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# *Cystoseira Gibraltarica* Extract as an Environmentally Sustainable Corrosion Inhibitor for Carbon steel in 1.0 M HCl Medium

**Moha Afrokh,[1,2] Said Baroud,[2] Mohamed Rbaa,[3] Issam Sadki,[2] Abdelhakim Hatimi,[2] Saida Tahrouch,[2] Mohamed Tabyaoui,[1] Abdellah Guenbour,[1] Ismail Warad[4] and Abdelkader Zarrouk[1,*]**

*[1]Laboratory of Materials, Nanotechnology and Environment, Faculty of Sciences, Mohammed V University, Av. Ibn Battouta, P.O. Box. 1014 Agdal-Rabat, Morocco*

*[2]Laboratory of Plant Biotechnology, Faculty of Sciences, University of Ibn Zohr B.P. 8106, Agadir, Morocco*

*[3]Laboratory of Agro-Resources, Polymers and Process Engineering, Department of Chemistry, Faculty of Sciences, Ibn Tofail University, PO Box 133, 14000, Kenitra, Morocco*

*[4]Department of Chemistry and Earth Sciences, PO Box 2713, Qatar University, Doha, Qatar*

*Corresponding Author, Tel.: +212665201397; Fax: +212537774261
E-Mail: azarrouk@gmail.com

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**Abstract-** This study set out to examine the *cystoseira gibraltarica* extract (CG) as an environmentally sustainable corrosion inhibitor for carbon steel in 1.0 M HCl medium. The corrosion inhibiting effect has been studied by conventional methods such as gravimetric (weight loss) and electrochemical (*EIS* and *PDP*) methods. All the methods used in this study confirm that *cystoseira gibraltarica* extract has a potential inhibitory effect of carbon steel in hydrochloric acid medium. The inhibitory efficiency reached at a maximum value of 90.3% at a concentration of 0.8 g/L, this efficiency was slightly decreased at high temperature. The results obtained by the polarization show that this ecological inhibitor acts as a mixed inhibitor. The parameters thermodynamics show that the studied inhibitor is adsorbed on the metal surface following Langmuir isotherm.

**Keywords-** Cystoseira gibraltarica extract, Carbon steel corrosion, Weight loss, Electrochemical methods, Langmuir isotherm

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## 1. INTRODUCTION

Studies on the corrosion of steel represent a growing field, because steel is very useful in several living areas (in the field of construction, in parts of houses, means of transport ...) [1]. But the fact that factories and users of metal-based products find the corrosion problem of these products, especially in aggressive environments such as acidic environments [2]. In view of this undesirable phenomenon of corrosion of the metal parts, it is obligatory to present the appropriate solutions to this phenomenon. Today, the researchers are presenting solutions against metal corrosion such as: coating protection, oxidation protection and inhibition protection. If we consider the economic side, the protection by the chemical inhibitors remains the best method [3,4].

The chemical inhibitors used for the corrosion of steels in aggressive media are either organic or inorganic compounds. According to the literature, organic and inorganic chemical inhibitors are very profitable against the corrosion of steels in aggressive media (HCl, $H_2SO_4$, $H_3PO_4$ ...) [5-11]. But always there is the problem of the toxicity of these products [12]. Today, the tendency towards the protection of metals by ecological inhibitors is sustainable, like the essential and vegetable oils of medicinal plants, also the extracts of medicinal plants [13,14].

Our solution and proposal in this study is the presentation of *Cystoseira gibraltarica* extract as an ecological inhibitor is durable against the corrosion of carbon steel in molar hydrochloric acid medium using gravimetric and electrochemical investigations.

## 2. MATERIALS AND METHODS

### 2.1. Plant material

*Cystoseira gibraltarica* (CG), a brown algae, was collected at low tide in the coastal area of Cape Ghir (30 ° 38'37 "N, 09 ° 53'20" W), about 43 km northwest of Agadir, Morocco. This seaweed is carefully washed to remove all traces of salt and dried. After that, it is reduced to a fine powder using an electric grinder.

### 2.2. Extraction

20 g of this CG algae powder was extracted with methanol using Soxhlet for 3 h. The extract obtained was concentrated by rotary evaporation. The extraction yield is 15.50%.

### 2.3. Corrosion test

The chemicals compositions of the used carbon steel (in wt %) is 0.370 of C, 0.230 of Si, 0.680 of Mn, 0.016 of S, 0.077 of Cr, 0.011 of Ti, 0.059 of Ni, 0.009 of Co, 0.160 of Cu and the remainder iron (Fe). The aggressive solution of 1.0 M HCl was prepared by dilution of analytical grade, 37% HCl with distilled water.

