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# Inhibition of Carbon Steel corrosion in Acid mediumin by *Eruca sativa* Extract

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## Abstract

Corrosion inhibition of *Eruca sativa* leaves extract on C-steel in 0.1 M $H_2SO_4$ was examined by weight loss and electrochemical techniques. It was found that the extract act as a good corrosion inhibitor for the tested system. The inhibition efficiency increases with increasing extract concentration and decreases with increasing temperature. The adsorption of inhibitor on the C-steel surface obeyed Langmuir adsorption isotherm. The calculated adsorption thermodynamic parameters indicated that the adsorption was spontaneous and exothermic process accompanied by an increase in entropy.

## 1. Introduction

Carbon steel, the most widely used engineering material in many applications. Corrosion problems arise as a result of interaction of the aqueous solutions with C-steel, especially during the pickling process in which the alloy is brought in contact with high concentrated acids. Use of inhibitors is one of the most practical methods for protection against corrosion especially in acid solutions to prevent unexpected metal dissolution and acid consumption [1]. Most of the well-known acid inhibitors are organic compounds especially those with N, O and S showed an effective corrosion inhibition [2,3], Via adsorption of the molecules on the metal surface, creating a barrier to corroding attack.

However, majority of these organic compounds are toxic for human being and environment as well. The new generation of environmental regulation requires the replacement of toxic inhibitors with non-toxic inhibitors. In view of this, many alternative eco-friendly corrosion inhibitors have now been developed. Ginger [4], henna [5], thym [6], jojoba oil [7], Chamomile (Chamaemelum mixtum L.), Halfabar (Cymbopogon proximus), Black cumin (Nigella sativa L.), and Kidney bean (Phaseolus vulgaris L.) [8,] Acacia Trees [9], Fenugreek, Xylopia Ferruginea [10,11], Phyllanthus Amarus [12] Bauhinia purpurea leaves [13], Hibiscus cannabinus [14] Black Pepper[15] Ficus glumosa gum[16] El-Etre et al. studied the corrosion inhibition of various metals like Cu, Al, Zn, steel using natural honey, opuntia, lawsonia Khillah extract[17–20]. Oguzie [21] studied the corrosion inhibition of leaf extracts of Occimum viridis, Telferia occidentalis, Azadirachta indicaand Hibiscus sabdariffaas well as extracts fromthe seeds of Garcinia kolaon mild steel corrosion in acidic solutions. Gunasekaran et al. [22, 23] studied the corrosion inhibition of steel by eco-friendly Zenth-oxylum alatumplant extract in HCl as well as in phosphoric acid medium. Justicia gendarussa[24], Punica granatum [25] garlic peel [26] Artemisia pallens[27] Osmanthus fragran[28] peel Fruit peel [29] coffee ground extracts[30]. Eruca sativa is an important member of the Brassicaceae family. Flavonoids are present in high concentrations in the epidermis of leaves and fruits of Eruca sativa. Quercetin, kaempferol and isorhamnetin, the main flavonols in Brassica crops, are most commonly found as O-glycosides. Kaempferol di-O-glycoside, Isorhamnetin mono-O-,di-O-, and tri-O-glycosides ,Quercetin monosinapoyl tri-O-glycoside.The main compound is Quercetin monosinapoyl tri-O-glycoside[31].

The present work devotes to investigate the effect of Eruca sativa extracts as a corrosion inhibitor for carbon steel in 0.1 M $H_2SO_4$ by weight loss in different temperature, potentiodynamic polarisation measurements. The thermodynamic parameters were also calculated and discussed.

## 2. Experimental

### *2.1. Medium.*

The aggressive solution of 0.1M $H_2SO_4$ was prepared by dilution of analytical grade $H_2SO_4$ (98%) with double distilled water and all experiment were carried out in unstirred solutions and at room temperature of 25±1°C

### *2.1.1. Extract preparation.*

Fresh leave of Eruca sativa were extensively washed under running tape water for removal of dust particles and epiphytic hosts normally found on the surface, followed by washing with sterilized distilled water. They were further air-dried on filter paper at room temperature and then powdered with the help of sterilized pestle and mortar. Dry powder was further extracted by using aqueous solvent.

