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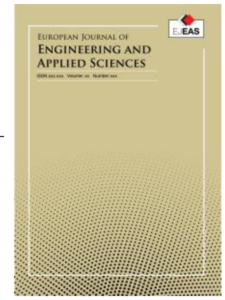

Research Article

Investigation of Temperature Correlations on Corrosion Inhibition of Carbon Steel in Acid Media by Flower Extract

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Abstract: Temperature relationships of Vigna subteranea flower (VSFE) extract on the corrosion of carbon steel in 2.0 M H_2SO_4 at 303-323 ˚K was studied by weight loss method. The obtained results show that VSFE extract acts as inhibitor for carbon steel in H_2SO_4 solution. The inhibition efficiency was found to increase with increase in VSFE extract concentration but decreased with acid concentration and temperature, which is suggestive of physical adsorption mechanism although chemisorption may play a part. The adsorption of VSFE onto the carbon steel surface was found to follow the Langmuir adsorption isotherm. The correlation coefficient (R^2) ranging from $0.9992 \geq R^2 \geq 0.9715$ was obtained. Both kinetic parameters (activation energy, pre-exponential factor, enthalpy of activation and entropy of activation) and thermodynamics of adsorption (enthalpy of adsorption, entropy of adsorption and Gibbs free energy) were evaluated and discussed from the effect of temperature on the corrosion and inhibition processes.

Keywords: *Acid corrosion, Carbon steel, Inhibition, Temperature correlations, Vigna subteranea flower extract*

Bitki Ekstraktı ile Asidik Ortamda Karbon Çeliğinin Korozyon İnhibasyonu Üzerine Sıcaklık Korelasyonunun İncelenmesi

Öz: Vigna subteranea çiçeği (VSFE) ekstraktının karbon çeliğinin korozyonunda sıcaklık ilişkisi 303-323 ˚K'de 2 M H_2SO_4 kullanılarak ağırlık kaybı metodu ile incelenmiştir. Elde edilen sonuçlara göre, VSFE karbon çeliği için H_2SO_4 çözeltisinde inhibitor gibi davranmaktadır. İnhbisyon veriminin VSFE ekstraktı konsantrasyonunun artışı ile arttığı, asit konsantrasyonu ve sıcaklıkla azaldığı belirlenmiştir. Buna göre, bir kısım kemisorpsiyon söz konusu olsa da, fiziksel adsorpsiyon mekanizması baskın bulunmuştur. VSFE'nin karbon çeliği yüzeyinde adsorpsiyonunun Langmuir adsorpsiyon isotermine uyduğu belirlenmiştir. Korelasyon katsayısının (R^2) $0.9992 \geq R^2 \geq 0.9715$ aralığında olduğu bulunmuştur. İki kinetik parametre de (aktivasyon enerjisi, pre-eksponansiyel faktör, aktivasyon entalpisi ve aktivasyon entropisi) ve adsorpsiyon termodinamiği (adsorpsiyon enthalpisi, adsorpsiyon entropisi ve Gibbs serbest enerjisi) değerlendirilmiş ve inhibisyon prosesi ve korozyona sıcaklık etkisi açısıdan tartışılmıştır.

Anahtar kelimeler: *Asit korozyonu, Karbon çeliği, Inhibisyon, Sıcaklık korelasyonu,* Vigna subteranea çiçeği ekstraktı

