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Corrosion inhibition of carbon steel by *Vitex agnus castus* leaves essential oils from the oasis of Tata

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Abstract: The corrosion inhibition of carbon steel in 1.0 M HCl solution of essential oils of *Vitex agnus castus* leaves obtained by hydrodistillation was studied using: weight loss, electrochemical impedance spectroscopy, and potentiodynamic polarization. The inhibition efficiency of the carbon steel attaints more than 79% at the concentration of 1g/L. This efficiency reaches 81.05% and 80.95%¨with the EIS and potentiodynamic polarization tests were used respectively. Polarization curves reveal that *Vitex agnus castus* essential oils were a mixed type inhibitor. Changes in impedance parameters (charge transfer resistance, R_t, and double-layer capacitance, C_{dl}) were indicative of adsorption of *Vitex agnus castus* essential oils on the metal surface leading to the formation of a protective film. The effect of the temperature on the corrosion behavior with the addition of the optimal concentration of *Vitex agnus castus* essential oils was studied in the temperature range 298– 328 K.

Keywords: *Vitex agnus castus,* Essential oils; Anticorrosion activity; Polarization *curves;* Adsorption.

1. Introduction

The oases of Tata, Morocco are located in the foothills of the Anti-Atlas and the Jbel Bani region. These oases, also known as the Bani oasis, have certain originality: they have a mountainous upwelling at rather low altitudes reaching 2531 m at the Adrar-n-Aklim north of the Oued Tata basin. Add to this the more southern latitude. These oases also have a relative diversity over very small distance. In return, the oases of Tata face difficult natural conditions and degradation of the environment: sustainable water resources very limited, very low quality of soil and plant resources solicited (Barathon *et al.,*2005; Abouri *et al.,* 2012). Chaste tree (*Vitex agnus castus* L.) native from arid and semi-arid Mediterranean and Western Asia is an aromatic, ornamental and deciduous shrub (Dogan *et al.,* 2011). Actually, *Vitex agnus castus* belongs to the *Lamiaceae* family (Ostovari *et al.,* 2009; Izionworu *et al.,*2020). In Morocco, *Vitex agnus castus* is widely distributed in the oasis of Tata. Corrosion is an important industrial problem: the cost of corrosion, which covers all means of fighting against

corrosion, the replacement of corroded parts or structures and the direct and indirect consequences of accidents due to corrosion, is estimated at 2% of the world gross product. Moreover, corrosion affects most industrial sectors and can cost billions of dollars each year (Hussain *et al.,* 2018). The means used against metal deterioration are synthetic products that are effective but highly toxic to humans and environment (Ostovari *et al.,* 2009; Izionworu*et al.,* 2020). Due to the toxicity of these corrosion inhibitors, there has been an increasing search for green corrosion inhibitors which are environmentally friendly (Ostovari *et al.,* 2009; Bentiss *et al.,* 1999; Obot et Obi-Egbedi 2010; Azzaoui *et al.,* 2017). Also, several studies have shown that essential oils have an excellent potential to inhibit corrosion in acidic environments.

2. Methodology

2.1 Collection Vitex agnus castus leaves and isolation of essential oils

The leaves of *Vitex agnus castus* were collected randomly in the oasis of Tata located in the southern east of Morocco during May-July 2015. Plant samples were air-dried in the shade then stored in the dark at 4°C. One hundred grams of air-dried leaves of *Vitex agnus castus* were subjected to hydro-distillation with a Clevenger-type apparatus according to the European Pharmacopoeia and extracted with one liter of distilled water for 3 hours. The essential oils obtained were dried under anhydrous sodium sulfate and stored at 4°C until use. The yield of the Essential oils was 0.21 % of dry weight (Asdadi *et al.,* 2020).

2.2 Anticorrosion activity

2.2.1 Materials and solutions preparation

The steel used in this study is a carbon steel (Euronorm: C35E carbon steel and US specification: SAE 1035) with a chemical composition (in wt %) of 0.380 % C, 0.230 % Si, 0.680 % Mn, 0.016 % S, 0.077 % Cr, 0.011% Ti, 0.059% Ni, 0.009% Co, 0.160 % Cu and the remainder iron (Fe). The aggressive solution of 1.0 M HCl was prepared by dilution of hydrochloric acid grade 37% (Merck) with bidistilled water. The concentrations range of *Vitex agnus castus* leaves essential oils employed were 0.05g/l to 1g/l. The *Vitex agnus castus* leaves essential oils were sonicated to obtain homogeneity before the anticorrosion test. The blank solution was prepared the same as above but without the essential oils (Batah *et al.,* 2017).

2.2.2 Weight loss measurements

Weight loss measurements are a first approach characterization of the inhibition of corrosion carbon steel samples with *Vitex agnus castus* leaves essential oils of leaves in hydrochoric medium (1.0 M HCl). Gravimetric measurements were carried out at a definite time interval of 6 hrs at room temperature using an analytical balance (precision ± 0.1 mg). Carbon steel specimens used have a rectangular form (2 × 2 × 0.08 cm^3). Gravimetric experiments were carried out in a double glass cell equipped with a thermo stated cooling condenser containing 80 mL of non-de-aerated test solution. After the immersion period, the steel specimens were withdrawn, carefully rinsed with bidistilled water, ultrasonic cleaning in acetone, dried at room temperature and then weighted. Triplicate experiments were performed in each case and the mean value of weight loss was calculated (Zarrok *et al.,* 2011).

