Influence of *Lavandula Dentata* essential oil on the corrosion inhibition of carbon steel in 1 M HCl solution

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Abstract

Within this study we are concerned by the corrosion inhibition of the essential oil of *Lavandula Dentata* (EOLD) in carbon steel in 1 M hydrochloric acid solution, the investigation was performed using gravimetric method, Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic polarization measurements. The results of electrochemical techniques and gravimetric measurements revealed that the EOLD tested as inhibitor leads to a systematic reduction in the kinetics of steel corrosion processes. Potentiodynamic polarization study has shown that EOLD is inhibitor of a cathodic type. The effect of temperature on the corrosion behavior of carbon steel in the presence and absence of EOLD was studied in the temperature range from 303 K to 333 K. The findings about Electrochemical Impedance Spectroscopy revealed a frequency distribution of the capacitance, simulated as constant phase element. Inhibition efficiencies, determined by potentiodynamic polarization, electrochemical impedance spectroscopy and weight loss methods, are in good agreement. The extracted of EOLD, by hydrodistillation, is analyzed by gas chromatography (GC), indeed, this technique of analysis has shown that its major component is: camphor (50.3%). The EOLD inhibitor adsorbed physically on the carbon steel surface obeys the Langmuir adsorption isotherm. The parameters of activation energy was fairly evaluated and discussed in this work.

Keywords: corrosion, *Lavandula Dentata*, inhibition, weight loss, polarization.

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Introduction

The corrosion is a phenomenon that can never be stopped but it will be possible to combat their effects. The use of inhibitors is one of the best methods aiming to protect metals against corrosion, especially in acid medium. Most corrosion inhibitors are organic compounds; they act by getting adsorbed on the metal surface and then block the active sites and improve the adsorption process [1–3]. In general, the adsorption is described by

two main types of interaction, namely physical adsorption and chemisorption. It depends on the charge of the metal and its nature, the chemical structure of the organic product and type of electrolyte. However, the majority of these compounds are not only expensive but also toxic for living organisms and environment. The toxic nature of these organic inhibitors has required research activities in recent times toward finding new alternative environmentally friendly acid corrosion inhibitors [4–8]. Recently, natural compounds have attracted much attention because they are recognized as environmentally friendly corrosion inhibitor of metal and alloys. These kinds of inhibitors are obtained from natural products such as plant extracts and essential oils [9–11]. It's worthy to note that in the last decade the most increasing concern is about green corrosion inhibitors that have been found to be of great efficiency against steel corrosion in acid media, this way of protection is the main subject of our work that consist of the examination of the inhibition effect of extracted oil from *Lavendula* on corrosion of the steel xC38 to 1 M hydrochloric acid. In this paper we have focused our attention to essential oil of *Lavandula Dentata* (EOLD) as corrosion inhibitor for steel in HCl media, our study has been performed by weight loss and electrochemical methods. Discussion is based on the analysis of oil chemical composition.

1. Materials and methods

1.1 The essential oil

The studied essential oil was firstly extracted from the flowers and dry stalks of *Lavandula Dentata*. To protect it from the air and the light it has been stored under average temperature of 4°C, in a dark glass flask hermetically closed. The extraction of essential oil of was conducted by hydrodistillation using a Clevenger type apparatus, and the obtained essential oil was dried under anhydrous sodium sulfate.

1.2 Preparation of the solution

The HCl 1 M solution was prepared by dilution of HCl 37% of analytical quality with bidistilled water. The test solutions were freshly prepared before every experiment by adding the oil directly to the corrosive solution.

1.3 Weight loss tests

The steel alloy having a composition (0.370% 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 S and the element Fe which is majority takes the rest of these percentages) are used for the measurements of weight loss. Before any measurement, the steels have been mechanically abraded with abrasive papers to different grit sizes (180, 320, 800, and 1200). The samples are washed with bi-distilled water, and dried. The concentration range of EOLD used in the corrosion tests was 0.6 to 3 g/L. After six hours, the sample was collected, washed, dried and weighed with precision.