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1. Introduction

Carbon steel has its application in Engineering construction, as well as in chemical and allied industries for holding alkalis, acids and salt solutions due to its availability, cheap and simple construction. Sequel to this carbon steel disintegrates when it comes in contact with the above industrial conditions [1, 2] and [3]. The consequences of the chemicals on metals can be reduced or protected efficiently by application natural inhibitors. According to a study of Ugi, et al, [4], H_2SO_4 is one of the major chemical used globally in almost all the industries like paper bleaching, cellulose fibers, pharmaceuticals, sugar bleaching, fertilizers, automobile batteries, water and coloring agents, sulfonation agents, treatment, amino acid intermediates, steel manufacturing, iron and steel pickling, gasoline, regeneration of ion exchange resins. Tetraoxosulphate VI acid popularly known as Sulfuric acid (H_2SO_4) is an important mineral acid that has substantial application in many fields has led researchers to investigate its effect of corrosion inhibitors. Furthermore, it is used in pickling solution for steel alloys. Influence of temperature on acidic corrosion and corrosion inhibition of iron and steel especially in H_2SO_4 solutions has acknowledged by a numerous investigations and researchers recently. The dependence of temperature on inhibitor efficiency (η) and the evaluation of thermodynamic data obtained for the corrosion process both in absence and presence of inhibitors was instrumental to some inferences regarding the inhibition mechanism [5-8]. The objective of this current work is to assess the influence of temperature on carbon steel corrosion in 2.0 M H_2SO_4 solutions in the absence and presence of different concentrations of *Vigna subteranea* flower extract using gravimetric method. Different thermodynamic variables for inhibitor adsorption on carbon steel surface were estimated and discussed. Kinetic parameters for carbon steel corrosion in absence and presence of the studied inhibitors were evaluated and interpreted.

2. Materials and Methods

2.1 Materials and Solution Preparation

Carbon steel sheets were sourced from Awka, Nigeria. The Carbon steel sheet was subsequently cut into coupons of 4 x 7 x 0.17cm polished, degreased and cleaned for further use as previous described in [9]. All reagents, chemicals used were of analytical grade and double distilled water was used for preparing solutions. The concentrated plant flower extracts were used for the preparation of inhibitor test solutions in the concentration range of 0.1mol/100 ml solution of 2.0 M H_2SO_4.

2.2 Sourcing of Plant Shells and Preparation of Extracts

The flower extracts used in this study were derived from *Vigna subteranea* plant sourced locally in Enugu state. The flowers were plucked and collected for analysis. Two hundred grams of sun dried and powdered flower were extracted with 95% ethanol at room temperature for 48hrs. The extracts were concentrated until the solvents were totally removed. The concentrated extracts were kept for advance use [10].

2.3 Weights Loss Measurements

The corrodent concentration was 0.2 mols and the volume of the test solution used was 100 ml. All tests were carried out in aerated solutions. The difference between the weight at a given time and the initial weight of the coupons was taken as the weight loss which was used to evaluate the corrosion rate and inhibition efficiency for carbon steel with different inhibitor concentrations as:

2.3.1 Corrosion Rate (CR)

The corrosion rate (CR) was calculated using Equation (1) [13, 14]

$$CR = \frac{M_1 - M_2}{At} \quad (1)$$

Where M_1 is weight in (mg) before immersion, M_2 is weight in (mg) after immersion, A is area in (cm^2) of the specimen, and t is the exposure time in (hours).

2.3.2 Inhibition efficiency (η%)

The inhibition efficiency (η%) of the extract was estimated using Equation (2); [12, 13]

$$\eta\% = \left[\frac{W_{blank} - W_{inh}}{W_{blank}}\right] \times 100 \quad (2)$$

Where W_{blank} and W_{inh} are weight losses in the absence and presence of inhibitor respectively.

The degree of surface coverage (θ) was computed from equation (3); [14]:

$$\theta = 1 - \frac{W_{inh}}{W_{blank}} \quad (3)$$

3. Results and Discussion

3.1 Effect of Temperature on Corrosion Rate Carbon Steel

The data of corrosion rate were obtained using Equation (1). The calculated values of corrosion rates of carbon steel in free and inhibited concentrations of VSFE extracts at varying temperatures are presented in Table 1. The data indicates that the rate of corrosion increases with increase in temperature and decrease on addition of inhibitors in all cases studied. The results of corrosion rates of carbon steel in 2.0 M H_2S0_4 are shown in Table 1 follows Arrhenius reactions since increase in temperature progressively increases corrosion rate. Assuming the rate of corrosion of carbon steel against the concentration of inhibitors is in line with the kinetic relationship according to [7] then,

$$\log CR = logK + AlogC_{inh} \quad (4)$$

Where *k* is the rate constant and is equal to *CR* at inhibitor concentration of unity, *A* is the reaction constant and a measure of inhibitor effectiveness and C_{inh} is the concentration of the VSFE extract. The plots of Figure 1 provides information on how the kinetic parameters (K and A) were evaluated from Equation (4) and presented in Table 2