2.2.3 Electrochemical tests

The electrochemical study was carried out using a potentiostat PGZ100 piloted by Voltamaster software. This potentiostat is connected to a cell with three electrodes thermostats with a double-wall (Tacussel Standard CEC/TH). A saturated calomel electrode (SCE) and platinum electrode were used as a reference and auxiliary electrodes, respectively. The material used for constructing the working

electrode was the same used for gravimetric measurements. The surface area exposed to the electrolyte is 0.04 cm^2 (Ziouche *et al.,* 2021). Potentiodynamic polarization curves were plotted at a polarization scan rate of 0.5 mV/s. Before all experiments, the potential was stabilized at free potential for 30 min. The polarization curves are obtained from −800 mV to −400 mV at 298 K. The solution test is there after de-aerated by bubbling nitrogen. Gas bubbling is maintained prior and through the experiments. In order to investigate the effects of temperature and immersion time on the inhibitor performance, some test was carried out in a temperature range 298–328 K.

The electrochemical impedance spectroscopy measurements are carried out with the electrochemical system (Tacussel), which included a digital potentiostat model Voltalab PGZ100 computer at E_{corr} after immersion in solution without bubbling. After the determination of steady-state current at a corrosion potential, sine wave voltage (10 mV) peak to peak, at frequencies between 100 kHz and 10 mHz are superimposed on the resting potential. Computer programs automatically controlled the measurements performed at rest potentials after 0.5 hour of exposure at 298 K. The impedance diagrams are given in the Nyquist representation. Experiments are repeated three times to ensure reproducibility. Inhibition efficiencies E_x (%) were calculated as follows:

- *For weight loss measurement:*

$$E_w (\%) = \frac{W^0_{corr} - W_{corr}}{W^0_{corr}} \times 100 \qquad \textbf{Eqn. 1}$$

Where W^0_{corr} and W_{corr} are the corrosion rates of steel due to the dissolution in 1.0 M HCl in the absence and the presence of definite concentrations of inhibitor, respectively.

- *For impedance measurements:*

$$E_{Rt} (\%) = \frac{R_t - R^0_t}{R_t} \times 100 \qquad \textbf{Eqn. 2}$$

Where R_t and R^0_t are the charge transfer resistance values with and without inhibitor, respectively.

- *For potentiodynamic polarisation measurements*:

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

Where I^0_{corr} and I_{corr} are the corrosion current densities in the absence and the presence of the inhibitor.

3. Results and Discussion

3.1 Effect of concentration

3.1.1 Gravimetric measurements

Weight loss methods are used to carry out the gravimetric measurements. This method of monitoring corrosion rate and inhibition efficiency is useful because of its simple application and high reliability (Bentiss *et al.,* 1999). Values of the inhibition efficiency and corrosion rate obtained by using this method of carbon steel for different concentrations of essential oils of *Vitex agnus castus* leaves in 1.0 M HCl at 298 K after 6 h of immersion are given in **Table 1.** The optimum concentration of inhibitor required to achieve the efficiency is found to be 1 g/L (Ew % = 79.53%). This inhibitor can be adsorbed on the metal surface by the interaction between the pairs of electrons of oxygen and the unsaturation of the inhibitor molecules and the metal surface. This process is facilitated by the presence of vacant orbitals of low energy in the iron atom, as observed in the transition group metals (Farssi *et al.,*

2020).The decrease in corrosion rate is likely due to the adsorption of molecules of the inhibitor on the surface metal and forming a molecular film layer or barrier between the metal and the corrosive medium according to Obot (Obot & Obi-Egbedi 2010)

Table 1. Corrosion rate and inhibition efficiency of carbon steel in 1.0 M HCl using *Vitex agnus castus* leaves essential oils at various concentrations.

Conc. (g / L)	**W_{corr} (mg. cm^{-2})**	**E_W (%)**
Blank	1.021	-
1.00	0.209	79.53
0,30	0.235	76.98
0,10	0.363	64.45
0,05	0.471	53.87