Air- dried powder of the respective plant part was mixed well in 100 ml sterilized distilled water and kept at room temperature for 24h on an orbital shaker with 150 rpm. The solution was further filtered using muslin cloth; the filtrate was centrifuged at 5000 rpm for 15 min. The supernatant thus obtained was filtered through Whitman's filter no.1, and then the filtrate was evaporated until decreases. The solid residue was collected and used in preparation of stock solution from which the desired concentration were prepared by dilution. The extract main component has the formulas shown in scheme 1.

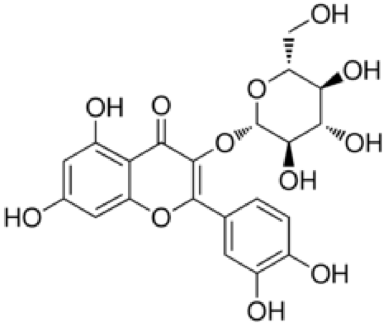

Fig 1: Quercetin 3-β-D-glucoside

## 2.2 Weight loss measurements.

Steel (L52), with 2.0x 2.0 x 0.05 cm sizes for each, were used for weight loss measurements. Weight loss experiment was carried out as described elsewhere [32]. The corrosion rate (CR) and the percentage protection efficiency IE (%) were calculated according to the following equations:

$$CR = \frac{\Delta m}{St},$$

$$IE(\%) = \frac{CR_{corr} - CR_{corr(inh)}}{CR_{corr}} \times 100 \ , \qquad (1)$$

Where $\Delta m$ (mg) is the mass loss, S ($dm^2$) is the area, $t$ (h) is the immersion period, and $CR_{corr}$ and $CR_{corr(inh)}$ are the corrosion rates of steel in absence and presence of the inhibitors, respectively.

The values of surface coverage for different concentrations at different temperatures were calculated from weight loss measurements and represented in Table1.

$$\theta = IE/100 \qquad (2)$$

### 2.3. Potentiodynamic polarization.

Potentiodynamic measurements were carried out using three-compartment glass cell and PS remote potentiostat and PS6 software for calculation of electrochemical parameters. Platinum electrode was used as a counter electrode (separated from the cell solution by a sintered glass frit) and saturated calomel electrode SCE (inside a Luggin probe) as a reference electrode.

A cylindrical rod embedded in araldite with an exposed surface area of 0.5 $cm^2$ was used. The electrode surface was polished with different grades of emery paper, degreased with acetone, and rinsed with distilled water.

The inhibition efficiency IE (%) was calculated from polarization measurements according to the relation given below:

$$IE(\%) = \frac{I_{corr} - I_{corr(inh)}}{I_{corr}} \times 100, \qquad (3)$$

Where $I_{corr}$ and $I_{corr(inh)}$ are current densities in free and inhibited acid, respectively. They are determined by extrapolation of the anodic and cathodic Tafel lines.

## 3. Result and Discussion

### *3.1. Weight loss Measurements.*

The effect of addition of the extract compound on the weight loss of steel in 0.1M $H_2SO_4$ as a corrosive medium was studied. The values of the corrosion rate (CR) and inhibition efficiency IE (%) obtained from weight loss at different concentration of extract at the temperature range of 25-60$^o$C are presented in Table 1. It has been found that the extract inhibits the corrosion of steel in sulphric acid solution at all concentration range (100-500ppm). The data given in Table1 revel that the corrosion rate decreases and the Inhibition efficiency of steel increases with the increasing of extract concentration up to 95% at 25$^o$C. The observed inhibition action of the *Eruca sativa* extract could be attributed to the adsorption of its components on C-steel surface.