Concerning the methods for the realization of the corrosion tests, we used electrochemical methods (*PDP* and *EIS*) and gravimetric methods (weight loss).

The inhibition efficiencies were calculated using following equations (1-3): respectively, for the gravimetric, polarization and impedance methods.

$$\eta_{\omega}\left(\%\right)=\frac{\omega^{0}_{corr}-\omega_{corr}}{\omega^{0}_{corr}}\times \tag{1}$$

$\omega^{0}_{corr}$ and $\omega_{corr}$ are the corrosion rates with and without the addition of *Cystoseira gibraltarica* extract.

$$\eta_{\mathrm{PP}}=\frac{i^{0}_{\mathrm{corr}}-i_{\mathrm{corr}}}{i^{0}_{\mathrm{corr}}}\times 100 \tag{2}$$

$i_{\mathrm{corr}}$ and $i^{0}_{\mathrm{corr}}$ are the corrosion current density with and without the addition of *Cystoseira gibraltarica* extract.

$$\eta_{\mathrm{EIS}}=\frac{R_{\mathrm{p}}-R^{0}_{\mathrm{p}}}{R_{\mathrm{p}}}\times 100 \tag{3}$$

$R_p$ and $R^{0}_{\mathrm{p}}$ are the polarization resistance with and without the addition of *Cystoseira gibraltarica* extract. The details for the gravimetric and electrochemical methods were published in another paper published by our Laboratory [15].

## 3. RESULTANTS AND DISCUSSION

### 3.1. Gravimetric measurements

The gravimetry we give us preliminary information on the inhibitory effect of a product tested. However, it is a simple method and does not require very complicated equipment [16].

**Table 1.** Gravimetric data at 303 K in 1.0 M HCl

| C (g/L) | $\omega_{corr}$ (mg cm$^{-2}$ h$^{-1}$) | $\eta_{\omega}$ (%) | θ |
|---|---|---|---|
| 0 | 2.40±0.09 | — | — |
| 0.05 | 0.69±0.07 | 71.2 | 0.712 |
| 0.10 | 0.66±0.08 | 72.5 | 0.725 |
| 0.50 | 0.45±0.03 | 81.2 | 0.812 |
| 0.80 | 0.32±0.02 | 86.7 | 0.867 |

The results obtained by the gravimetric investigation are given in Table 1 and Figure 1. If we see the results of Table 1, we observe that the ecological inhibitor has a corrosion inhibiting effect of carbon steel in 1.0 M. HCl. Increasingly, the inhibitory efficacy increases with the content of ecological inhibitor up to 86.7% at 0.80 g/L. The Figure 1 shows that addition of ecological inhibitor causes a decrease in corrosion rate values.

The good corrosion inhibiting potency of *Cystoseira gibraltarica* extract comes from multiple chemical compositions of this extract [16].

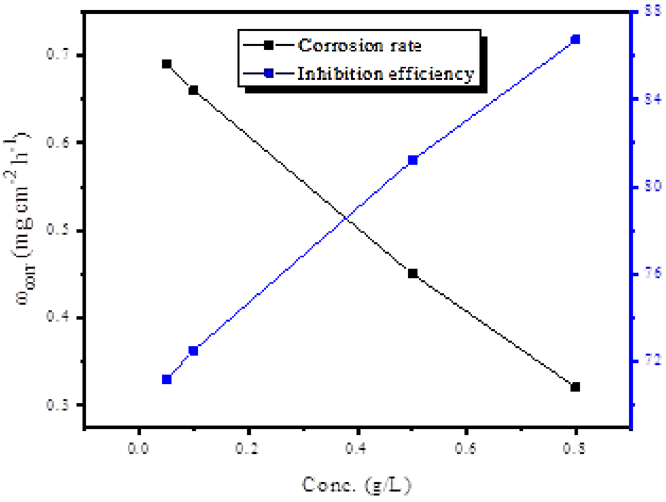

**Fig. 1.** Variation of the rate of corrosion the inhibition efficiency as a function of the concentration of the extract CG in 1.0 M HCl at 303 K and for an immersion time of 6 h

