

# Isotherm, Kinetic and Thermodynamic Studies for the Sorption of Mercury (II) onto Activated Carbon from *Rosmarinus officinalis* Leaves

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## Abstract

The present work deals with the equilibrium adsorption of Hg(II) onto carbonized *Rosmarinus officinalis* leaves (ACROL) as a new adsorbent from aqueous solution and it has been investigated. ACROL samples were prepared by physical carbonization at 773 K for 1 h. Titration method was used to determine the concentration of Hg(II) before and after adsorption onto ACROL by ethylenediaminetetraacetic acid, EDTA, as chelating agent. Batch equilibrium studies were carried out under different experimental conditions such as Hg(II) concentration and temperature. The relationship between the amount of Hg(II) onto ACROL can be described using four two-parameter isotherm models. The equilibrium sorption data were analyzed using Freundlich, Langmuir, Dubinin-Radushkevich (DRK) and Temkin isotherms. The experimental results were found to fit the Langmuir isotherm model with a monolayer adsorption capacity of 588.2 mg/g at 318 K, while they were found to fit the Freundlich isotherm model at 298 K. The  $K_L$  was decreased with increasing temperature, indicating a bond strength between Hg(II) and ACROL decreased with increasing temperature and sorption is exothermic. From DRK isotherm, free energy,  $E$ , was higher than 31 kJ/mol suggesting the Hg(II) adsorption onto ACROL chemical sorption. The thermodynamic studies revealed that the process is spontaneous nature of Hg(II) adsorption by ACROL and exothermic. The findings from this research show that ACROL has capability to remove Hg(II) from aqueous solutions.

## Keywords

Adsorption, Hg(II), Isotherm, Kinetic, Thermodynamic, *Rosmarinus officinalis* Leaves

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## 1. Introduction

The removal of heavy metals released from industrial water into environments is a matter of great interest due to their toxicity and industrial activities in large amount of non-degradable heavy metals [1]. In 1999, 48 tons of mercury was emitted globally into atmosphere from coal-fire power plants in US [2] [3]. Hg(II) is water-soluble in water and can be converted to methylmercury in the presence of micro-organisms, which is a highly toxic form [4]. Mercury metal can be transported over long distance and may be bio-accumulated in the food chain like fish and interfere with ozone depletion in the arctic [3]. Increased applications of Hg(II) have led to increase in possible risks to the environment, so the removal of Hg(II) becomes crucial.

The treatment of waste waters from Hg(II) is one of the growing needs in environmental applications. However, several processes are useful to remove these heavy metals from water and wastewater including chemical precipitations, ion exchange, membrane filtration, adsorption flotation, and coagulation flocculation [5] [6]. Adsorption is one of the most widely used methods for removal of heavy metals from waste waters due to low-cost sorbent and eco-environmental materials [7]. Several adsorbents have been used to remove the heavy metals from waste waters such as olive stones, palm kernel oil, pumpkin seeds, melon seeds etc. [8]-[10]. *Rosmarinus officinalis* Leaves (ACROL) is used as a decorative plant in gardens and many medical uses [11]. Also, ACROL are used to flavor foods such as roast meats [11]. Due to the natural abundance in the environment and cheap especially in Libya, ACROL can be applied for heavy metal removal from aqueous solution. Also, in our previous work, we reported that the ACROL could remove Hg(II) from aqueous solution; in which the adsorption capacity depended on agitation time (75 min), pH of 2.4 and dosage of ACROL [12]. The aims of this research are to investigate the possibility of using ACROL as efficient alternative adsorbent materials to remove Hg(II) from aqueous solution using different isotherm models and to evaluate thermodynamic and kinetic of Hg(II) adsorption onto ACROL.

## 2. Materials and Methods

### 2.1. Chemicals and Instruments

All the chemicals used in this work were purchased from Merck, Germany and BDH, England and used without further purifications. The stock solution of Hg(II) was prepared by dissolving mercury acetate in distilled water and the desirable concentrations of Hg(II) were obtained by diluting the stock solution, in 100 - 400 mg/L range. The solution pH was measured by pH Jenway 3505 Model and adjusted using HCl and NaOH. Ethylenediaminetetraacetic acid (EDTA) was used for EDTA titration method. Xylenol orange was used as indicator. Conductivity meter (Philips, PW-9527); FT-IR (IR200) were used.