3.1.2 Electrochemical impedance spectroscopy measurements

The corrosion behavior of steel in acidic solution by *Vitex agnus castus* leaves essential oils was also investigated by the electrochemical impedance spectroscopy at 298K after 30 min of immersion. Nyquist plots obtained for frequencies ranging from 100 kHz to 10 mHz at open circuit potential for carbon steel in 1.0 M HCl in the presence of various concentrations of *Vitex agnus castus* leaves essential oils are shown in **Figure 1**. The impedance diagrams obtained are not perfect semicircles and the difference was attributed to frequency dispersion (Benali *et al.,* 2007) and the diameters of semicircles increases with the inhibitor concentration. The fact that impedance diagrams have a semicircular appearance shows that the corrosion of steel is controlled by a charge transfer process that takes place at the electrode/solution interface and the transfer process controls the corrosion reaction of carbon steel and the presence of inhibitor does not change the dissolution mechanism of carbon steel (Larabi *et al.,* 2004; Rosliza *et al.,*2008). The equivalent circuit model employed for this system is presented in **Figure 2**. The resistance R_s is the resistance of the solution; R_t reflects the charge transfer resistance and C_{dl} is the double layer capacitance. Values of the charge transfer resistance R_t were obtained from these Nyquist diagrams by determining the difference in the values of impedance at low and high frequencies (Dehri et Özcan 2006). Values of the double-layer capacitance C_{dl} were calculated from the frequency at which the impedance imaginary component $-Z_i$ is maximum using the equation:

$$f(-Zi_{max}) = \frac{1}{2\pi C_{dl} R_t} \quad \textbf{Eqn. 4}$$

Table 2 gives the values of double-layer capacitance C_{dl}, the charge transfer resistance R_t, and inhibition efficiency E_{Rt} (%). These results show that the presence of *Vitex agnus castus* leaves essential oils to reduce the C_{dl} values and enhances the values of R_t. The decrease in C_{dl} is due to the adsorption of *Vitex agnus castus* essential oils molecules function on the metal surface leading to the formation of a surface film in the acidic solution (Bentiss *et al.,* 1999). Thus, the decrease in C_{dl} values and the increase in R_t values and consequently of inhibition efficiency may be due to the gradual replacement of water molecules) by the adsorption of the oil molecules on the metal surface, decreasing the extent of dissolution reaction (Bentiss *et al.,* 1999; Muralidharan *et al.,* 1995). The efficiency reaches 81.05 % at 1.0 g/L.

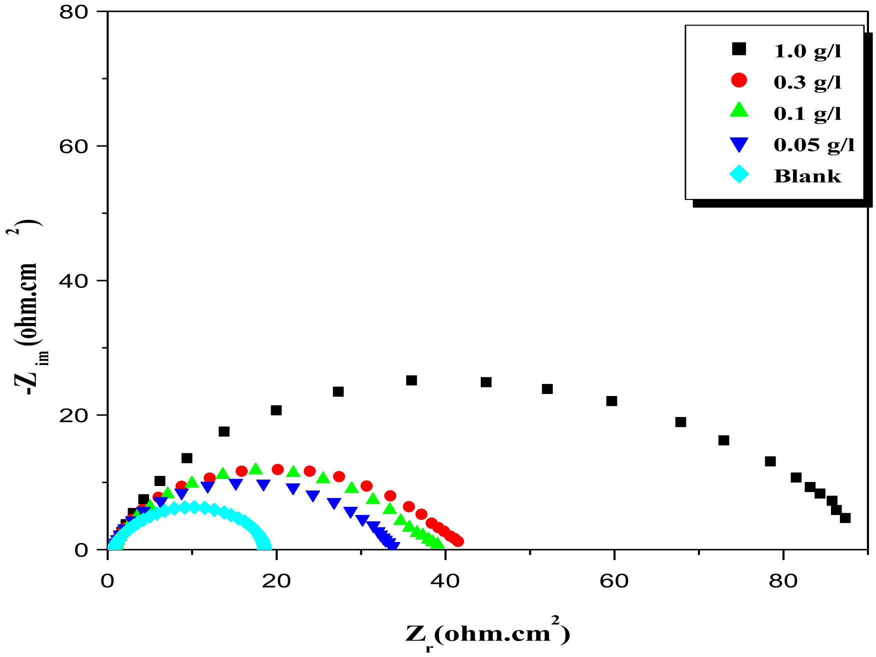

Figure 1. Nyquist diagram for Carbon steel electrode showing the effect of *Vitex agnus castus* leaves essential oils at E_{corr} after 30 min of immersion.

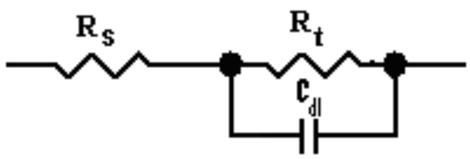

Figure 2. The electrochemical equivalent circuit used to fit the impedance spectra.

Table 2. Impedance parameters for corrosion of carbon steel in 1.0 M HCl at different concentrations of *Vitex agnus castus* leaves essential oils at 298 K

Conc.(g/L)	R_t(Ω.cm^2)	f_{max}(Hz)	C_{dl}(μF/cm^2)	E_{Rt} (%)
Blank	18	40	221.161	-
1.00	95	40	41.90	**81.05**
0.30	43	40	23.69	**58.14**
0.10	37	40	33.74	**51.35**
0.05	31	63	27.77	**41.93**

3.1.3 Potentiodynamic polarization study

Figure 3 shows the polarisation curves of Carbon steel in 1.0 M HCl and in the presence of different concentrations (0.02 – 0.5 g/l) of carob seed oil. To investigate the effect of *Vitex agnus castus* leaves essential oils on the mechanism of corrosion reaction of carbon steel, potentiodynamic polarization curve in HCl solution in the presence and the absence of various concentrations (0.05 – 1.0 g/l) of *Vitex agnus castus* leaves essential oils was plotted at 298K. With the increase of *Vitex agnus*

castus essential oil concentrations, both anodic and cathodic branches of the Tafel plot shift to lower values, at all investigated concentrations when inhibitors were added. This result indicated that the organic constituents of *Vitex agnus castus* leaves essential oils reduce the anodic dissolution of carbon steel and also retards the hydrogen evolution reaction. It is suggested that the *Vitex agnus castus* leaves essential oils act as a mixed type inhibitor.