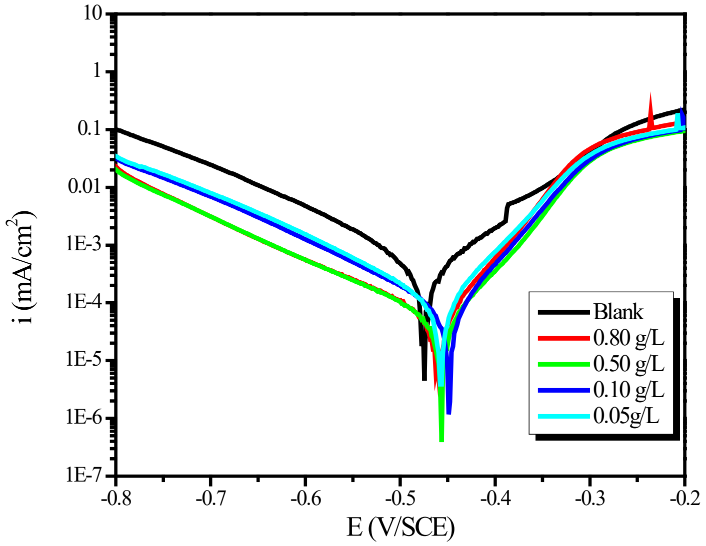

**Fig. 2.** Tafel Curves of the C-steel in 1.0 M HCl in the absence and the presence of different concentrations of CG at 303 K

### 3.2. Potentiodynamic polarization measurements

The Potentiodynamic polarization allows us to obtain information on the parameters of the corrosion process [17]. The results obtained by the gravimetric investigation are given in Table 2 and Figure 2.

**Table 2.** Electrochemical data of C-steel in 1.0 M HCl without and with additions of CG extract at 303 K

| C (g/L) | $-E_{corr}$ (mV/SCE) | $i_{corr}$ ($\mu A/cm^2$) | $\beta_a$ (mV/dec) | $-\beta_c$ (mV/dec) | $\eta_{PP}$ (%) |
|---|---|---|---|---|---|
| Blank | 477.2±1.1 | 430.9±1.3 | 111 | 118.6 | — |
| 0.80 | 462.9±1.0 | 44.9±0.9 | 53.6 | 106.5 | 89.6 |
| 0.50 | 459.5±0.9 | 56.2±0.8 | 66.1 | 144.7 | 86.9 |
| 0.10 | 451.2±0.9 | 74.6±1.1 | 59.1 | 120.6 | 82.7 |
| 0.05 | 459.8±0.8 | 99.9±0.7 | 62.4 | 118.6 | 76.8 |

In Table 2 there is a clear trend of decreasing in the values of current density ($i_{corr}$) with increasing concentrations of ecological inhibitor (CG), which causes an increase in inhibitory efficiency values up to an optimum value of 89.57% at 0.80 g/L. Another observation, the addition of the ecological inhibitor (CG) causes variations in the values of the anodic and cathodic slopes ($\beta_a$ and $\beta_c$) and also in the values of the corrosion potential ($E_{corr}$). This result shows that our inhibitor acts on the surface of steel as a mixed inhibitor [18]. The potentiodynamic polarization (*PDP*) investigation results are in good agreement with the gravimetric investigation results.

### 3.3. Impedance spectroscopy measurement

To the extract the global parameters of corrosion process, we carried out a study by the impedance spectroscopy. EIS test was performed to evaluate the effect of the *cystoseira gibraltarica* extract (CG) on the electrode surface. Figure 3 demonstrate the Nyquist plots of C-steel in 1.0 M HCl solution without (inset figure) and with *cystoseira gibraltarica* extract (CG). The Nyquist spectra for all plots, demonstration one single depressed semicircle that their center is under the real axis. The diameter of semicircle increases by increasing concentration of *cystoseira gibraltarica* extract (CG), indicating strengthening of protective film. Depression is feature of solid electrodes that attributed to inhomogeneity and roughness of the surface throughout corrosion [19].