**Table 1:** Corrosion parameters for steel corrosion in an aqueous solution of 0.1M$H_2SO_4$ in absence and presence of different concentration of inhibitor from weight loss measurements.

| Conc. (ppm) | Temperature | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 25$^0$C | | | 40$^0$C | | | 50$^0$C | | | 60$^0$C | | |
| | CR mg.$cm^{-2}$.$h^{-1}$ | IE % | θ | CR mg.$cm^{-2}$.$h^{-1}$ | IE % | θ | CR mg.$cm^{-2}$.$h^{-1}$ | IE % | θ | CR mg.$cm^{-2}$.$h^{-1}$ | IE % | θ |
| 0.0 | $1.04x10^{-4}$ | - | - | $1.38x10^{-4}$ | - | - | $1.8x10^{-4}$ | - | - | $2.77x10^{-4}$ | - | - |
| 50 | 3.47*10-5 | 66 | 0.66 | 7.63*10-5 | 45 | 0.45 | 1.1*10-4 | 36 | 0.36 | 2.10*10-4 | 26 | 0.26 |
| 100 | $1.90x10^{-5}$ | 82 | 0.82 | 5.2x10-5 | 62 | 0.62 | $6.7x10^{-5}$ | 57 | 0.57 | $1.47x10^{-4}$ | 48 | 0.48 |
| 200 | $8.00x10^{-6}$ | 92 | 0.92 | $2.1x10^{-5}$ | 84 | 0.84 | $3.05x10^{-5}$ | 82 | 0.82 | $9.02x10^{-5}$ | 67 | 0.67 |
| 300 | $6.00x10^{-6}$ | 93 | 0.93 | $1.25x10^{-5}$ | 89 | 0.89 | $1.6x10^{-5}$ | 88 | 0.88 | $6.2x10^{-5}$ | 75 | 0.75 |
| 400 | $4.00x10^{-6}$ | 94 | 0.94 | $9.7x10^{-6}$ | 93 | 0.93 | $1.5x10^{-5}$ | 90 | 0.90 | $4.0x10^{-5}$ | 78 | 0.78 |
| 500 | $3.81x10^{-6}$ | 95 | 0.95 | $6.9x10^{-6}$ | 93 | 0.93 | $1.2x10^{-5}$ | 91 | 0.91 | $3.00x10^{-5}$ | 80 | 0.80 |

Further inspection of Table (1) reveals that, as the temperature is increased the corrosion rate increases and the inhibition efficiency decreases. This result suggests a possible desorption of some of the adsorbed inhibitors from the metal surface at higher temperatures. Such behavior shows that the additives were physically adsorbed on the metal surface [33-34]. The relationship between the inhibition efficiency and concentration is represented in Fig1 shows that as the concentration increases, the inhibition efficiency increases up to 300ppm. This result suggests that at the concentration range of (100-300 ppm), the whole surface is nearly covered by the adsorbed molecules. Thus further increase in the concentration has a lesser effect on the corrosion inhibition process.