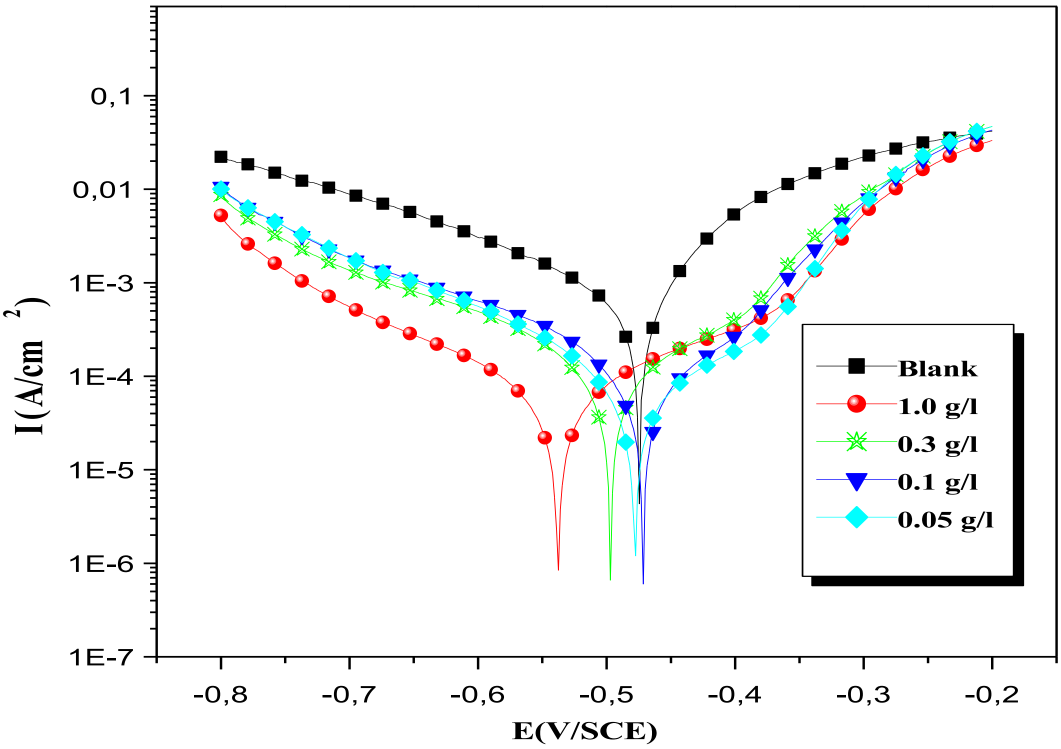

Figure 3. Tafel curves of carbon steel in 1.0 M HCl solution in the presence of *Vitex agnus castus* leaves essential oils.

Table 3 gives the values of kinetic corrosion parameters as the corrosion potential E_{corr}, corrosion current density I_{corr}, Tafel slope b_c, and inhibition efficiency for the corrosion of carbons teel in 1.0 M HCl with different concentrations of *Vitex agnus castus* leaves essential oils. From **Table 3,** it can be concluded that:

The magnitude of βc changed with the increments of *Vitex agnus castus* leaves essential oils inhibitor concentration; however, the change was not following any trend. This fact supported the random changes that occurred in *Ecorr* values along with the addition of the inhibitor.

The corrosion current densities I_{corr} values were estimated by Tafel extrapolation of the cathodic curves to the open circuit corrosion potential decrease with increasing inhibitor concentration.

The values of inhibition efficiency EI (%) which are given in **Table 3** increase with the addition of the inhibitor concentration signifying that the inhibitors effectively retarded the polarization process at the metal–acid interface; as a consequence, the corrosion process was inhibited (Ji *et al.,* 2015) and the value of EI (%) reached a maximum value (80.95%) at 1.0 g/L.

Table 3. Electrochemical parameters of carbon steel in 1.0 M HCl solution by *Vitex agnus castus* leaves essential oils at different concentrations.

Conc. (g/L)	E_{corr} (mV/SCE)	I_{corr} (µA/cm^2)	$-b_c$ (mV/dec)	E_I (%)
Blank	469	588	168	-
1.00	540	112	139	**80.95**
0.30	499	192	191	**67.35**
0.10	474	247	219	**57.99**
0.05	480	292	179	**50.34**

The results obtained from the polarization technique in acidic solution were in good agreement with those obtained from the electrochemical impedance spectroscopy:

3.2 Effect of temperature

Temperature can affect the interaction between the carbon steel electrode and the environment acid medium in the presence and absence of studied inhibitors. EIS measurements were taken at various temperatures in the absence and the presence of different concentrations of *Vitex agnus castus* leaves essential oils. Figs. 4 and 5 show the Nyquist plots of carbon steel by *Vitex agnus castus* leaves essential oils at different temperatures.