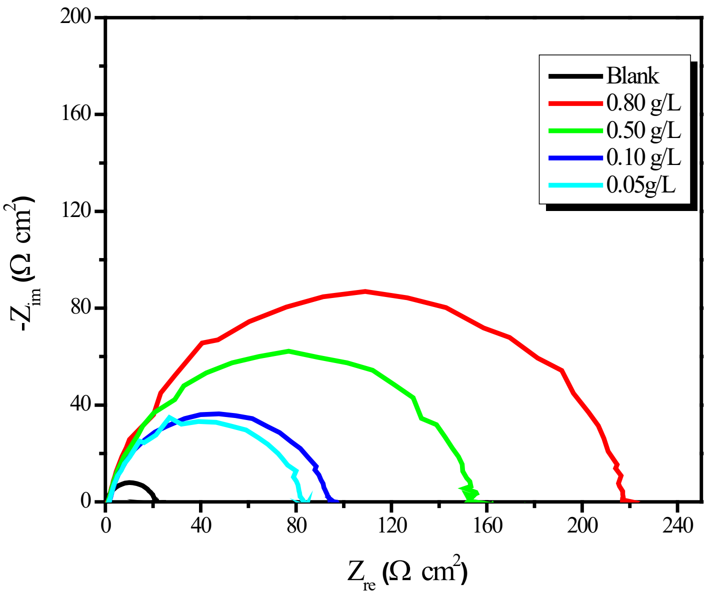

**Fig. 3.** Nyquist diagrams for the CS electrode in 1.0 M HCl without and with CG extract at 303 K

All impedance data are simulated (representative example Figure 5) using the equivalent circuit presented in Fig. 4. The results of EIS are fitted by using the electrical equivalent circuit in Fig. 4 that consists of $R_s$ and $R_p$ and CPE, represent the solution resistance, polarization resistance and constant phase element (instead of the double layer ($C_{dl}$)), respectively. The impedance ($Z$) of CPE is expressed by using the following statement [20]:

$$Z_{CPE} = Q^{-1}(j\omega)^{-n} \tag{4}$$

In equation 4, $Q$ is the CPE constant, $\omega$ is the angular frequency, $j^2 = -1$ is the imaginary number and $n$ representation the degree of surface inhomogeneity subsequent surface roughness, inhibitor adsorption, porous layer formation, etc. The results are listed in Table 3.

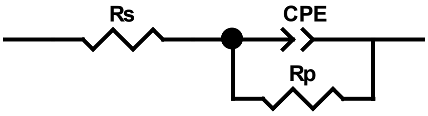

**Fig. 4.** Equivalent circuit for the C-steel /electrolyte system

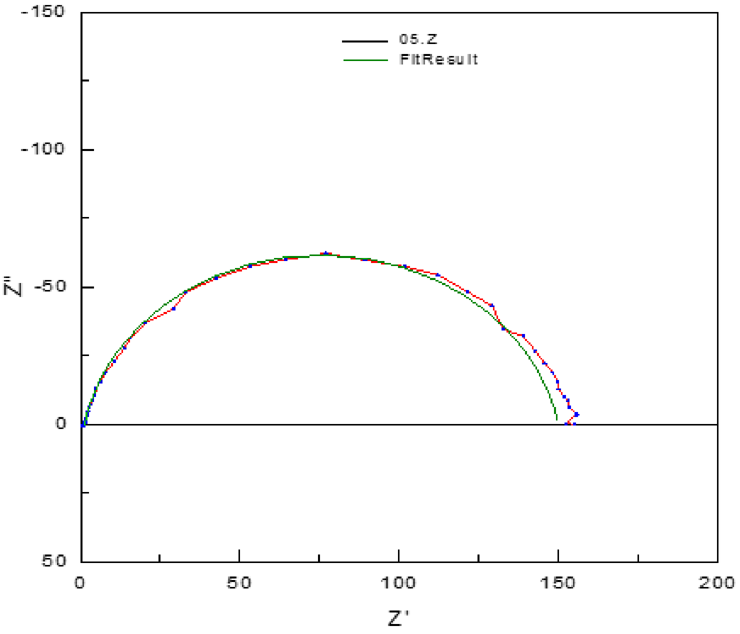

**Fig. 5.** Representative example of EIS Nyquist diagrams simulated in 1.0 M HCl with 0.5 g/L of CG extract at 303 K

**Table 3.** EIS data for C-steel in a molar solution of hydrochloric acid with and without CG

| C (g/L) | $R_s$ (Ω $cm^2$ | $R_p$ (Ω $cm^2$) | $f_{max}$ (Hz) | n | $C_{dl}$ (µF/$cm^2$) | $\chi^2$ | η (%) |
|---|---|---|---|---|---|---|---|
| Blank | 0.560±0.007 | 021.0±0.2 | 40.00 | 0.865±0.004 | 173.0 | 0.004 | — |
| 0.80 | 0.430±0.006 | 217.7±1.5 | 15.83 | 0.907±0.002 | 46.2 | 0.002 | 90.3 |
| 0.50 | 0.520±0.008 | 152.8±1.1 | 15.84 | 0.896±0.003 | 65.8 | 0.005 | 86.2 |
| 0.10 | 0.506±0.005 | 092.2±0.9 | 25.01 | 0.888±0.004 | 69.1 | 0.005 | 77.2 |
| 0.05 | 0.487±0.007 | 077.8±0.5 | 25.13 | 0.881±0.001 | 81.4 | 0.002 | 73.0 |

