table2. The data show that increasing oil organic extracted concentration, decreases the corrosion current density (I_{corr}) and increases the inhibition efficiency of oil organic extracted. Both the anodic and cathodic tafel slopes, namely $ß_a$ and $ß_c$ respectively are varying from the blank values indicating the mixed mode of inhibition. E% increases with inhibitor concentration to attain a maximum value of 87.3 % at 1.2g.L^{-1}of [L].Therefore, it could be concluded that organic oil adsorbs onto both anodic and cathodic sites of the stainless steel surface.

3.1.2. Electrochemical impedance spectroscopy

More information about the metal/acidic solution characteristics is possible by using electrochemical impedance spectroscopy (EIS). EIS diagrams of stainless steel were plotted in

concentrated phosphoric acidic solution in the presence of organic oil extract [L] at 298 K. Fig. 3 shows Nyquist plots obtained in uninhibited and inhibited acidic solutions at different concentrations of organic oil [L].

The Nyquist diagram (Fig. 3) show that in the absence as well as presence inhibitor of concentration: 0.6 and 0.8g.L^{-1} can be seen that the plots exhibit depressed a single semicircles at high frequencies changing in a straight lines with slopes smaller at lower frequencies range indicating a Warburg impedance [37-39]. Such a diffusion process indicates that the corrosion mechanism is controlled not only by the charge transfer but also by the diffusion process of some relative ions [37,38,40-42].

In another hand, while increasing the concentration of inhibitor until 1.2g.L^{-1}, the Nyquist plots shows in this part only one capacitive loop in the entire frequency range. Without the appearance of a Warburg tail at low frequencies. The unfinished semicircle-arc was attributed to charge transfer process occurring at the metal/electrolyte interface or related to the surface passive film property [43, 55]. to account for the corrosion behavior of the stainless steel in concentrated phosphoric acidic solution with the presence of the inhibitor and to simulate the metal/acidic solution interface, an equivalent circuit model consists of solution resistance (Rs), double layer capacitance (C_{dl}), charge-transfer resistance (R_{ct}) has been shown in Fig.4 in which the C_{dl} and R_{ct} are parallel to each other.
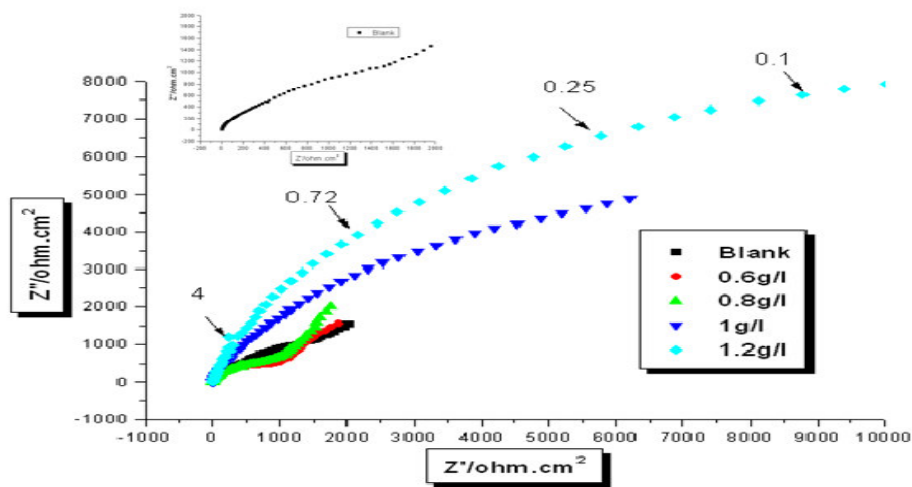

Figure 3. Nyquist diagrams for stainless steel in 5.5M H$_3$PO$_4$ containing different concentrations of [L].
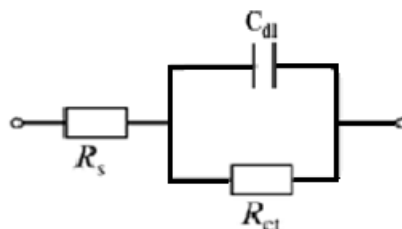