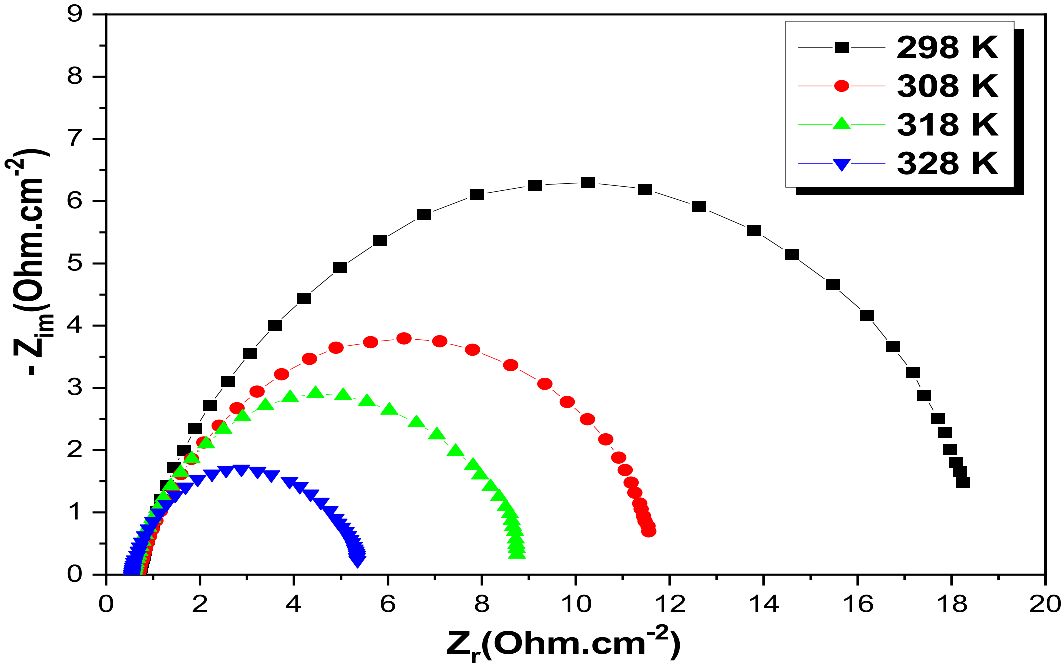

Figure 4. Nyquist diagrams for Carbon steel in 1 M HCl at different temperatures

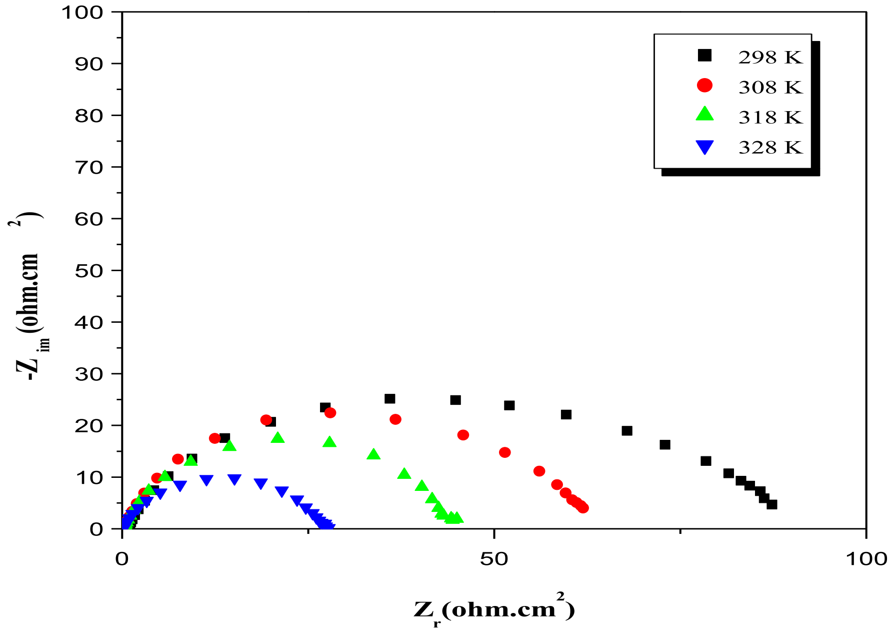

Figure 5. Nyquist diagrams for Carbon steel in 1 M HCl + 0.5 g/l of *Vitex agnus castus* leaves essential oils at different temperatures.

Corresponding data are given in **Table 4.** In the studied temperature range (298–328 K) the values of R_t decreases with increasing temperature both in uninhibited and inhibition efficiency for leaves of *Vitex agnus castus* leaves essential oils inhibitor increase slightly in the range of temperatures studied. Generally, the corrosion rate of steel in acid solution increase with raising temperature. This is due to the decrease of hydrogen evolution over potential (Ji *et al.,* 2015).