According to the data in Table 3, we observed that the addition of the ecological inhibitor causes decrease in the values of the polarization resistance ($R_p$) and an increase in the values of the double layer capacitance ($C_{dl}$). This can be explained by the strong adsorption of the inhibitor (CG) on the surface of carbon steel (*CS*) [21]. However, the exponent ***n*** of *CPE* increases with concentration, which can be attributed to a some decrease the degree of inhomogeneity of the electrode surface resulting from the molecules adsorption on the active centers. The inhibitory efficiency increases with increasing concentration of ecological inhibitor (90.3% for 0.80 g/L of CG). Figure 3 shows that the impedance diagrams obtained are half-circles. This shows that there is a mechanism of charge transfer between the systems (inhibitor/solution/metal) [22].

The impedance spectroscopy (*EIS*) results are in good agreement with the gravimetric and the potentiodynamic polarization (*PDP*) investigation results.

### 3.4. Effect of temperature and activation parameters

The temperature effect is an instrumental in changing our understanding and approach to the management of the corrosion process of steel in acidic media [23]. The results obtained from the effect of temperature are given in Table 4 and Figure 6 and 7.

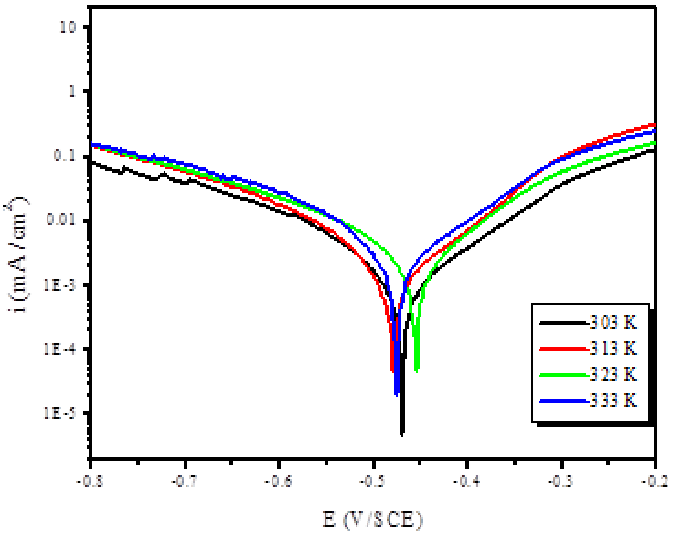

**Fig. 6.** Tafel curves of C-steel in uninhibited electrolyte at diverse temperatures

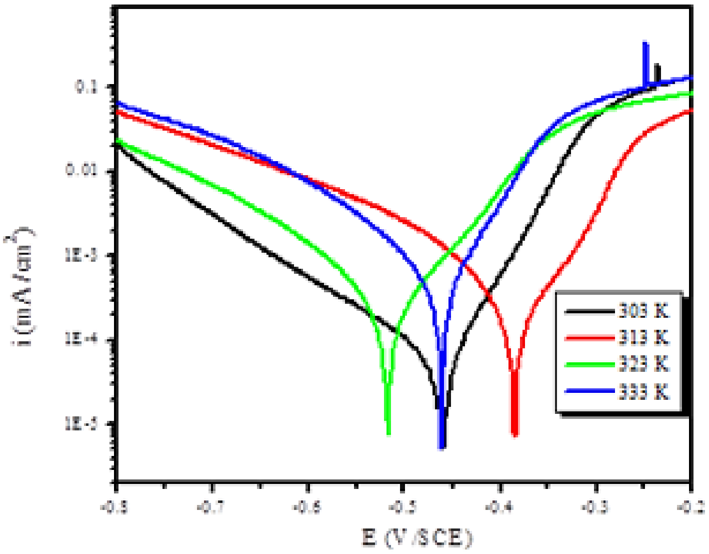

**Fig. 7.** Tafel curves of C-steel in 1.0 M HCl + 0.80 g/L of CG at diverse temperatures

**Table 4.** Tafel parameters of C-steel in 1.0 M HCl in the absence and the presence of 0.80 g/L of CG at diverse temperatures