### 2.2. Sample Collection, Preparation and Carbonization

ACROL was collected from Sabha City, Libya (Figure 1) and washed with distilled water to remove fines and dirt. The raw sample was dried at 40°C for 3 h, crushed and sieved to a particle size with 90 µm mesh and then was later immersed in diethyl ether for overnight until the solvents evaporated in order to remove the volatile oils. Then, it was placed in crucible and carbonized at 773 K for 1 h. The carbon produced was allowed to cool to room temperature for overnight, sieved again with 90 µm, put in an air tight bottle and stored in desiccators [12].

### 2.3. Characteristics of ACROAL

The proximate composition of was determined according to Ofomaja *et al.*, 2010 with slightly modifications [13]. The ACROL solution pH, conductivity, salinity, solubility and hydroscopic moisture content were determined using different method using American Society for Testing and Materials (ASTM) such as D3838-80, D4607-94, D2866-94 and D 2867-99.

### 2.4. Batch Adsorption Studies

The Optimization parameters for Adsorption of Hg(II) ions onto ACROL from Aqueous Solutions were reported previously by our group [12]. In order to determine the ACROL-Hg(II) adsorption isotherms, 40 mg of ACROL was added to varied Hg(II) concentrations from 100 to 400 mg/L Hg(II) at constant pH 2.4, 300 rpm and T =



Figure 1. *Rosmarinus officinalis* leaves (ACROL samples site. \*Sample site.

298, 308 and 318 K. The ACROL-Hg(II) suspensions were placed on a stirring plate for 75 min to establish the equilibrium condition. Then, 10 ml aliquots of the ACROL-Hg(II) suspensions were filtered using filter paper (Whatman No. P4). The residual Hg(II) concentration was determined by EDTA titration method using xylenol orange as indicator.

## 2.5. Data Analysis

Equation (1) is the percent of Hg(II) removal by ACROL and Equation (2) is amount of adsorption at equilibrium ( $q_e$ , mg/g):

$$\text{sorption\%} = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

$$q_e = \left( \frac{C_o - C_e}{w} \right) v \quad (2)$$

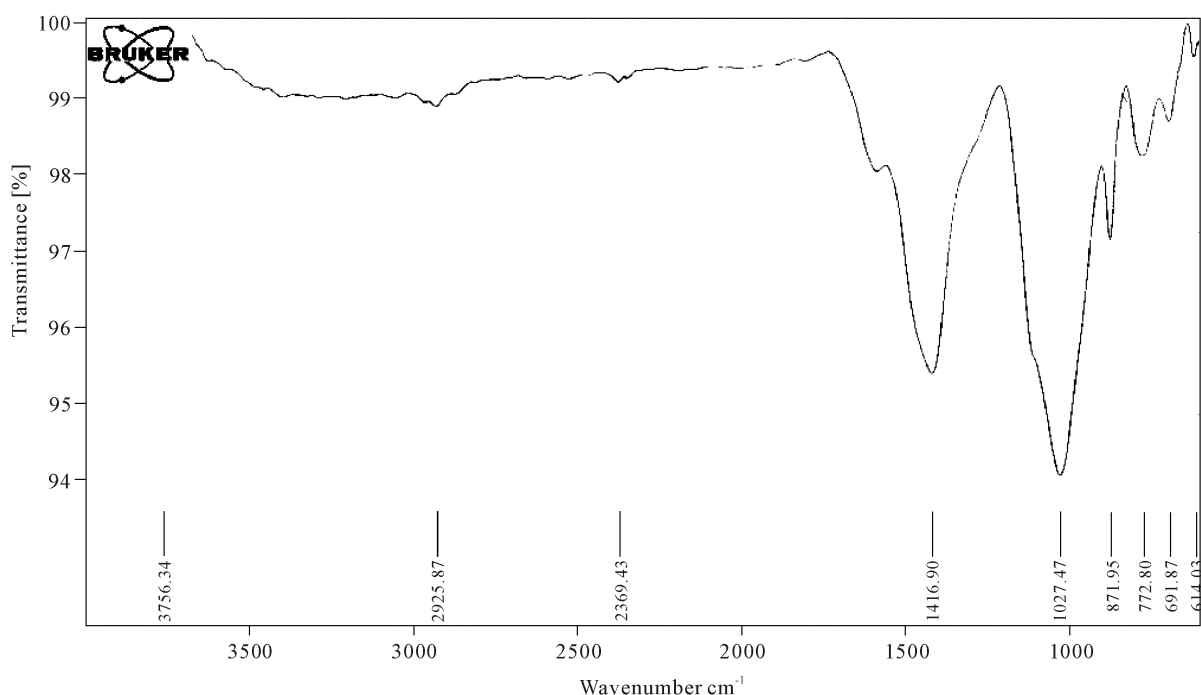
where:  $C_o$  and  $C_e$  = initial and equilibrium concentrations of Hg(II);  $v$  = total volume;  $w$  = weight of adsorbent. All the experiments were repeated for triplicate values.