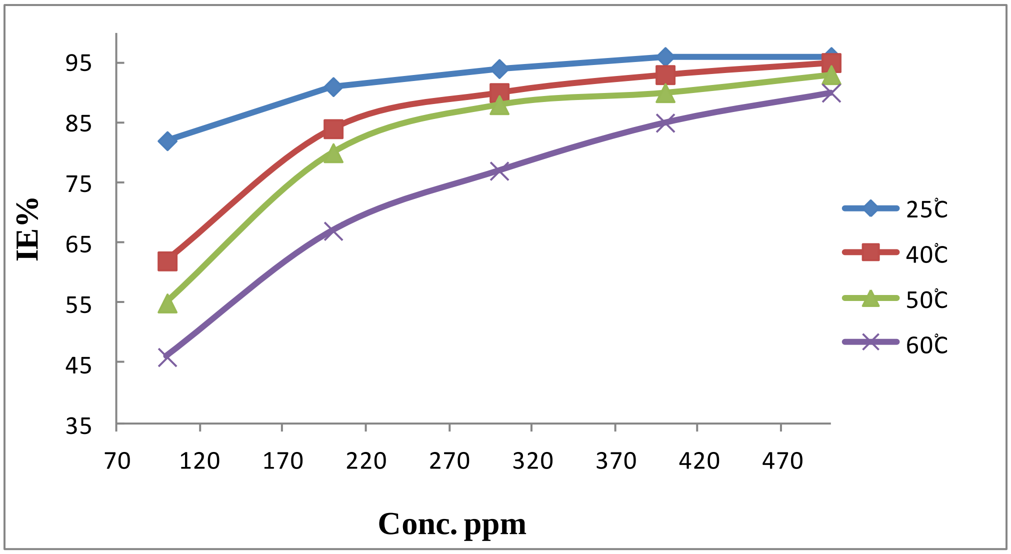

Fig1: Inhibition efficiency of inhibitor at different concentration, 100ppm, 200ppm, 300ppm, 400ppm, 500ppm.

### *3.2. Adsorption isotherm*

Basic information about the interaction between the inhibitor molecules and carbon steel surface can be provided by the adsorption isotherm. In the range of studied temperature, the best correlation between the extract concentration and surface coverage was obtained using Langmuir adsorption isotherm that given by [35]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (4)$$

Where $K_{ads}$ is the adsorption equilibrium constant of the inhibitor adsorption process and $C$ is the inhibitor concentration.

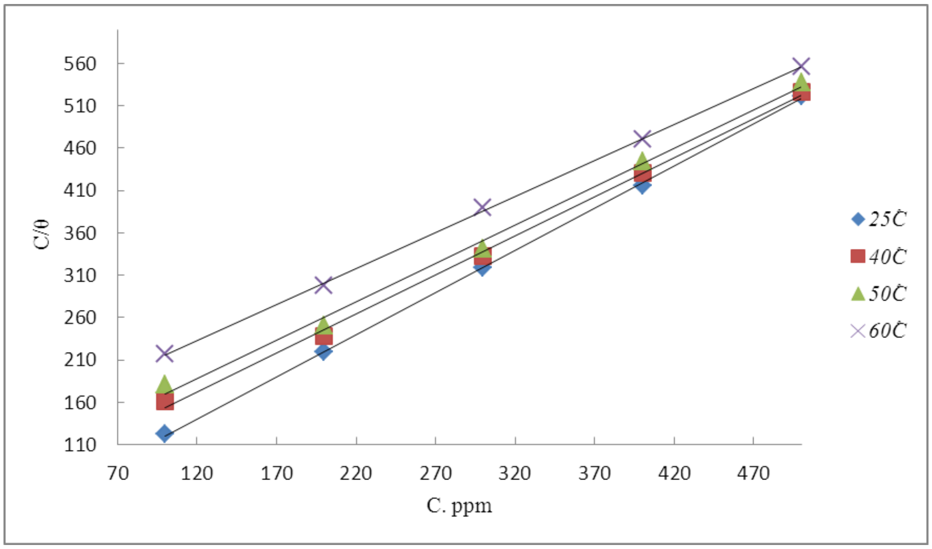

Fig2: Langmuir's adsorption isotherm plot for the adsorption of inhibitor in 0.1 M $H_2SO_4$ at different temperature.

The linear regressions between $C/\theta$ and $C$ for each temperature over concentration range (100 – 500 ppm) are shown in Fig 2 and the adsorption parameters are listed in Table 2. The results show very high correlation coefficients and the slopes values are close to one. These finding confirm that the adsorption of the inhibitor molecules in 0.1M $H_2SO_4$ follows Langmuir adsorption isotherm.

The values of $K_{ads}$ were calculated from the intercepts of the straight lines on the $C/\theta$ – axis. The $K_{ads}$ was related to the standard free energy of adsorption, $\Delta G^o_{ads}$ according to the following equation [36]:

$$K_{ads} = \frac{1}{55.5}\ \exp\left(\frac{-\Delta G^0_{ads}}{RT}\right) \tag{5}$$

The value of 55.5 is the molar concentration of water in mol $dm^{-3}$. The obtained value of $\Delta G^o_{ads}$ is listed in Table (2).