| Medium | T (K) | $-E_{corr}$ (mV/SCE) | $i_{corr}$ ($\mu$A/cm$^2$) | $\beta_a$ (mv/dec) | $-\beta_c$ (mv/dec) | $\eta_{PP}$ (%) |
|---|---|---|---|---|---|---|
| 1.0 M HCl | 303 | 477.2±1.1 | 0430.9±1.3 | 111 | 118.6 | — |
| | 313 | 481.8±1.6 | 0966.7±1.8 | 91.0 | 73.8 | — |
| | 323 | 457.7±1.0 | 1506.9±1.1 | 87.2 | 87.6 | — |
| | 333 | 479.6±0.7 | 1966.4±1.7 | 110.9 | 94.1 | — |
| CG | 303 | 462.9±1.0 | 044.9±0.9 | 53.6 | 106.5 | 89.6 |
| | 313 | 387.7±1.2 | 132.2±0.8 | 66.0 | 65.5 | 86.3 |
| | 323 | 519.6±0.8 | 302.9±0.6 | 97.8 | 123.1 | 79.9 |
| | 333 | 463.8±1.5 | 515.2±1.4 | 61.2 | 111.3 | 73.8 |

From Table 4, it is observed that the increase in temperature causes an increase in the values of the current density (*$i_{corr}$*). Therefore the inhibitory efficiency values decrease until 73.8% at 333 K, then this ecological inhibitor is profitable at high temperature.

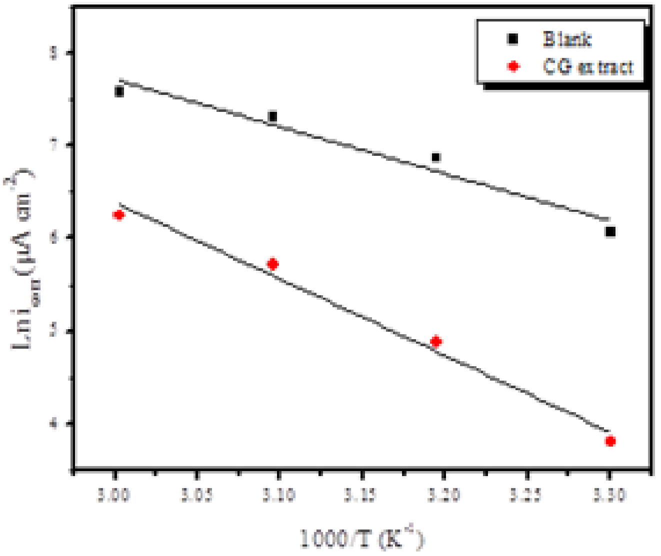

**Fig. 8.** Arrhenius plots for C-steel obtained in 1.0 M HCl only and with 0.80 g/L of CG extract

From the temperature effect, we have calculated several thermodynamic parameters of activations such as:

The activation energy (*$E_a$*) was calculated by the Arrhenius' equation (5) (Figure 8):

$$i_{corr} = A\exp\left(\frac{-E_a}{RT}\right) \quad (5)$$

where $A$ is a pre-exponential factor and $E_a$ is the activation energy. Plots of Ln ($i_{corr}$) versus 1000/T in both uninhibited and inhibited electrolytes are depicted in Figure 8. The linear regression of these latter results in the value of activation energy.

The apparent activation energy ($E_a$) of the corrosion reaction in corrosion media can be calculated by plotting Ln $i_{corr}$ with 1/T. From this curve, the straight line is obtained which can be calculated from its slope.

The activation enthalpy ($\Delta H_a$) and the activation entropy ($\Delta S_a$) were calculated by the investigation of the Arrhenius' equation (6) (Figure 9)

$$i_{corr} = \frac{RT}{Nh}\exp\left(\frac{\Delta S_a}{R}\right)\exp\left(\frac{\Delta H_a}{RT}\right) \tag{6}$$