Table 1 Calculated values of corrosion rates (mg/cm²hr) and inhibition efficiencies of carbon steel in 2.0 M H_2S0_4

Inhibitor	Con. Inh. (g/l)	CR (mg/cm²hr)			η (%)		
		303 ˚K	323 ˚K	343 ˚K	303 ˚K	323 ˚K	343 ˚K
VSFE	0	26.5	28.5	30.7	-	-	-
	0.2	16.5	19.5	21.3	37.7	31.6	30.0
	0.4	14.1	17.5	17.5	47.2	38.6	41.7
	0.6	10.3	14.2	15.5	62.3	50.9	48.3
	0.8	9.1	12.5	13.5	66.0	56.1	55.0
	1	7.4	9.5	12	73.6	63.2	60.0

The values of reaction constant A from Table 2 were all negative implying that the rate of corrosion process is inversely proportional to the inhibitor concentration, that is gradual increase in concentration of the extracts cause improvement in the inhibitor efficiency and effectiveness. This can be attributed to high negative value of constant *A* due to alteration of CR and extract concentration reproducing enhanced inhibitive properties for all the inhibitors studied. Furthermore, constant values of A can be used in comparing the inhibitive performance of two acids at varying temperatures. A values were negative at all temperatures revealing the active nature of H_2S04. Also, progressive increase in temperature points to increase in inhibitory activity of VSFE extract. Table 2 equally disclose that the values of k rises as the temperature moves up progressively.

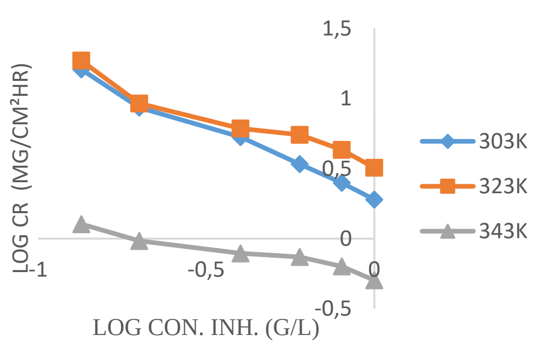

***Figure 1.** Plot of log CR versus log C_{inh} of carbon steel corrosion in 2.0 M H_2SO_4 containing VSFE extract at varying temperatures.*

Table 2 Kinetic parameters for corrosion of carbon in 2.0 MH_2SO_4

Inhibitor	Temp (K)	Kinetic parameters	
		A	K
VSFE	303	-0.5834	0.8527
	323	-0.4483	1.0044
	343	-0.4201	1.0844

3.2 Influence of Temperature on Inhibition Efficiency (η %)

Table 1 presents the relationship between η (%) with VSFE extract concentrations at different temperatures in 2.0 M of H_2SO_4. The result of Table 1 reveals that in the presence of H_2SO_4 the inhibition efficiency increased with increase in the inhibitor concentrations. This is attributed to the adsorption of inhibitor molecules on the carbon steel/solution interface thereby preventing the activity of the aggressive environment. The effect of temperature on inhibition efficiency of the inhibitor at all concentrations and temperatures studied showed a significant increase in the inhibitor efficiency as the temperature rises to 343K. Chemical adsorption mechanism was proposed due the fact that increase in temperature leads to increasing efficiency. Inhibition efficiency was calculated from Equation (2). Due to several transformations resulting from rapid etching and desorption as well as inhibitor decomposition and/or rearrangement on the surface of the metal, the effect of temperature on the inhibited acid-metal reaction is highly complex. Although, close observation revealed that some inhibitors can work efficiently at high temperature or low temperature [15-18] with acid-metal systems.