## 3. Results and Discussion

### 3.1. Characterization of Biosorbent

Fourier transform infrared (FT-IR) spectroscopy was used to evaluate the functional groups on the surface of ACROL within the range of 600 - 4000  $\text{cm}^{-1}$ , as shown in Figure 2. FT-IR measurement for ACROL showed the presence of the following groups: O-H stretching (a broad peak at 3756.34  $\text{cm}^{-1}$ ), C-H stretching (a weak peak at 2925.87  $\text{cm}^{-1}$ ), C=O (1690.03  $\text{cm}^{-1}$ ), N-H (1416.90  $\text{cm}^{-1}$ ) and C-N (1027.47  $\text{cm}^{-1}$ ) [13].

Physico-chemical characteristics of ACROL are shown in Table 1. The characterization of ACROL as adsorbent is an important analysis to understand the behavior or mechanism of Hg(II) removal onto ACROL surface. Also, the extent of heavy metal adsorption onto biomass is influenced by the physico-chemical properties



**Figure 2.** FTIR spectrum of ACROL.

**Table 1.** Physico-chemical characteristics of ACROL.

Physico-chemical properties	Results
pH	7.58
Bulk density (g/cm <sup>3</sup> )	0.40
Moisture content (%)	1.33
Solubility (%)	0.202
Cellulose content (%)	65.0
Conductivity (μs/cm)	489
Salinity (ppm)	266

of biomass [14]. The value of pH 7.58 obtained in this research is useful for most applications. The moisture content tended to be low of ACROL indicating that activated carbon of ACROL was properly prepared and handled. Bulk density of ACROL was relatively high comparing to other activated carbon. The higher bulk density is the better filterability of activated carbons and the lower limit of bulk density for activated carbon application is 0.25 g/cm<sup>3</sup>. The conductivity and salinity values were noted to be higher than good conductivity for adsorption process ranged from 52 to 70 μs/cm [10]. This may be due to the presence of leachable ash as undesirable impurities and acid-water wash may not be able to reduced leachable ash.

## 3.2. Adsorption Isotherm

### 3.2.1. Freundlich Isotherm

The Freundlich isotherm is usually used for heterogeneous surface energy systems (non-uniform distribution of sorption heat) [15] [16]. Equation (3) is the Freundlich expression:

$$\log q_e = \log k_F + \frac{1}{n} \log C_e \quad (3)$$

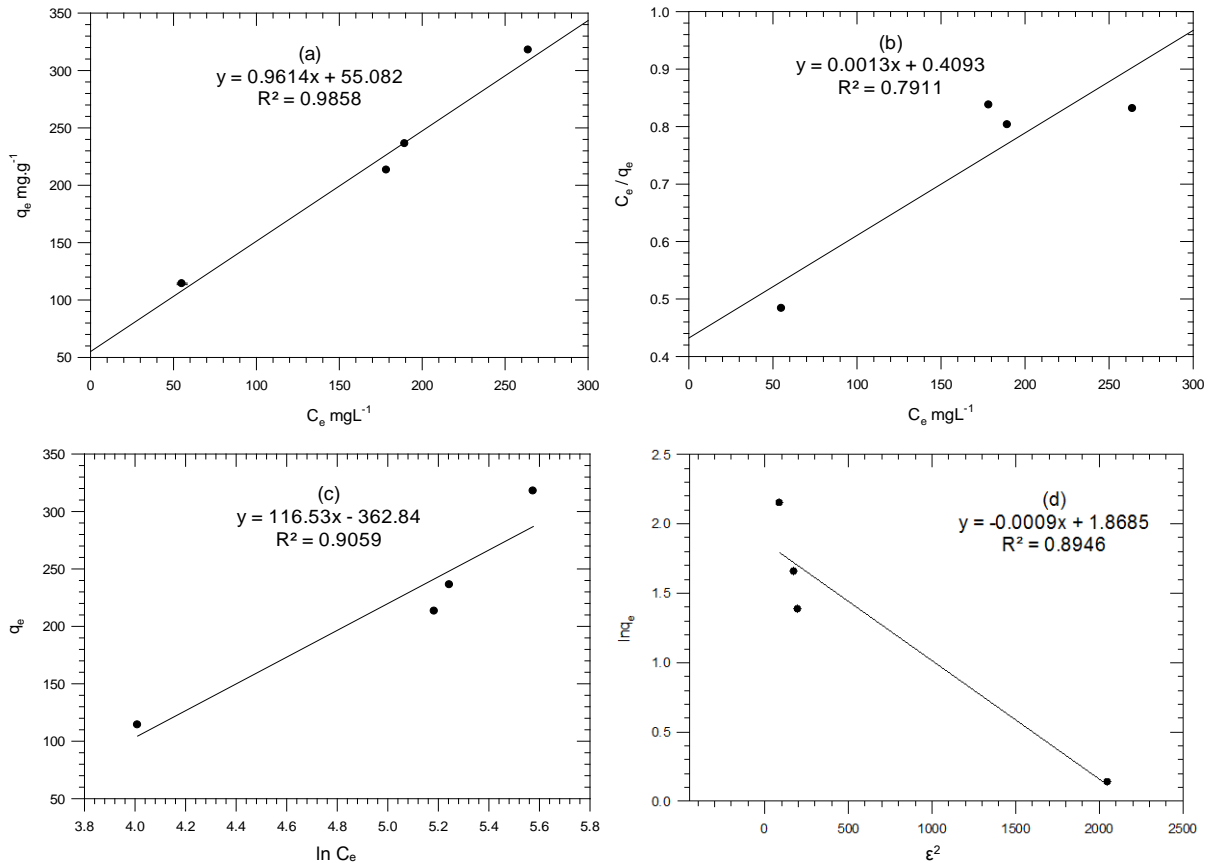
where:  $K_f$  and  $n$  are Freundlich constants, the  $K_f$  is adsorption capacity while  $n$  is biosorption intensity;  $q_e$  = amount of Hg(II) per unit mass of adsorbate (mg/g);  $C_e$  = equilibrium concentration (mg/L). The  $q_e$  versus  $C_e$  plot allows determining the Freundlich constants. The results of adsorption isotherm models are shown in **Figure 3(a)**. The adsorption constants and the correlation coefficients are also listed in **Table 2**. According to Freundlich isotherm, the  $1/n$  values lie between 0 and 1, this indicates favorable Hg(II) adsorption onto ACROL at low concentration and strong bond between Hg(II) and ACROL surface [17] [18]. Also,  $n$  values have the same magnitude suggesting that the retention of Hg(II) ions from solution take place by ionic interactions in the first moment [19]. The Freundlich equation represents the best fit experimental data with  $R^2 = 0.9818$  than other isotherm equations at low temperature.

### 3.2.2. Langmuir Isotherm

The Langmuir isotherm is usually used for a monolayer adsorption at specific homogenous sites on ACROL surface [16] [20]. Equation (4) is the Langmuir expression:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (4)$$

where:  $q_m$  = monolayer sorption capacity (mg/g) and  $K_L$  = Langmuir equilibrium constant (L/g) [15]. A plot of  $C_e/q_e$  versus  $C_e$  yields a straight line, shown in **Figure 3(b)** and values are listed in **Table 2**. According to Langmuir model,  $q_m$  values are increased with increasing temperature solution indicating that the Hg(II) ions is favorable adsorbed by ACROL at low temperature. However,  $K_L$  values are decreased with increasing temperature of solution from 298 to 318 K, therefore, less adsorption of Hg(II) onto the surface of ACROL would be