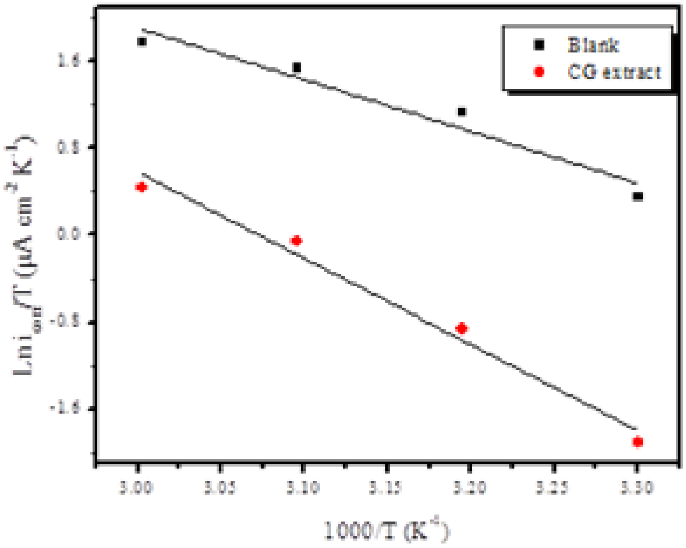

**Fig. 9**. Transition state plot for C-steel obtained in 1.0 M HCl only and with 0.80 g/L of CG extract

**Table 5.** Activation parameters for the carbon steel corrosion in 1.0 M HCl in the absence and the presence of of CG extract

| Medium | $R^2$ | $E_a$ (kJ mole$^{-1}$) | $\Delta H_a$ (kJ mole$^{-1}$) | $\Delta S_a$ (J mole$^{-1}$ K$^{-1}$) |
|---|---|---|---|---|
| Blank | 0.98 | 33.69 | 31.01 | -89.13 |
| CG extract | 0.99 | 65.57 | 64.74 | -121.08 |

Light of Table 5, we note that the value of the activation energy ($E_a$) of ecological inhibitor is greater than the blank solution. This result can be explained by the formation of the physical and chemical interactions between the molecules of ecological inhibitor and the metallic surface [24]. The positive value of activation enthalpy ($\Delta H_a$) according to the literature showed that the corrosion process is endothermic [24]. The negative values of $\Delta S_a$ indicate that the activated complex tends to association more than dissociation, in the rate determining step. Therefore, when going from reactants to the activated complex, decrease in disordering occurred [25].

### 3.5. Adsorption isotherm

The adsorption isotherm describes the adsorption behavior of organic compounds for better understand the adsorption mechanism. Different adsorption isotherms such as Langmuir, Temkin, Freundlich, Bockris–Swinkels and Flory–Huggins isotherms can be used to distinguish the adsorption action of the inhibitors. Langmuir adsorption isotherm, which defined by equation (7) was established to be the best fit from all of the examined isotherms. The $R^2$ (correlation coefficient) value was utilized as a criterion to conclude the best fit adsorption isotherm. Perfectly linear plot was obtained with a correlation coefficient of 0.99995 and slope of 0.1817. Langmuir isotherm is given as:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \tag{7}$$

where ($C_{inh}$) is the extract concentration, $\theta$ is the degree of coverage of surface and $K_{ads}$ is the equilibrium constant of adsorption–desorption procedure. The inhibition efficiency ($\eta_\omega$) as $\theta=\eta_\omega/100$ from weight loss data was used for estimate of the θ for different concentrations at 303 K. The relationship between $C_{inh}/\theta$ and $C_{inh}$ at 303 K is shown in Fig. 10.

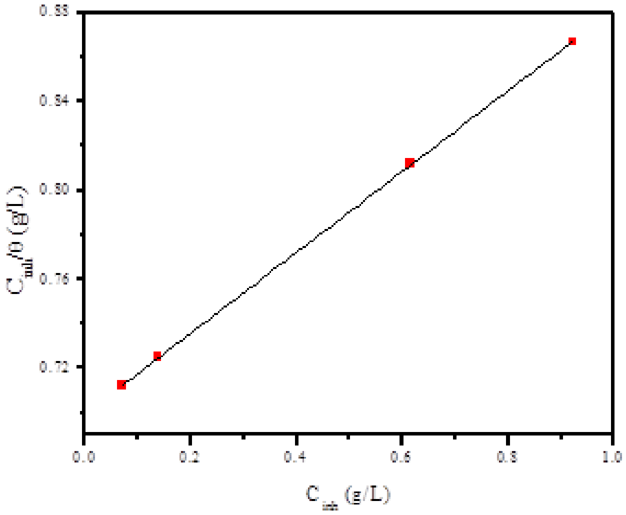

**Fig. 10.** Isotherm of Langmuir adsorption of CG extract on the surface of steel at 303 K

Though the linearity of the Langmuir plot may be taken to suggest that the adsorption of *Cystoseira gibraltarica* extract follows the Langmuir isotherm, the considerable deviation of the slope from unity indicates that the isotherm cannot be strictly applied. Langmuir isotherm postulates monolayer adsorption hence no interaction between the adsorbate species on the C-steel surface and in such circumstances, the slope should be unity. The deviation of the slope from unity as observed in this study could be interpreted to mean that there are interactions between adsorbate species on the C-steel surface as well as changes in adsorption heat with increasing surface coverage [26,27], factors that were ignored in the derivation of Langmuir isotherm.