1.4 Electrochemical tests

The electrochemical study was realized by means of a potentiostat of PGZ100 piloted by Volta Master software. Potentiodynamic polarization curves were plotted at a scanning rate of 2 mV/s. The polarization curves are obtained from -800 mV to -300 mV. In order to study the impact of the temperature, the tests have been realized in a temperature range of 303–333 K. The measurements of electrochemical impedance spectroscopy have been performed at frequencies in the range between 100 kHz and 10 MHz. The diagrams of impedance are given in the Nyquist and Bode representation. The electrochemical experiments are performed in a Pyrex cell equipped with a conventional assembly with three electrodes, steel as working electrode (WE), platinum as auxiliary electrode (AE) and saturated calomel electrode $\text{Hg}/\text{Hg}_2\text{Cl}_2/\text{KCl}$ (SCE) as reference electrode.

2. Result and discussion

2.1 Composition of the essential oil

The analysis of the essential oil of *Lavandula Dentata* was performed using CG to allow the identification of 37 components, which accounted for 89% in the total of oil. The retention time of the volatile compounds and their percentage are summarized in Table 1.

Table 1. Composition of essential oil of *Lavandula Dentata*.

N	Compounds	RI apol	RI pol	HE% apol	RI Bed
1	α -Pinene	929	1021	0.1	936
2	Camphene	943	1065	0.2	950
3	1-Octen-3-ol	959	1439	0.3	962
4	β -Pinene	970	1109	0.5	978
5	<i>p</i> -Cymene	1011	1265	0.1	1015
6	1,8-Cineole	1020	1207	1.9	1024
7	Limonene	1020	1197	0.5	1025
8	<i>o</i> -Cresol	1035	1919?	0.2	1037
9	Linalool oxide E THF	1056	1432	1.0	1058
10	Fenchone	1067	1389	0.7	1069
11	Linalool oxide Z THF	1071	1460	0.8	1072
12	Linalol	1085	1536	2.2	1086
13	Hotrienol	1087	1613	0.2	1083
14	α -Campholenal	1103	1479	0.2	1105
15	Nopinone	1105	1571	0.5	

N	Compounds	RI apol	RI pol	HE% apol	RI Bed
16	Pinocarveol	1108	1651	0.7	
17	<i>trans</i> -Pinocarveol	1125	1641	6.2	1126
18	Camphor	1125	1508	50.3	1123
19	<i>cis</i> -Verbenol	1129	1647	0.3	1132
20	<i>p</i> -Mentha-1,5-dien-8-ol (isomer I)	1130	1651	0.2	1127
21	Pinocarvone	1139	1555	1.1	1137
22	<i>p</i> -Mentha-1,5-dien-8-ol (isomer II)	1146	1717	0.8	
23	Borneol	1150	1686	3.0	1150
24	<i>p</i> -Methylacetophenone	1154	1752	0.5	1157
25	Cryptone	1156	1663	0.7	1160
26	<i>p</i> -Cymen-8-ol	1160	1829	1.2	1169
27	Myrtenal	1170	1613	1.6	1172
28	α -Terpineol	1172	1686	0.4	1176
29	Myrtenol	1179	1776	1.6	1178
30	Verbenone	1180	1681	0.5	1183
31	<i>trans</i> -Carveol	1197	1816	0.7	1200
32	Cuminaldehyde	1211	1761	0.5	
33	Carvone	1215	1715	0.6	1214
34	β -Selinene	1479	1708	1.5	1486
35	Caryophyllene oxide	1567	1962	1.5	1578
36	β -Eudesmol	1634	2206	4.1	1641
37	β -Bisabolol oxide	1639	2105	0.8	
38	<i>cis</i> -14-nor-Muurool-5-en-4-one	1659	2254	0.3	
39	α -Bisabolol	1664	2194	0.5	1673
40	Cryptomerione	1699	2255	0.4	
TOTAL				89.4	

RI Bed = retention indices on the apolar column of literature (König et al., 2001; National Institute of Standards and Technology, 2008).

RI apol = retention indices on the apolar column (Rtx-1).

RI pol = retention indices on the polar column (Rtx-Wax).

The oil was dominated by oxygenated monoterpenes (70%), followed by oxygenated sesquiterpenes (15%) and monoterpene hydrocarbons (12.5%). The sesquiterpene hydrocarbons accounted only for (2.5%) of the total oil.