Figure 4. Equivalent circuit model for the corrosion of stainless steel in 5.5M H3PO4 at 298 K

Table 2. Impedance parameters and inhibition efficiency for the corrosion of stainless steel in 5.5M H3PO4 at various concentrations of [L] at 298 K

	Blank	0.6g.L^{-1}	0.8 g.L^{-1}	1 g.L^{-1}	1.2 g.L^{-1}
R_T (Ω cm^2)	204	529	560	736	1645
C_{dl} (μF cm^{-2})	173.8	150.2	131.6	107	98.7
% E	-	61.4	63.7	72.2	87.50

The parameters deduced are grouped in Table 2. Charge-transfer resistance values (R_t) and double layer capacitance values (C_{dl}) have been obtained from impedance measurements as described previously. The following relation is used to calculate E (%):

$$E\% = \left(1 - \frac{R_T^{\cdot}}{R_T}\right).100 \quad (2)$$

Where $R_T^{\cdot}$ R_T and are the charge-transfer resistance values with and without inhibitor, respectively.

Examination of Table 3 reveals that, more and more the [L] concentration increases, R_T rises to higher values showing that [L] inhibit corrosion reaction. The decrease of C_{dl} is interpreted by the adsorption of inhibitor molecules on the metal surface [56- 57]. The results obtained from the polarization technique in concentrated phosphoric acidic solution were in good agreement with those obtained from the electrochemical impedance spectroscopy (EIS) with a small variation.

3.2. Effect of temperature

The temperature can modify the interaction between the stainless steel electrode and the acidic medium in the absence and the presence of the inhibitor. Polarizations curves for stainless steel in 5.5M H$_3$PO$_4$ without and with organic oil [L] in the temperature range 293 – 353°K were recorded and the corresponding data are given in Table 3. In the studied temperature range, the corrosion current density increases with increasing temperature both in uninhibited and inhibited solutions and the inhibition efficiency of [L] decreases with temperature. The corrosion current density of stainless steel increases more rapidly with temperature in the absence of the inhibitor. These results confirm that [L] acts as a good inhibitor in the range of temperature studied. The corrosion reaction can be regarded as an Arrhenius-type process, the rate being given by:

$$I_{corr} = K. \exp\left(\frac{E_a}{RT}\right) \quad (3)$$

where k is the Arrhenius pre-exponential constant, and E_a is the activation corrosion energy for the corrosion process. Fig. 5 presents the Arrhenius plots of the logarithm of the corrosion current density vs. reciprocate of temperature, for 5.5 M H_3PO_4, without and with addition of [L]. The E_a values were determined from the slopes of these plots and are calculated to be Ea = 10.56 kJ/mol and 23.51 kJ/mol .While the higher value of the activation energy of the process in an inhibitor's presence when compared to that in its absence is attributed to its physical adsorption [58,61], its chemisorption is pronounced in the opposite case [62-64]. The higher value of E_a in the presence of [F] compared to that in its absence and the decrease of its E% with temperature increase can be interpreted as an indication of physical adsorption .

Table.3 the influence of temperature on the electrochemical parameters for stainless steel electrode immersed in 5.5 M H_3PO_4 and in 5.5 M H_3PO_4 + 1.2g.l-1 [L]

	1M H_3PO_4		1.2g.l^{-1}+H_3PO_4		%E
	E_{corr}	I_{corr}	E_{corr}	I_{corr}	
293K	132	41.9	119	5.3	87.3
313K	136.8	55.0	133	11.5	79.0
333K	156.8	72.9	175.6	19.5	73.2
353K	159.1	83.4	154	31.0	62.8