3.3 Adsorption Considerations

The experimental data for *Vigna subteranea* flower extract generated from Equation (3) was fitted into Langmuir adsorption isotherm and a number of expressions were obtained for the isotherm at equilibrium. It is important to note the degree of surface coverage θ differs with the concentrations of the inhibitors at all temperatures investigated [19]. Langmuir adsorption isotherm model, can be expressed mathematically as in Equation (5); [6, 19-21].

$$\frac{C}{\theta} = \frac{1}{Kads} + C \quad (5)$$

Where *C* is the inhibitor bulk concentration in g/l, K_{ads} is the equilibrium constant of adsorption.

Figure 2 represents the plot of Log (C/θ) versus Log C signifying that the adsorption of the plant extracts is in line with Langmuir isotherm. The adsorption parameters were calculated and presented in Table 3. The adsorption isotherm used has correlation coefficients (R^2) ranging from $0.9992 \geq R^2 \geq 0.9715$ with slope almost equal to 1. K_{ads} rises with rise in temperature [22] as seen in Table 3.

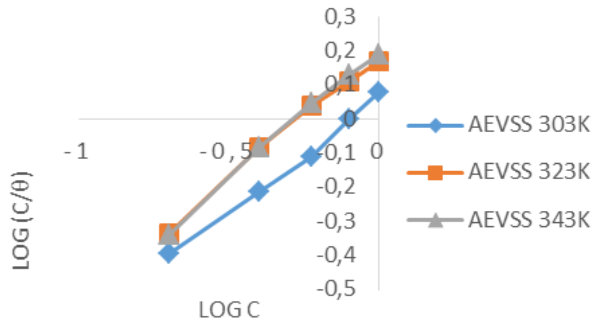

Figure 2. *Langmuir adsorption isotherm for carbon steel corrosion in H_2SO_4*

Table 3 Adsorption parameters obtained from Langmuir adsorption isotherm

Inhibitor	Temp (K)	Adsorption parameters		
		R^2	K_{ads}	slope
VSFE	303	0.9969	0.1827	2.4909
	323	0.9964	0.3431	1.3573
	343	0.9915	0.0749	3.9510

Technique 1

The free energy of adsorption ($\triangle G_{ads}$) was related to equilibrium constant (K) of the inhibitor constituents by applying Equation (6) [23, 24] and shown in

$$K_{ads} = \frac{1}{C_{H20}} \exp\left(\frac{-\Delta G^{\circ}{}_{ads}}{RT}\right) \tag{6}$$

The plot gives a linear relationship as indicated in Figure 3 with $\triangle H_{ads}$ as intercept and putting the values of $\triangle H_{ads}$ obtained into Equation (7) the values of $\triangle S_{ads}$ with other thermodynamic properties studied were computed at all temperatures.

3.4 Thermodynamic Studies

The universally accepted thermodynamic adsorption parameters include the heat of adsorption ($\triangle H_{ads}$) the entropy of adsorption ($\triangle S_{ads}$), and the free energy of adsorption ($\triangle G_{ads.}$). These parameters can be calculated using different mathematical methods depending on the values of K_{ads} from adsorption isotherms, at different temperatures:

Where C_{H20} is the concentration of water molecules (mol/L) at metal/solution interface, K_{ads} is the equilibrium constant signifying the strength between adsorbate and adsorbent [25]. Then, the obtained $\triangle G_{ads}$ values were plotted against T (Figure 3) in accordance with the basic equation [26]:

$$\Delta G_{ads} = \Delta H_{ads} - T\Delta S_{ads} \tag{7}$$

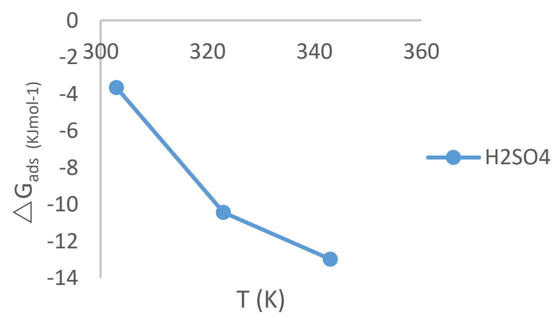

Figure 3. *Plot of $\triangle G_{ads}$ against T*

Technique 2

The K_{ads} depends on temperature as can be seen from Equation (8) below;

$$K_{ads} = exp\left(\frac{\Delta S_{ads}}{T}\right) exp\left(-\frac{\Delta H_{ads}}{RT}\right) \tag{8}$$

Rewriting Equation (8):

$$\log K_{ads} = \frac{\Delta S_{ads}}{2.303R} - \frac{\Delta H_{ads}}{2.303RR} \tag{9}$$

Here, plot of K_{ads} against 1/T Figure 4 gives a straight line slope of $-\frac{\Delta H_{ads}}{2.303R}$. By putting the calculated values of $\triangle H_{ads}$ into Equation (9) the data for $\triangle S_{ads}$ was equally computed for all temperatures. Then the values of $\triangle S_{ads}$ and $\triangle H_{ads}$ was combined using Equation (7) to get $\triangle G_{ads}$ values.

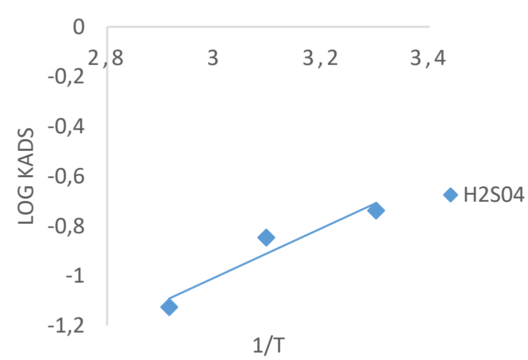

Figure 4. *Plot of K_{ads} against 1/T*

Technique 3

Gibbs-Helmholtz Equation [27, 28]

$$\left[\frac{\partial(\Delta G_{ads}/T)}{\partial T}\right]p = -\frac{\Delta H_{ads}}{T^2} \tag{10}$$

Integrating (10);

$$\int \frac{\partial(\Delta G_{ads})}{T} = -\int \frac{\Delta H_{ads}}{T^2} dT \tag{11}$$

Equation (11) can be gotten from Equation (10) as

$$\frac{\Delta G_{ads}}{T} = \frac{\Delta H_{ads}}{T} + constant\ t \quad (12)$$

The relationship between ${\Delta G_{ads}}/{T}$ and ${1}/{T}$ gives a straight line with $\triangle H_{ads}$ as the slope Figure 5. The free energy of adsorption $\triangle G_{ads}$ was computed as done in technique 1 from Equation (6). Then Equation (7) was used to calculate $\triangle S_{ads}$ and other thermodynamic parameters as in technique 1 for the all the systems and presented in Table 4.

Technique 4

The heat of adsorption was computed using Van Hoff equation [29-31]:

$$\log K_{ads} = -\frac{\Delta H_{ads}}{2.303RT} + constant\ t \quad (13)$$

Also, Figure 4 denotes the correlation Equation (13) with a straight line slope of $\triangle H_{ads}$ in 2.0 M H_2SO_4 solutions. Equations (6) and (7) were used to evaluate $\triangle G_{ads}$ and $\triangle S_{ads}$ accordingly.