**Figure 3.** Plots for Freundlich (a), Langmuir (b), Temkin (c) and DRK (d) isotherms for Hg(II)-ACROL.

**Table 2.** Adsorption isotherm constants of Hg(II) ions on ACROL.

<i>T</i>	Freundlich constants			Langmuir constants			
	$K_f$	$1/n$	$R^2$	$K_L$	$q_m$	$R_L$	$R^2$
K	L/g			L/g	L/mg	Range	
298	0.5761	0.4518	0.9818	0.011	294.1	2.150 - 5.462	0.9709
308	0.9116	0.651	0.9723	0.005	454.6	1.516 - 3.004	0.9767
318	1.2555	0.7282	0.9286	0.004	588.2	1.418 - 2.619	0.956

<i>T</i>	Temkin constants				DRK constants			
	$K_T$	$q_m$	$B$	$R^2$	$B$	$q_m$	$E$	$R^2$
K	J/mol	L/g			J/mol	L/g	J/mol	
298	0.0772	74.98	35.26	0.9364	0.0003	220.1	40.83	0.8832
308	0.0393	114.2	23.15	0.8904	0.0005	254.3	31.62	0.8701
318	0.0351	143.4	18.44	0.8078	0.0005	291.6	31.62	0.8227

40 mg of ACROL was added to 100 ml of 100.8, 263.8, 284.0 and 361.1 mg/L of Hg(II) at constant pH 2.4, and 300 rpm. The suspensions were stirred for 75 min.

expected at higher temperature. This indicates that the adsorption process is exothermic. On the other hand, the  $K_L$  values were very slightly decreased with increasing temperature indicating that the adsorption of Hg(II) onto ACROL using Langmuir model cannot be evaluated using  $K_L$ . From  $R^2$  of Freundlich and Langmuir models, the Hg(II) adsorption process onto ACROL was followed Freundlich at low temperature, while it was followed Langmuir models with increasing temperature indicating the desorption of Hg(II) multilayer onto ACROL was taken place with increasing temperature of solution. The  $K_L$  was decreased with increasing temperature, indicating a bond strength between Hg(II) and ACROL decreased with increasing temperature and sorption is exothermic. Equation (5) is used to determine the constant separation factor,  $R_L$ :

$$R_L = \frac{1}{1 + bC_o} \quad (5)$$

The value of  $R_L$  indicates the isotherm shape to be unfavorable ( $R_L > 1$ ), favorable ( $0 < R_L < 1$ ) and irreversible ( $R_L = 1$ ). The  $R_L$  values were  $>1$ , which confirmed that the Hg(II) adsorption onto ACROL is unfavorable under these conditions using Langmuir isotherm for each temperature studied.

### 3.2.3. Temkin Isotherm

The Temkin isotherm is usually used for heterogeneous surface energy systems (non-uniform distribution of sorption heat) [15] [16].

$$q_e = \frac{RT}{b} \ln A + \frac{RT}{b} \ln C_e \quad (6)$$

where:  $B = \frac{RT}{b}$  constant related to heat of sorption (J/mol) obtained From the Temkin plot ( $q_e$  versus  $\ln C_e$ );  $A$  (slope) = Temkin isotherm equilibrium binding constant (L/g);  $b$  (intercept) = Temkin isotherm constant;  $R$  = universal gas constant ( $8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ )  $T$  = Temperature at 298, 308 and 318 K, shown in **Figure 3(c)** and listed in **Table 2**. The heat of Hg(II) adsorption ( $b$ ) is directly related to coverage of Hg(II) onto ACROL due to adsorbent-adsorbate interaction. It was decreased with increasing temperature from 35.26 to 18.44 J/mol, as listed in **Table 2**. This indicates that the heat of adsorption of Hg(II) onto the surface of ACROL decreases with increasing temperature from 298 to 318 K and the sorption is exothermic. Also, the  $b$  values is higher than 80 KJ/mol which indicating a chemical adsorption process and is a quite higher that reveals the strong ionic interaction between Hg(II) and ACROL [21].  $R^2$  is found to be the poorest fit of all experimental data.