Table 2: Thermodynamic parameters for the adsorption of Eruca sativa on steel surface.

| T<br>($^0$C) | K | $\Delta H^o$<br>$kJmol^{-1}$ | $\Delta S^o$<br>$kJmol^{-1}K^{-1}$ | $\Delta G^o$<br>$kJmol^{-1}$ |
|---|---|---|---|---|
| 25 | 1.17 | -427 | 1.40 | - 10.29 |
| 40 | 1.11 | | 1.38 | - 10.68 |
| 50 | 1.05 | | 1.35 | - 10.87 |
| 60 | 1.00 | | 1.31 | - 11.07 |

The negative values of $\Delta G^o_{ads}$ indicate spontaneous adsorption of the inhibitor molecules on the carbon steel surface while their magnitude suggests the strong interaction between inhibitor molecules and the metal surface [37, 38].

Generally, the energy values of - 20kJ $mol^{-1}$ or less negative are associated with an electrostatic interaction between charged molecules and charged metal surface, physisorption; those of -40 kJ $mol^{-1}$ or more negative involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate covalent bond, chemisorption [39]. From this estimation, it can be concluded that the extract are physically adsorbed on the charged steel surface thus creating an electrostatic interaction [40].

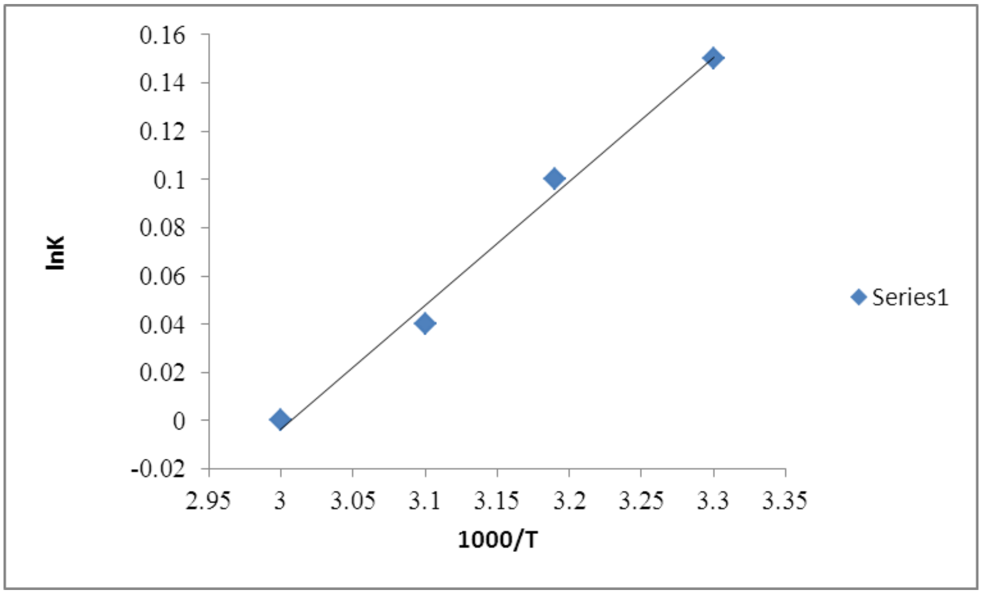

Fig 3: Van't Hoff plot

The adsorption heat, $\Delta H^{o}_{ads}$, can be calculated according to the Van't Hoff equation [41]

$$\ln K_{ads} = \left(\frac{-\Delta H^{o}_{ads}}{RT}\right) + const. \qquad (6)$$

The adsorption heat is obtained by plotting ln $K_{ads}$ and $1/T$ (Fig3). The adsorption heat could be approximately regarded as the standard heat of adsorption, $\Delta H^{o}_{ads}$, under experimental conditions.