Table 4. Thermodynamic parameters for the adsorption of *Vitex agnus castus* leaves essential oils in 1.0 M HCl on the carbon steel at different temperatures.

Inhibitor	Temp. (K)	R_{ct} (Ω.cm^2)	f_{max}(Hz)	C_{dl}(µF/cm^2)	E.I(%)
Blank	298	18	40	221.16	-
	308	11	63	229.78	-
	318	8	100	199.04	-
	328	5	158	201.56	-
Essential oil	298	95	40	41.90	**81.05**
	308	60	50	53.08	81.66
	318	43	63	58.78	81.39
	328	27	63	93.61	81.48

On the other hand, the values of R_t were employed to calculate values of the corrosion current density (I_{corr}) at various temperatures in the absence and presence of *Vitex agnus castus* leaves essential oils using the following equation (Mansfeld *et al.,* 1982):

$$I_{corr} = R.T.(z.F.R_t)^{-1} \qquad \textbf{Eqn. 5}$$

Where R is the universal gas constant (R = 8.31 $J.K^{-1}.mol^{-1}$), T is the absolute temperature, z is the valence of iron (z = 2), F is the Faraday constant (F = 96485 coulomb) and R_t is the charge transfer resistance.

The activation parameters for the corrosion process were calculated from Arrhenius type plot according to the following equation:

$$\log I = -\frac{E_a}{2.303\,R\,T} + \log A \qquad \textbf{Eqn. 6}$$

Where E_a is the apparent activation energy, A is the pre-exponential factor, R the universal gas constant and T the absolute temperature.

The variations of the logarithm of the ln (Icorr) of carbon steel in HCl containing 1.0 g/L of L Leaf of *Vitex agnus castus* leaves essential oils used with reciprocal of the absolute temperature are presented in **Figure 6.**

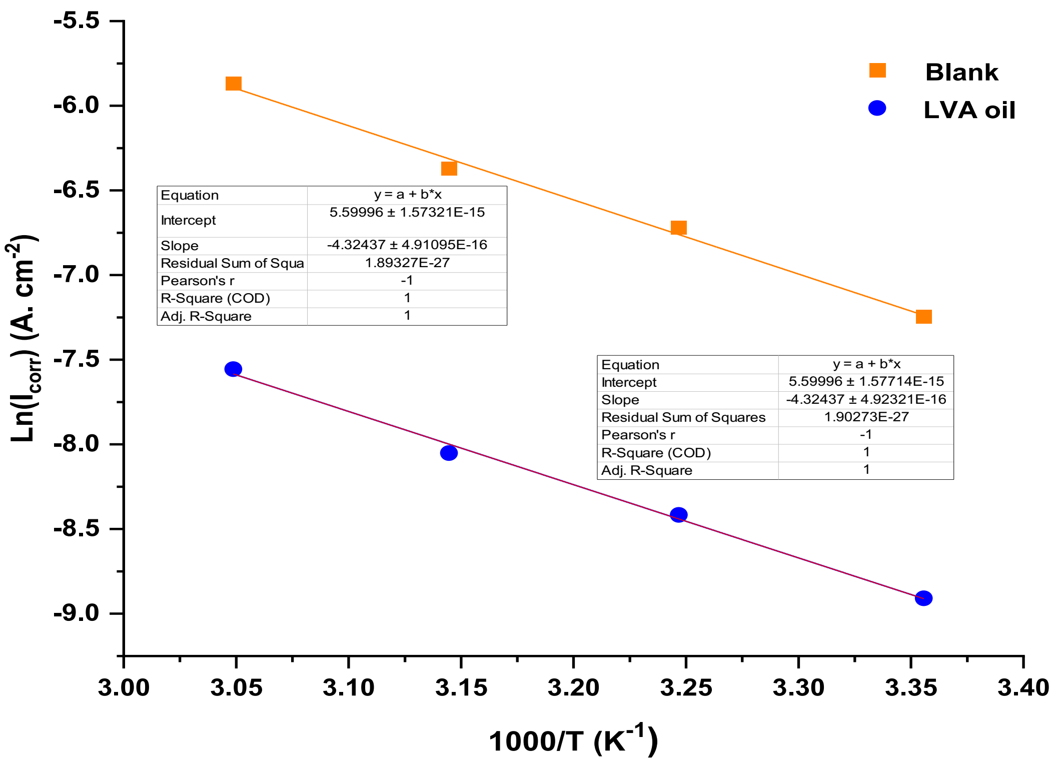

Figure 6. Arrhenius plots of Carbon steel in 1.0 M HCl by 1 g/l *Vitex agnus castus* leaves essential oils

The value of Ea presented in **Table 5** indicates that the value determined in 1.0 M HCl containing Leaves of *Vitex agnus castus* leaves essential oils is slightly less than that for an uninhibited solution. The variation of the Ea values can be attributed to the positions of the inhibitor molecules on the metal surface (Tebbji *et al.,* 2007; Behpour *et al.,* 2012). The relationships between the temperature

dependence of percentage inhibition efficiency IE of an inhibitor and the activation energy Ea can be classified into three groups according to temperature effects (Sathiya Priya *et al.,* 2008):