The essential oil was characterized by a high content of camphor (50.3%) followed by other components with relatively small amounts: trans Pinocarveol (6.2%); β -Eudesmol (4.1%); Borneol (3.0%); Linalol (2.2%). The 30 other compounds are present in the essential oil of (L.D) with low quantities.

Several studies have been published on chemical composition of different types of *Lavandula* [12].

Verma *et al.* have studied the essential oil composition of *Lavandula officinalis* flowers grown in Uttarakhand (India), they identified 37 monoterpene compounds. The major components were: linalyl acetate (47.56%), linalool (28.06%), lavandulyl acetate (4.34%) and α -terpineol (3.7%) [13].

Garsia-Vellejo *et al.* have also studied the essential oil composition of *Lavandula angustifolia subsp. pyrenaicia* in Guinea. The main component was linalool (20.1-65.9%), followed by borneol (6.3-32%) and camphor (2.4-13.5%)[14].

The *Lavandula dentata* studied in Tunisia and Algeria was predominated by 1,8-cineole (38.4 and 33.5%, resp.) [15].

In general, this variation in the chemical composition can be understandable according to exogenous factors: the period of sunshine, the nature and the composition of the ground.

2.2 Gravimetric measurements

The mass loss measurement of the steel in a solution HCl (1 M) in the absence and in the presence of different concentrations of OLD allows assessing the corrosion rate by the following relationship [16]:

$$W = \Delta m / (S \cdot t) \quad (1)$$

Where: Δm is the loss of mass expressed in mg; t is the time of immersion in h; S is the surface of the specimen in cm^2 .

In the case of the weight loss method, the inhibition efficiency ($E\%$) was determined by the following relation:

$$E\% = \frac{(W_0 - W_{\text{inh}})}{W_0} \cdot 100 \quad (2)$$

Where: W_0 is the corrosion rate in absence of inhibitor and W_{inh} stands for the corrosion rate in the presence of inhibitor.

The results obtained at ambient temperature according to the method described in the experimental part are summarized in Table 2.

Weight loss results allowed us to calculate the mean corrosion rate; it is obvious that the corrosion rate decreases while the inhibition efficiency increases with increasing

inhibitor concentration reaching a maximum value of 90% at concentration of 2.4 g/L at 303 K.

Table 2. Efficiency and corrosion rate in the absence and presence of different concentrations of EOLD at 303 K.

Concentration (g/l)	Δm (mg)	W (mg/cm ² ·h)	E (%)
blank	30.4	0.81	—
0.6	17.8	0.50	39
1.2	11.7	0.31	62
1.8	6.6	0.18	78
2.4	2.9	0.08	90

2.3 Polarization study

The use of electrochemical methods (polarization curves, electrochemical impedance spectroscopy) is essential in order to better understand the mechanisms involved. Figure 1 represents the polarization curves of the carbon steel electrode in 1 M HCl in the absence and in the presence of various concentrations of EOLD.

Figure 1. Polarization curves for carbon steel in 1 M HCl containing various concentrations of EOLD at 303 K.

The corrosion parameters including corrosion potential (E_{corr}), corrosion current density (I_{corr}), inhibition efficiency ($E\%$) and the surface coverage degree θ are gathered in Table 3.

Table 3. Electrochemical parameters for carbon steel in 1 M HCl at different concentrations of EOLD at 303 K.

CC (g/l)	<i>E</i> (mV)	<i>I</i> _{corr} (μA/cm ²)	<i>E</i> %	θ
Blank	–366	600	–	–
0.6	–385	213	64	0.64
1.2	–413	125	79	0.79
1.8	–440	39	93	0.93
2.4	–523	30	95	0.95

The efficiency *E*% was calculated from polarization measurements using the following relation:

$$E\% = \frac{(I_{\text{corr}} - I_{\text{corri}})}{I_{\text{corr}}} \cdot 100 \quad (3)$$

The surface coverage degree θ due to the adsorption of EOLD of the metal on the surface was deduced using the following relation:

$$\theta = \frac{(I_{\text{corr}} - I_{\text{corri}})}{I_{\text{corr}}} \quad (4)$$

Where *I*_{corr} and *I*_{corri} are the corrosion current densities for steel electrode respectively in the uninhibited and inhibited solutions. These values of the corrosion potential were determined by extrapolating the Tafel lines.