Technique 5

By adding $\triangle G_{ads}$ term in Equation (6), the terms in Equation (6) equals that in Equation (7) and a new expression was obtained as shown below

$$K_{ads} = \frac{1}{C_{H2O}} exp\left[\frac{\Delta S_{ads}}{T} - \frac{\Delta H_{ads}}{RT}\right] \quad (14)$$

Eq. (14) can be rewritten as;

$$logK_{ads} = \left[-\log C_{H2O} + \frac{\Delta S_{ads}}{2.303R}\right] - \frac{\Delta H_{ads}}{2.303RT} \quad (15)$$

Equation (15) is comparable with Eq. (9) except the intercept, where a new term was introduced in the intercept of Equation (15), this is $C_{H\ 2O}$ –log [32]. Figure 4 also represents the relation in Equation (15). Although it can be used to compute $\triangle H_{ads}$ whereas $\triangle S_{ads}$ and $\triangle G_{ads}$ can be evaluated from Equations (6 and 7). Hence forward, the negative values of $\triangle G_{ads}$ reveals the spontaneous nature of the extracts on the carbon steel surface [33-35]. The occurrence of endothermic process, results in decrease of $\triangle G_{ads}$ (becomes more negative) with increasing temperature which favours inhibitor adsorption on the metal surface. Additionally, the heat of adsorption ($\triangle H_{ads}$) provides a useful information on the mechanism of inhibitor adsorption. ($\triangle H_{ads}$) shows positive values for all the range of temperatures studied showing that the corrosion inhibition of carbon steel in 2.0 M H_2SO_4 solutions proceeds by chemical adsorption for the studied inhibitor species on the metal surface [36-38]. The negative $\triangle S_{ads}$ values was complemented with decrease in the entropy energy change which occurs whenever the adsorption process is exothermic [39-41]. The effect of temperature can be clearly established using Arrhenius and transition state equations (Equations 16 and 17) [42-44].

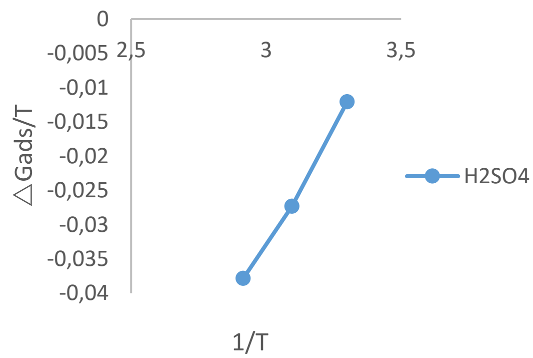

Figure 5. *Plot of $\triangle G_{ads}/T$ against 1/T*

$$log\ CR = \log A - \frac{E_a}{2.303RT} \quad (16)$$

Where, A is the pre-exponential factor, E_a is the activation energy, R is the universal gas constant, T is the absolute temperature. Figure 6 gives a linear relationship between logs CR versus 1/T. from which the Ea values were evaluated. The entropy of activation, ΔS°, the enthalpy of activation ΔH°, were obtained using Transition state Equation (17) [42].

$$log\left(\frac{CR}{T}\right) = \left[\left(log\left(\frac{R}{Nh}\right)\right) + \left(\frac{\Delta S^*}{2.303R}\right)\right] - \frac{\Delta H^*}{2.303RT} \quad (17)$$

N is the Avogadro number, h is the Planck constant. A plot of log (CR/T) versus 1/T as shown in Figure 7 gives a straight line with a slope of (−ΔH*/2.303 R) and an intercept of log (R/Nh + ΔS°/2.303 R) from which the values of ΔH*and ΔS* were deduced.

The gradual addition of VSFE extract causes increase in activation energy to a value lower than that of the blank solution implying that the action of VSFE extract on carbon steel corrosion in 2.0 M H_2SO_4 solutions was through chemical adsorption. The values of Ea are bigger than the values of $\triangle H^*$ indicating that the process involves gaseous reaction which cause reduction in reaction volume. The negative values entropy of activation ($\triangle S^*$) in the absence and presence of inhibitor show that the activated complex in the rate determining step represents an association rather than dissociation, meaning that, a decrease in disordering takes place on going from reactants to the activated complex [43]. The more negative ($\triangle S^*$) is the high the inhibition efficiency.