(i) IE decreases with increase in temperature, Ea (inhibited solution) >Ea (uninhibited solution);
(ii) IE increases with increase in temperature, Ea (inhibited solution) <Ea (uninhibited solution);
(iii) IE does not change with temperature, Ea (inhibited solution) = Ea (uninhibited solution)

Activation parameters such as enthalpy and entropy of corrosion process may be evaluated from the temperature effect. An alternative formulation of Arrhenius equation is (Benabdellah *et al.,* 2006; Ouali *et al.,* 2010; Dahmani *et al.,* 2010):

$$I_{corr} = \frac{R.T}{Nh}.\exp(\frac{\Delta S^*}{R}).\exp(-\frac{\Delta H^*}{RT}) \qquad \textbf{Eqn. 7}$$

Where h is plank's constant, N is Avogado's number, ΔS*and ΔH*are the entropy and enthalpy of activation, respectively.

Table 5. The value of activation parameters E_a, ΔH* andΔS*for carbon steel in 1.0M HCl in by 1.0 g/L of *Vitex agnus castus* leaves essential oil

Inhibitor	**E_a(kJ/mol)**	**ΔH^*(kJ/mol)**	**ΔS^*(J/mol)**	**E_a -ΔH^*(KJ/mol)**
Blank	36.38	33.79	-191.53	2.60
Essential Oil	35.28	32.69	-209.38	2.60

Straight lines are obtained with a slope (-ΔH* /R) and intercept (Ln R/Nh + ΔS* /R) from which the ΔH* and ΔS* values are calculated **Table 5.** The positive sign of the enthalpy (ΔH*) reflects the endothermic nature of the steel dissolution process indicating that the dissolution of carbon steel is slow in the presence of *Vitex agnus castus* leaves essential oils (Mu, Li, et Li 2004). The large negative value of ΔS* for carbon steel in 1 M HCl implies that the activated complex is a rate-determining step, rather than the dissociation step.

The linear regression coefficients are close to one, indicating that the corrosion of carbon steel in 1.0 M HCl solution may be elucidated using the kinetic model. A close inspection of the data in **Table 6** shows that the activation energy is lower in the presence *Vitex agnus castus* leaves essential oil **(Figure 7).** The decrease of E_a is typical of the chemisorption process (Merah *et al.,* 2008). According to Equation 6, low values of A and high values of E_a lead to lower corrosion rates.

3.3 Adsorption Isotherm

The adsorption behavior of the inhibitor molecules on the electrode surface can be used to elucidate the inhibition mechanism of inhibitors. Adsorption isotherm can provide more information on the interaction between the metal surface and the inhibitor essential oil. The surface coverage values (θ) determined by weight loss method **(Table 1)** at different concentrations of *Vitex agnus castus* leaves essential oils inhibitor in acidic solution were used to explain the excellent adsorption isotherm, consequently, establish the adsorption process from the experimental results obtained. To investigate the inhibitor adsorption mechanism, several adsorption isotherms were fitted and the Langmuir adsorption isotherm gives the best description of the adsorption behavior of the inhibitor on carbon steel electrode according to Langmuir adsorption isotherm equation:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad \textbf{Eqn. 8}$$

Where C_{inh} is the inhibitor concentration and K_{ads} is the adsorptive equilibrium constant. The linear relationship of C_{inh}/θ versus C_{inh} in different concentrations of inhibitor presented in **Figure 8**, possesses a slope near to unit with the correlation coefficient R^2 that is close to 1, thus, the adsorption of *Vitex agnus castus* leaves essential oils on steel surface obeys the Langmuir adsorption isotherm.

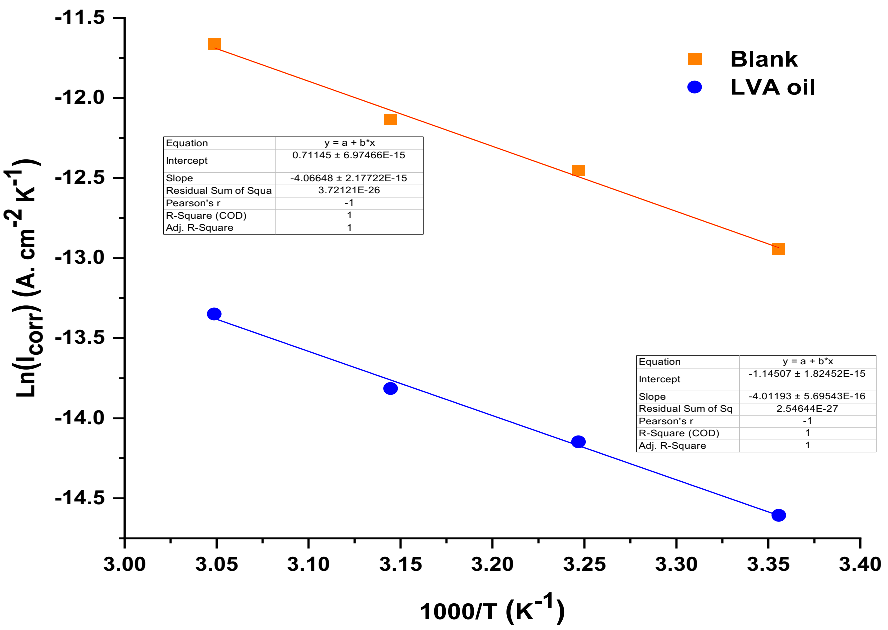

Figure 7. Relation between Ln (Icorr/T) and 1000/T at different temperatures.