According to the thermodynamic basic equation, the standard entropy of adsorption $\Delta S^{o}_{ads}$ could be calculated from the following equation [42]:

$$\Delta G^{o}_{ads}=\Delta H^{o}_{ads}-T\Delta S^{o}_{ads} \qquad (7)$$

All the obtained thermodynamic parameters are listed in Table 2. In any case, the negative sign of $\Delta H^{o}_{ads}$ suggests that the adsorption of inhibitor is an exothermic process, which means that inhibition efficiency decreases with raising the temperature. The behavior can be interpreted on the basis that increasing temperature leads to the increase of the desorption amount of the adsorbed inhibitor molecules from the steel surface.

### *3.3. Kinetic parameters*

The kinetic model represents an important tool for further explanation of the corrosion inhibition mechanism. Fig.4 represents the plots of the logarithm of the corrosion rate $R_{corr}$ (mg $cm^{-2}h^{-1}$) of carbon steel vs. 1000/$T$ for carbon steel in 0.1M $H_2SO_4$ in absence and presence of inhibitor. The apparent activation energy ($E_a$) was calculated by using Arhenuis equation [43]:

$$\ln(R_{corr}) = \frac{-Ea}{RT} + A \qquad (8)$$

where $E_a$ is the apparent activation energy of carbon steel corrosion in 0.1M $H_2SO_4$ solution, $R$ is the general gas constant, $A$ is the Arrhenius pre-exponential factor and $T$ is the absolute temperature. The values of $E_a$ were calculated from the slopes of the straight lines.

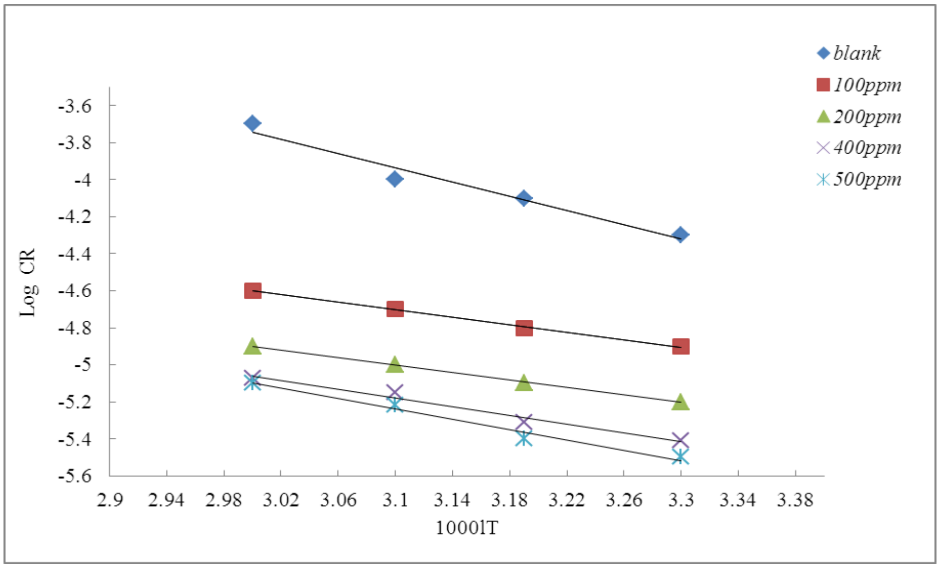

Fig4: Relation between logCR and the reciprocal of the absolute temperature of steel in 0.1M $H_2SO_4$ containing different concentration of inhibitor, blank, 100ppm, 300ppm, 400ppm, 500ppm.