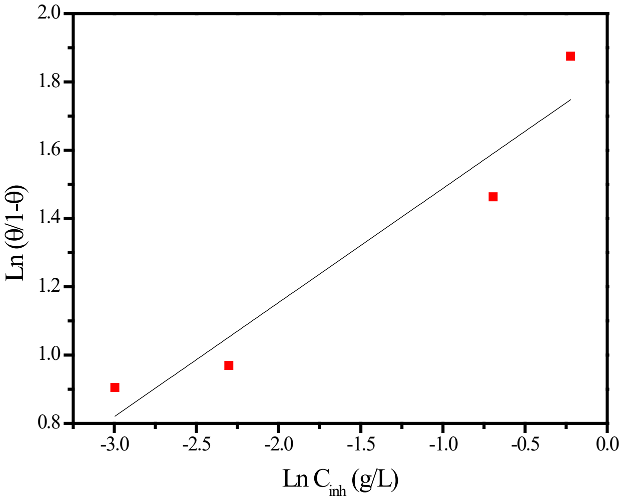

**Fig. 11.** El-Awady's isotherm adsorption model of Cystoseira gibraltarica extract on the C-steel surface in 1.0 M HCl at 303 K

The experimental data were then fitted into the modified form of the Langmuir isotherm known as El-Awady kinetic–thermodynamic adsorption isotherm model which can appropriately represent the adsorption behavior of *Cystoseira gibraltarica* extract onto C-steel surface. The El-Awady isotherm is given as [28]:

$$Ln\frac{\theta}{1-\theta} = LnK' + yLn\,C_{inh} \tag{8}$$

where $C_{\mathrm{inh}}$ is the molar concentration of inhibitor, θ is the degree of surface coverage, $K'$ is a constant related to the equilibrium constant of the adsorption process, $K_{ads}$, by the following relationship:

$$K_{ads} = K'^{1/y} \tag{9}$$

where 1/y represents the number of active sites of the C-steel surface occupied by one molecule of inhibitor. $K_{ads}$ is related to the free energy of adsorption $\Delta G_{\mathrm{ads}}$ by the equation:

$$K_{ads} = \frac{1}{C_{eau}} \exp\left(\frac{-\Delta G^{\circ}_{ads}}{RT}\right) \qquad (10)$$

where R=8.314 J $mol^{-1}$ $K^{-1}$ is the universal gas constant, T the thermodynamic temperature and the concentration of $H_2O$ in the solution is 1000 g/L.

Curve fitting the experimental data for the *Cystoseira gibraltarica* extract to the thermodynamic–kinetic adsorption isotherm model is shown in Fig. 11.

Linear plot was obtained with a correlation coefficient of 0.8881. The computed values of adsorption parameters cand 1/y obtained were 6.1867 L/g and 2.9914 and, respectively. The value of 1/y is greater than one, suggesting that a given inhibitor molecule occupies more than one active site [29]. The value of $K_{ads}$ is large and indicates strong adsorption of the *Cystoseira gibraltarica* extract onto the C-steel surface.

Generally, for values of the standard Gibbs free energy of adsorption around -20 $kJmol^{-1}$ or less negative, the type of adsorption is regarded as physisorption; those around -40 $kJmol^{-1}$ or more negative are associated with chemisorption [30-32]. However, it is difficult to distinguish between chemisorption and physisorption only based on these criteria, especially when charged species are adsorbed. The possibility of Coulomb interactions between adsorbed cations and specifically adsorbed anions can increase the $\Delta G_{ads}$ energy even if no chemical bond appears [33]. However, the calculation of the standard Gibbs free energy of adsorption value of *Cystoseira gibraltarica* extract is not possible because the molecular mass of the extract components is not known. This limitation is noted by some authors in the case of the plant extracts used as corrosion inhibitors for steel in acidic media [34,35].

## 4. CONCLUSION

This study highlight the importance of utilizing *Cystoseira gibraltarica* extract as an environmentally sustainable corrosion inhibitor for carbon steel in 1.0 M HCl by the gravimetric and electrochemical studies. The data obtained by all methods show that the extract has a potential corrosion inhibition effect (90.3% at 0.8 g/L). Polarization studies show that the extract acts as a mixed inhibitor. However, this inhibitor remains effective at high temperature.

The thermodynamic adsorption and activation parameters show that this inhibitor adsorbs to the metal surface following the Langmuir isotherm.

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