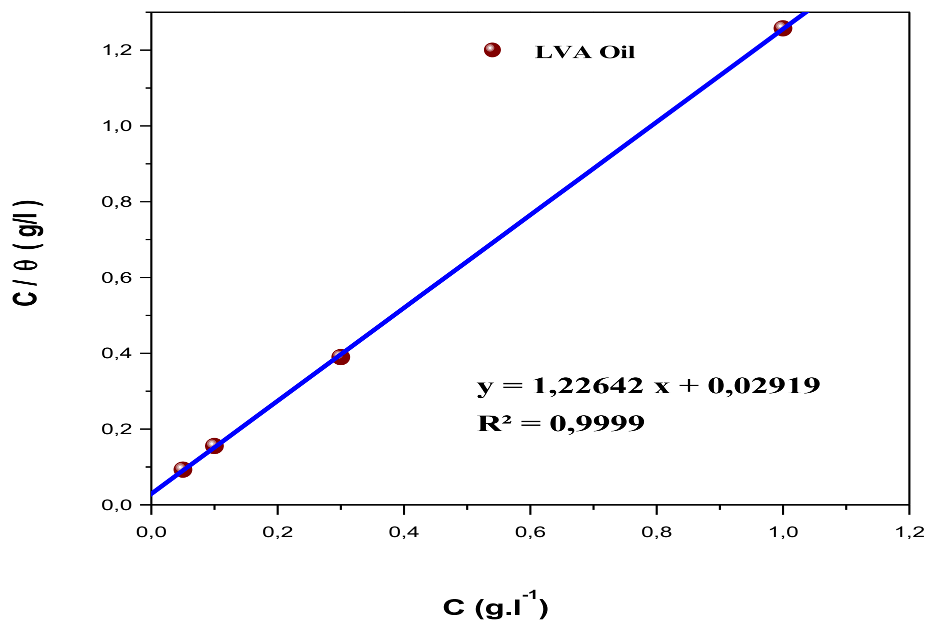

Figure 8. Langmuir adsorption of *Vitex agnus castus* leaves essential oils on the carbon steel surface in 1.0 M HCl solution at 298K.

Agreement was determined by examining inhibition efficiency (E %) values obtained by weight loss, EI and EIS methods (**Figure 9**).

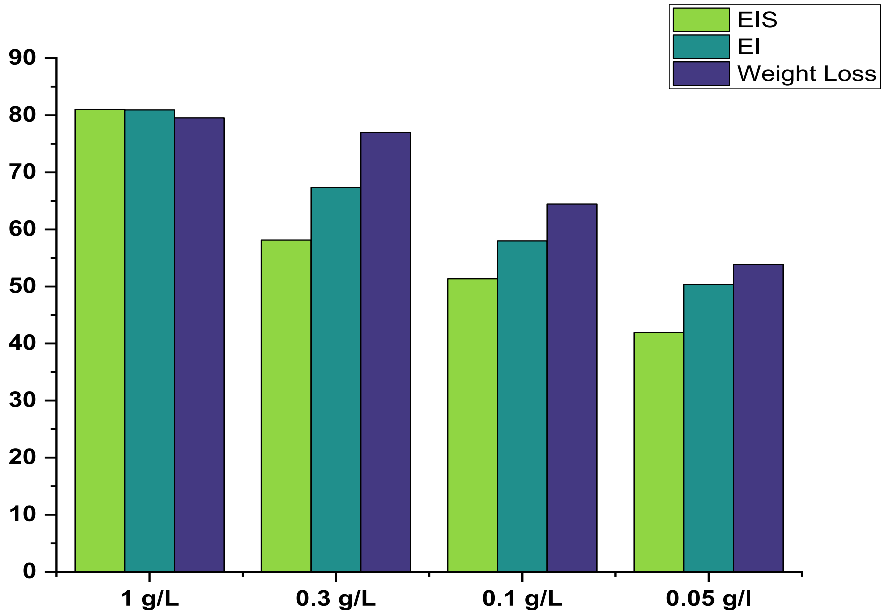

Figure 9. Comparison of inhibition efficiency (E %) values obtained by weight loss, EI and EIS methods.

Conclusion

Vitex agnus castus leaves essential oils turn to be an eco-friendly inhibitor for carbon steel in 1.0 M HCl solution. From the experimental results the following conclusions can be deduced: that chemical analysis shows that the oil and that *Vitex agnus castus* leaves essential oils mainly act as good inhibitors the for the corrosion of carbon steel in 1.0 M HCl. Also, the inhibition efficiency increases with the concentration of inhibitor.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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