Table 3: Activation parameters of the dissolution of steel in 0.1M $H_2SO_4$ in the absence and presence of inhibitor.

| Conc. ppm | $E_a$ $kJmol^{-1}$ | ΔH $kJmol^{-1}$ | ΔS $Jmol^{-1}$ |
|---|---|---|---|
| 0.0 | 24.8 | 193 | 9.2 |
| 100 | 57.4 | 268 | 2.1 |
| 200 | 59.3 | 243 | 3.4 |
| 300 | 55.5 | 243 | 3.4 |
| 400 | 59.3 | 294 | 3.4 |
| 500 | 56.0 | 281 | 1.2 |

The values of $E_a$ in the table are higher for inhibited solution than the uninhibited one. This indicates a strong inhibitive action of the additives by increasing the energy barrier for the corrosion process, emphasizing the electrostatic character of the inhibitor's adsorption on the steel surface (physisorption) [20]. However, such type of inhibitors performs a good inhibition at ordinary temperature with considerable loss in inhibition efficiency at elevated temperatures. An alternative formula of the Arrhenius equation is the transition state equation [44]:

$$R_{corr} = \frac{RT}{Nh} \exp\left(\frac{\Delta S^*}{R}\right) \exp\left(\frac{-\Delta H^*}{RT}\right) \qquad (9)$$

Where, $h$ is the Planck's constant, $N$ the Avogadro's number, $\Delta S^*$ the entropy of activation, and $\Delta H^*$ the enthalpy of activation. A straight line relationship is obtained by plotting ln ($R_{corr}/T$) vs. $1000/T$ (Fig.5) with a slope of ($-\Delta H^*/R$) and an intercept of $[(\ln(R/Nh)) + (\Delta S^*/R)]$, from which the values of $\Delta S^*$ and $\Delta H^*$ were calculated and listed in Table3.

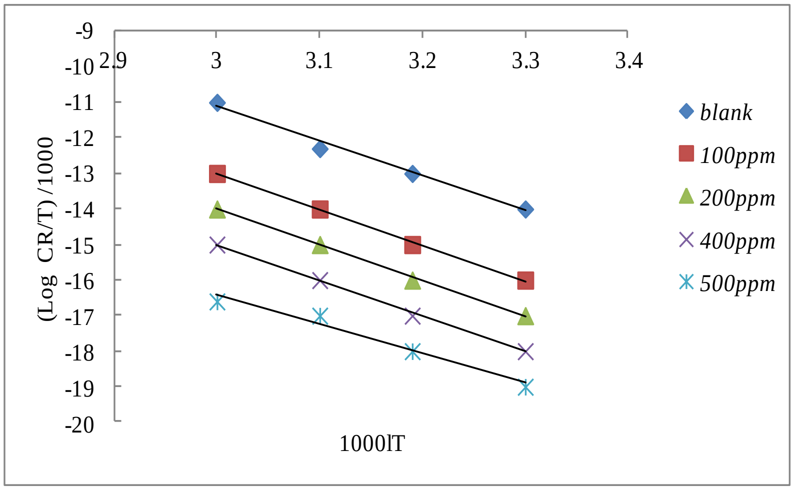

Fig5: Arrhenius plot of log (CR/T) against (1/T) for the dissolution of steel in 0.1M $H_2SO_4$ in the absence and presence of inhibitor.

The positive signs of the activation enthalpy ($\Delta H^*$) reflects the endothermic nature of the steel dissolution process. The $\Delta S^*$ values are positive, which are opposite to the usual expectation that the adsorption is an exothermic process and always accompanied by a decrease of entropy. The reason can be explained as follows: the adsorption of organic inhibitor molecules from the aqueous solution can be regarded as a quasi-substitution process between the organic compounds in the aqueous phase [Org(sol)] and water molecules at the electrode surface [$H_2O$(ads)] . In this situation, the adsorption of extract is accompanied by the desorption of water molecules from the electrode surface. Thus, while the adsorption process for the inhibitor is believed to be exothermic and associated with a decrease in entropy of the solute, the opposite is true for the solvent. The thermodynamic values obtained are the algebraic sum of the adsorption of organic inhibitor molecules and the desorption of water molecules. Therefore, the gain in entropy is attributed to the increase in solvent entropy. The positive values of $\Delta S^*$ suggest that the adsorption process is accompanied by an increase in entropy, which is the driving force for the adsorption of extract on the C-steel surface [45].