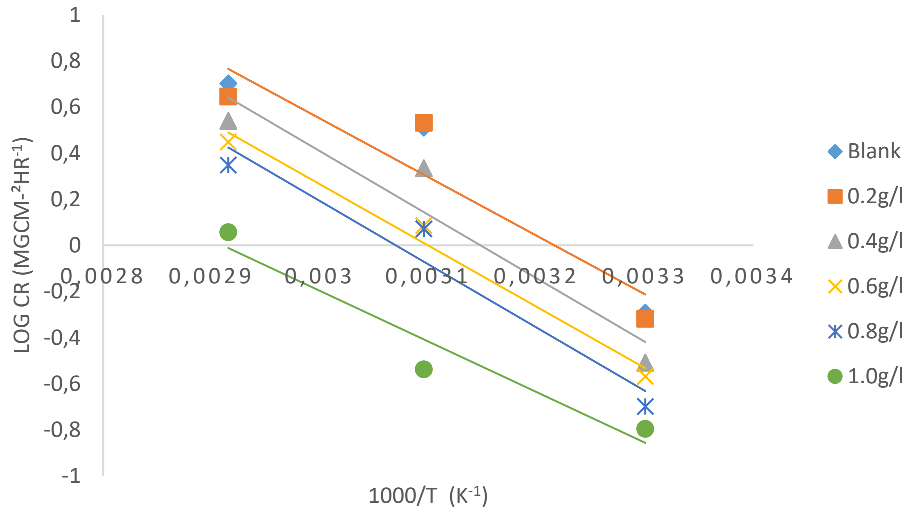

Figure. 6. *Arrhenius plots of log CR against 1/T for carbon steel in H_2SO_4*

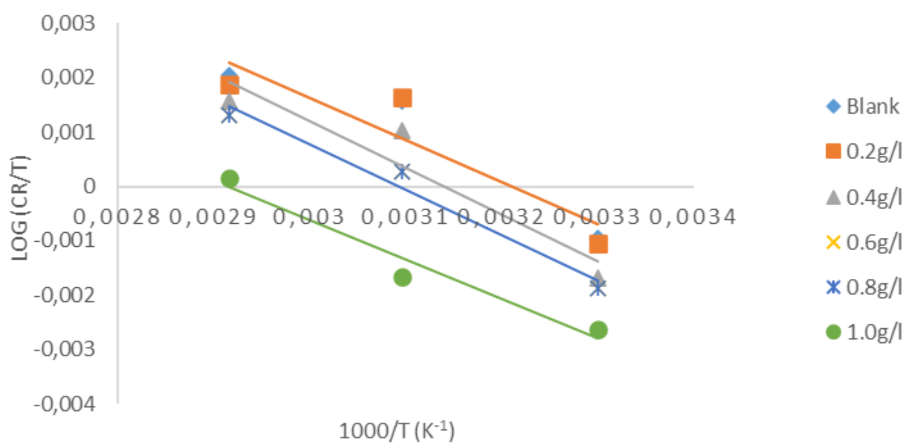

Figure 7. *Transition state plot of log CR/T against 1/T for carbon steel in H_2SO*

4. Conclusions

The inhibition efficiency of *Vigna subteranea* flower extract in 2.0M H_2SO_4, increases with increasing extract concentration and solution temperature. The extract inhibits carbon steel corrosion in H_2SO_4 at all inhibitor concentrations and solution temperatures. The retarding action of *Vigna subteranea* flower extract in 2.0M H_2SO_4, were done by adsorption of the extract species on carbon steel surface. The adsorption process is spontaneous and obeys Langmuir adsorption isotherm in H_2SO_4 at all temperatures studied. Thermodynamic data for both inhibitor adsorption and carbon steel corrosion proposes the existence of chemical adsorption for the inhibitor species on carbon steel from H_2SO_4 solution.

Author Contribution

Data curation - Author Name Surname(NS); Formal analysis - NS; investigation - NS; Experimental Performance - NS; Data Collection - NS;, Processing - NS; Literature review - NS; Writing - NS; review and editing – NS.

Declaration of Competing Interest

The authors declare no conflicts of interest with respect to the research, authorship, and/or publication of this article.

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