The parallel cathodic Tafel plots obtained in Figure 1 indicates that the reduction mechanism is not influenced in by the presence of inhibitor. The addition of EOLD has a pronounced inhibitive effect on the cathodic part. This indicates that EOLD is a cathodic inhibitor type and the adsorption process of the molecules in the tested inhibitor is occurring on the cathodic sites which leads to cathodic inhibition reaction [17]. The electrochemical data extracted from the polarization curves presented in Table 3 indicate that the values of *E*_{corr} shifted in the negative direction and the corrosion current densities (*I*_{corr}) values decreased obviously in the presence of inhibitor. The values of inhibition efficiency increases with increasing of EOLD concentration. The maximal protection was about 95% at 2.4 g/l.

2.4 Effect of temperature

The increase of temperature on inhibition of corrosion shows that several phenomena occur on the electrode surface such as desorption of the inhibitor or molecules whose inhibitor can themselves undergo either decomposition or rearrangement [18]. The study of effect of temperature on the corrosion inhibition of carbon steel in 1 M HCl was investigated by potentiodynamic polarization technique in the temperature range from 303 to 333 K.

Figure 2 shows that the increase of the temperature have a slight effect on the corrosion potentials; but leads to a higher corrosion rate (I_{corr}). The extracted results of potentiodynamic curves are shown in Table 4.

Table 4. Electrochemical parameters for carbon steel in 1 M HCl at different concentrations of EOLD at 303 K.

CC (g/l)	T (K)	E (mV)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	$E\%$
Blank	303	– 368	600	–
	313	–423	815	–
	323	–417	1334	–
	333	–430	2010	–
2.4 g/L	303	–437	30	95
	313	–502	50	94
	323	–543	210	84
	333	–551	344	83

From this result, it can be concluded that, on the temperature increase, the current density values increases in both solutions, and the value of inhibition efficiency slightly decreases in favor of temperature increasing. This can be explained by the desorption of some molecules from the metal surface by increasing the temperature [19, 20], in spite of this decrease the EOLD remains as an a good inhibitor at 333 K, it reaches 83% at concentration of 2.4 g/l.

Figure 2. Polarisation curves for carbon steel in 1 M HCl + 2.4 g/l EOLD at various temperatures.

2.5 Electrochemical impedance spectroscopy (EIS)

Electrochemical impedance spectroscopy measurement has been used to study corrosion inhibition processes. It enables investigating the resistive and capacitive behavior at the interface [21].

The Nyquist diagram with and without our inhibitor study shows only one depressed capacitive loop at the higher frequency range (HF) and one capacitive time constant in the Bode-phase plot (Figure 3). This observation indicates that the corrosion of carbon steel in acidic solution is controlled by a charge transfer process.[22, 23]

Also, the diameter of the depressed semicircle increases gradually with the increase of the EOLD concentration and consequently the inhibition efficiency increases.

The plots of impedance diagrams are not perfect semicircles this can be explained by the frequency dispersion effect as a result of inhomogeneous of electrode surface.

The results of EIS are simulated by the equivalent circuit shown in Figure 4. In this equivalent circuit, R_s is the solution resistance, CPE is the constant phase element and R_{ct} present the charge transfer resistance whose value is a measure of electron transfer across the surface. The constant phase element is composed of a component Q_{dl} and a coefficient n . The parameter n quantifies the surface inhomogeneousness resulting from inhibitor adsorption, impurities, formation of porous layers, *etc.* [24, 25].

Figure 3. EIS Nyquist diagrams and Bode-phase plots of carbon steel in 1 M HCl without and with different concentrations of EOLD at 303 K.

Figure 4. Electrical equivalent circuit used for modeling the interface carbon steel/1 M HCl solution without and with EOLD.

So the capacitance is deduced from the following relation:

$$C_{dl} = \sqrt[n]{Q \cdot R_{ct}^{1-n}} \quad (5)$$

Where Q is the constant phase element (CPE) and n is a coefficient of surface inhomogeneity.

The impedance of the CPE is expressed as:

$$Z_{CPE} = Q^{-1}(i\omega)^{-n} \quad (6)$$

Where ω is the angular frequency (in rad s^{-1}) and $i^2 = -1$ is the imaginary number.