Physical adsorption is a result of electrostatic attraction between charged metal surface and charged species in the bulk of the solution. Positively charged species can also protect the positively charged metal surface acting with a negatively charged intermediate, such as acid anions adsorbed on the metal surface [65, 66].
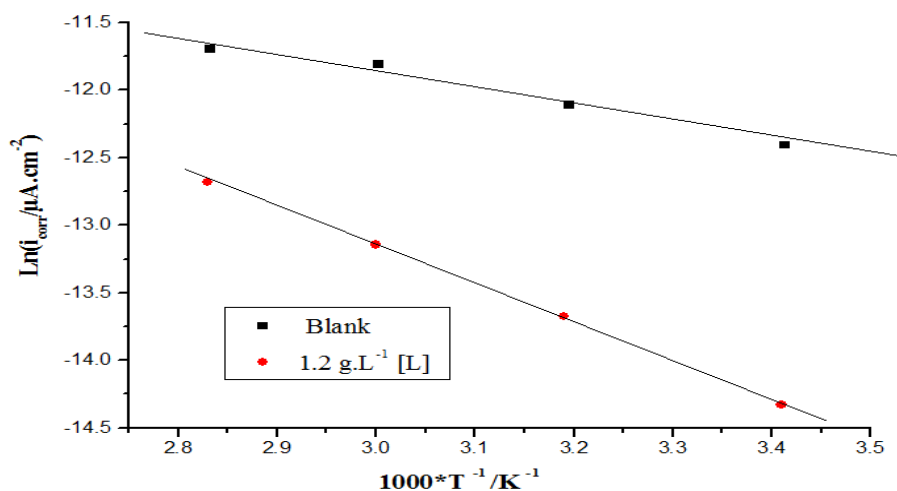

Figure 5. Arrhenius straight lines calculated from corrosion rate of stainless steel in 5.5M H_3PO_4 and 5.5 M H_3PO_4 + 1.2g.l^{-1} [L]

3.3. Adsorption isotherm

The degrees of surface coverage (θ) of different concentrations of [L] in 5.5M H_3PO_4, where θ is the ratio E (%)/100. The plot of C/θ versus ln C (Fig.6) shows a straight line indicating that the adsorption phenomenon of [L] on the stainless steel surface obeys to the Langmuir isotherm model:

$$K = \frac{1}{55.5} \exp\left(-\frac{\varDelta G^0}{RT}\right) \qquad (4)$$

The linear correlation coefficient ($R^2 = 0.9998$) is close to unity suggests that the adsorption of [L] on metal surface obeys well the Langmuir adsorption isotherm. The reciprocal of intercept of the straight lines C/θ axis is $K_{ads} = 138690$ L.g^{-1}. The use of such extract is so complex and contains infinite components; consequently, the determination of $\varDelta G^\circ_{ads}$ value has no meaning [67-69].
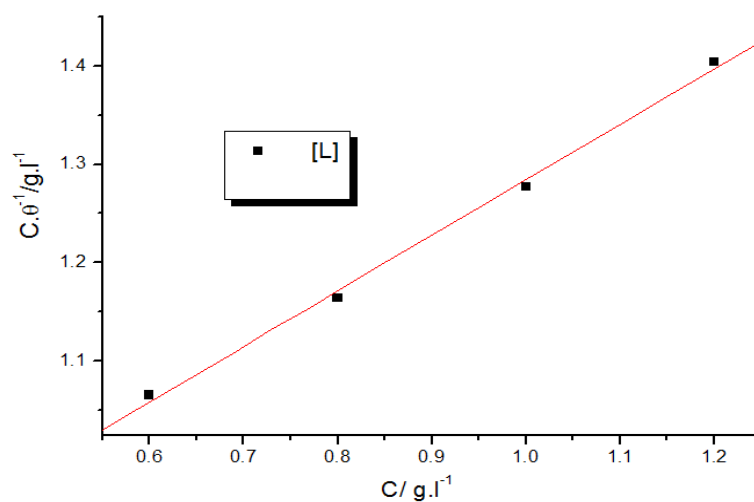

Figure 6. Langmuir isotherm adsorption model on the stainless steel surface of [L] in 5.5 M H3PO4.

4. CONCLUSION

- Electrochemical study showed that plant extract is a good corrosion inhibitor and acts as a mixed-type inhibitor in concentric phosphoric acid.
- The inhibition efficiency increases with increasing inhibitor concentrations to attain a maximum value of 87.5% for inhibitor [l] at 1.2g.$L^{-1.}$
- The inhibition efficiency of [L] decreases slightly with the temperature and the addition of [L] leads to increase of corrosion activation energy.
- [L] Adsorbs on the stainless steel surface according to the Langmuir isotherm.

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