### 3.4. Polarization studies.

The effect of addition of various concentration of the natural extracted compound on the anodic and cathodic polarization curves of steel in $0.1MH_2SO_4$ solution at $25^0C$ Fig6.

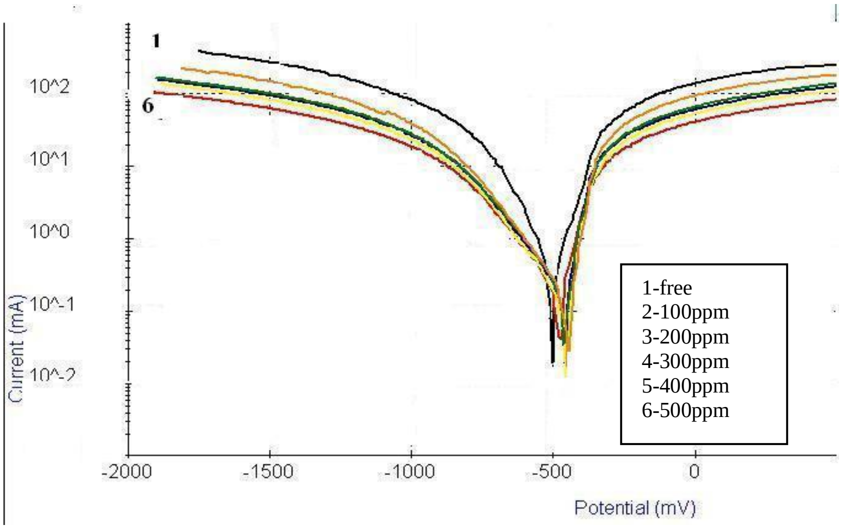

Fig.6. Polarization curves of steel in free and inhibited acid solutions

Electrochemical parameters such as corrosion current density ($i_{corr}$), corrosion potential ($E_{corr}$), Tafel constant ($b_a$ and $b_c$), and percentage inhibition efficiency (*IE %*) were calculated from Tafel plots shown in Table 4.

Table 4: Electrochemical parameters for steel in absence and presence of inhibitor in $0.1MH_2SO_4$ solution at $25^0C$ obtained from Tafel polarization curves.

| Conc. ppm | $-E_{corr}$ mv | $-B_C$ mv/dec | $B_A$ mv/dec | $C_R$ | $I_{corr}$ $\mu A/cm^2$ | IE% |
|---|---|---|---|---|---|---|
| Free | -654 | -235 | 464 | 185 | 8.4 | ---- |
| 50 | -670 | -376 | 524 | 14.6 | 1.9 | 77 |
| 80 | -554 | -433 | 359 | 26 | 1.2 | 85 |
| 100 | -531 | -244 | 153 | 19.3 | 0.88 | 89 |
| 200 | -517 | -243 | 143 | 11.3 | 0.52 | 93 |
| 300 | -463 | -280 | 99 | 9.2 | 0.42 | 95 |
| 400 | -442 | -261 | 69 | 6.1 | 0.28 | 96 |
| 500 | -436 | -240 | 62 | 5.5 | 0.27 | 96 |

It can be observed from the potentiodynamic polarization curves that the presence of extract caused a decrease in both the anodic and cathodic current densities, with a more pronounced drop in the anodic branch. These results could be explained by the adsorption of organic compounds present in the extracts at the active sites of the electrode surface, which also led to the retardation of metallic dissolution and hydrogen evolution and consequently slowed the corrosion process.

Consequently, the ($I_{corr}$) decreased with increasing the concentration of the extracts, indicating that the inhibitive property of these extracts on the corrosion of steel in acid solutions. Demonstrating that this extract acts as a mixed-type inhibitor with predominantly anodic characteristics

## 4. Conclusions

- The *Eruca sativa* Extract act as good inhibitors for acid corrosion of C-steel.
- The inhibition efficiency increases with increased concentration
- The inhibition efficiency decreases as the temperature was increased.
- The adsorption of extract on steel surface is a spontaneous process and follows Langmuir adsorption isotherm.

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