Table 5. Impedance parameters for corrosion of carbon steel in 1 M HCl without and with different concentrations of EOLD at 303 K.

CC (g/l)	R_s ($\Omega \cdot \text{cm}^2$)	R_{ct} ($\Omega \cdot \text{cm}^2$)	n	Q ($S^n / \Omega \cdot \text{cm}^2$)	C_{dl} ($\mu\text{F} \cdot \text{cm}^{-2}$)	E (%)
Blanc	0.62	24.42	0.813	$3.19 \cdot 10^{-4}$	127.39	—
0.6	0.75	41.9	0.812	$2.29 \cdot 10^{-4}$	86.89	42
1.2	1.22	85.98	0.799	$1.71 \cdot 10^{-4}$	70.89	72
1.8	0.74	133.12	0.804	$1.39 \cdot 10^{-4}$	58.58	82
2.4	2.12	181.9	0.794	$1.18 \cdot 10^{-4}$	37.11	87

Figure 5. EIS Nyquist diagram and Bode-phase plots for carbon steel / 1 M HCl + 2.4 g L⁻¹ of EOLD interface: (■-■-■) experimental; (—) fitted data using structural model in Figure 4.

The simulation of Nyquist and Bode plot, with the proposed model, show a great agreement with experimental data. An excellent fitting was obtained due to the accordance of the measured impedance plots and those calculated by the used equivalent circuit model.

2.6 The thermodynamic parameters of adsorption

2.6.1 Adsorption study

Adsorption isotherms are very important to understand the mechanism of corrosion inhibition reactions, so to find more about the mode and type of adsorption of EOLD on the surface, experimental results have been examined with several adsorption isotherms. Indeed, the establishment of adsorption isotherms that describe the adsorption behavior of corrosion inhibitor is important as they provide important keys about the nature of metal inhibitor interaction. In this work, the best correlation between the experimental data and the functions of the isotherms was obtained using the adsorption of Langmuir isotherm.

The relation of the Langmuir's adsorption isotherm expressed as follows: [26]

$$\frac{\theta}{1-\theta} = KC_{\text{inh}} \quad (7)$$

The rearrangement of this equation gives:

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K} + C_{\text{inh}} \quad (8)$$

With

$$K_{\text{ads}} = \frac{1}{C_{\text{H}_2\text{O}}} \cdot \exp\left(-\frac{\Delta G_{\text{ads}}^0}{RT}\right) \quad (9)$$

Where C_{inh} is the inhibitor concentration, K_{ads} is the adsorptive equilibrium constant; θ is the fraction of the surface covered calculated by potentiodynamic polarization method; and ΔG_{ads}^0 Free energy standard of adsorption. R is the universal gas constant, T the thermodynamic temperature and the concentration of water in the solution is 1000 g L^{-1} .

The experimentally plotted isotherms (points) and calculated one (lines) are depicted in Figure 6. A perfect fit is observed with the regression coefficient up to 0.99, this finding is suggesting that; the adsorption of oil on the steel surface obeys Langmuir's adsorption isotherm.

From the intercept of the straight line C_{inh}/θ vs C_{inh} , the K_{ads} value is 2.29 L g^{-1} .

The calculated ΔG_{ads}^0 value using Eq. (8) is -19.513 kJ mol/L . The negative sign of ΔG_{ads}^0 indicates that spontaneity of the adsorption process and the stability of the double layer adsorbed on the metal surface [27, 28].

Generally the values ΔG_{ads}^0 close or slightly higher than -20 kJ/mol are explained by electrostatic interaction between the adsorbed molecules and the metallic surface (physisorption). Concerning values range neighboring to -40 kJ/mol , they are due to interfacial transfer between the molecules of the inhibitor and the surface of the metal with formation of covalent band (chemisorptions) [29–33].

Figure 6. Model of the Langmuir isotherm as a function of concentration.

Within this study we have found that the calculated ΔG_{ads}^0 are very close to -20 kJ/mol indicates that the mechanism of adsorption of the tested inhibitor may be of physisorption type.

2.6.2 Determination of the activation energies

The energies of activations (E_a) for the reaction of corrosion of the carbon steel in HCl, in the absence and presence of EOLD have been calculated from the Arrhenius equation of the type:

$$w_{\text{corr}} = A \cdot e^{\left(-\frac{E_a}{RT}\right)} \quad (10)$$

This kinetic model has been used to explain the mechanism of corrosion inhibition [34].

The variation of the apparent activation energy (E_a) in the absence and presence of the inhibitor is widely discussed in the literature [35–38]. It was shown that the values of E_a have increased or decreased in the presence of an inhibitor.

An alternative formula for the Arrhenius equation allows the determination of the enthalpy and the entropy, for the intermediate complex in the transition state for the corrosion of the carbon steel in HCl, in the absence and presence of different concentrations of EOLD were obtained by applying the following equation:

$$w_{\text{corr}} = \left(\frac{RT}{Nh}\right) \exp\left(\frac{\Delta S_a}{R}\right) \exp\left(-\frac{\Delta H_a}{RT}\right) \quad (11)$$

With N : Avogadro number and h : Planck constant.

Figure 7: Right of Arrhenius calculated from speed of corrosion of the steel in: 1 M HCl and 2.4 g/L

The variation of the $\ln\left(\frac{w_{\text{corr}}}{T}\right)$ as a function of the inverse of the temperature is a right (Figure 8) with a slope equal to $\left(-\frac{\Delta H_a^0}{R}\right)$ and an intercept equal to $\left(\ln\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S_a^0}{R}\right)\right)$.

The data values of the enthalpies ΔH_a^0 and the entropies ΔS_a^0 are gathered in Table 6.

Table 6. Calculated E_a , ΔH_a^0 and ΔS_a^0 in function of the concentration.

CC (g/l)	ΔH_a^0 (kJ/mol)	ΔS_a^0 (J/mol)	E_a (kJ/mol)	$E_a - \Delta H$ (kJ/mol)
blank	34.13	-80.25	36.59	2.46
2.4	70.75	15.76	73.38	2.63

While analyzing results an increase in activation energy is pretty noticeable in presence of the inhibitor studied. This modification of value of E_a may be due to the change in the mechanism of the corrosion process in the presence of adsorbed inhibitor molecules [39, 40].

According to these result, the higher value of the activation energy in the presence of 2.4 g/l of EOLD can be explained by the molecules of our inhibitor are adsorbed on the surface by connections of electrostatic nature (physisorption) [41–43].

Figure 8. Variation of $\ln(I_{\text{corr}}/T)$ versus $1000/T$ for blank and 1 M HCl + 2.4 g/l of EOLD.

In addition the E_a and ΔH_a^* values vary in the same path with the inhibitor concentration. This result enables to verify the known thermodynamic relation between E_a and ΔH_a^* [44]:

$$E_a + \Delta H_a^* = RT \quad (12)$$

The calculated values are very close to RT which is equal to $2.52 \text{ kJ} \cdot \text{mol}^{-1}$ at 303 K.

Also the positive sign of ΔH_a^* show the endothermic nature of the corrosion process suggesting that the dissolution of carbon steel is slow in the presence of inhibitor [45–51].

Moreover the large negative value of ΔS_a for carbon steel in 1 M HCl implies that the activated complex is the rate determining step, rather than the dissociation step. In the presence of the inhibitor, the value of ΔS_a increases and it is generally interpreted as an increase in disorder given that the reactants are converted to the activated complexes [52–57].

Conclusion

All the tested methods have shown that the essential oil of *Lavandula Dentata* exhibits excellent inhibition properties for the corrosion of carbon steel in 1 M HCl at 303 K, and the inhibition efficiency, increases with increase in the inhibitor concentration.

The EIS results indicate that the double layer capacitances decrease with increasing of inhibitor concentration, this fact may be explained on the basis of adsorption of this inhibitor on the carbon steel surface.

The analysis of the essential oil of *Lavandula Dentata* shows that its composition is dominated by the camphor (50.3%). The corrosion process is inhibited by the adsorption of EOLD on the steel surface and the adsorption of the inhibitor is well fitted by Langmuir isotherm model. The calculated value of ΔG_{ads} and the energy of activation suggest that the adsorption mechanism of EOLD on carbon steel surface in 1 M HCl solution is mainly due to physisorption.

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