### 3.2.4. Dubinin-Radushkevich (DRK) Isotherm

DKR isotherm is used to determine the apparent energy of Hg(II) adsorption onto ACROL and has the linear form as following equation:

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (7)$$

$$\varepsilon = RT \ln \left( 1 + \frac{1}{C_e} \right) \quad (8)$$

where:  $q_m$  is maximum sorption capacity (mg/g);  $\beta$  is a activity coefficient constant related to sorption energy;  $\varepsilon$  is Polanyi potential. The DKR parameters are calculated from the slop of the plot of  $\ln q_e$  versus  $\varepsilon^2$  gives  $\beta$  ( $\text{mol}^2/\text{J}^2$ ) and exp (intercept) gives  $q_m$  (mg/g) in **Figure 3(d)** and listed in **Table 2**. The  $\beta$  gives the mean free energy ( $E$ ) of sorption per molecule of the sorbate when it is transferred to the surface of the solid from infinity in the solution:

$$E = \frac{1}{\sqrt{2\beta}} \quad (8)$$

The value of  $E$  is  $>8$  that reveals the sorption process follows chemical sorption, as listed in **Table 2**.

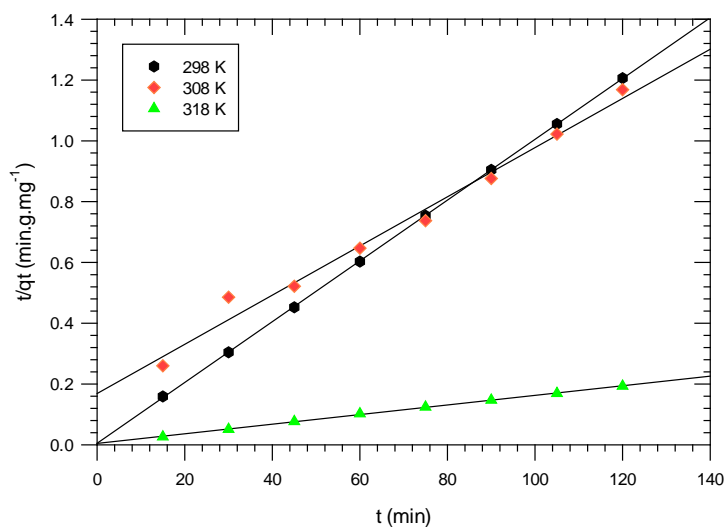
### 3.3. Adsorption Kinetics

Two kinetic models were used to fit experimental data, Lagergren pseudo-first order Equation (9) and pseudo-second-order Equation (10):

$$\log(q_e - q_t) = \log q_e - \left( \frac{K_1}{2.303} \right) t \quad (9)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_2^2} + \frac{t}{q_2} \quad (10)$$

where:  $q_e$  is the adsorption equilibrium capacity (mg/g),  $q_t$  is the amount of Hg(II) adsorbed (mg/g) at time  $t$ ,  $k_1$  is the rate constant of pseudo-first-order ( $\text{min}^{-1}$ ),  $q_2$  is the maximum adsorption capacity (mg/g) and  $k_2$  is the rate constant of pseudo-second-order ( $\text{g/mg min}$ ) [16]. These parameters were obtained from slope and intercept of pseudo-first-order and pseudo-second-order plots between  $\log(q_e - q_t)$  versus  $t$  and  $(t/q_t)$  versus  $t$  [20]. **Figure 4** shows pseudo-second-order plots for the Hg(II) adsorption onto ACROL surface and the kinetic parameters



**Figure 4.** Pseudo-second order kinetic plots for the adsorption of Hg(II) onto ACROL at various temperatures. ACROL (40 mg) was added to 100 ml of 300 mg/L Hg(II), pH2. 4 and at 298, 308 and 318 K.

for tow models are presented in **Table 3**. From the values of  $R^2$ , the experimental data follow pseudo-second-order kinetics comparatively better than pseudo-first-order. Also, Equation (10) is used to determine the best-fit for kinetic models by the square sum of errors (SSE) values:

$$SSE = \sum \frac{(q_{e(exp)} - q_{e(cal)})^2}{q_{e(exp)}} \quad (11)$$

The lowest value of SSE is the best model for the particular system. It is clear from SSE that pseudo-second-order kinetic has lower SSE value than pseudo-first-order kinetic.

### 3.4. Adsorption Thermodynamics

Equation (11) is used to calculate the thermodynamic equilibrium constant ( $K_c$ ) for Hg(II)-ACROL adsorption:

$$K_c = \frac{C_{Ae}}{C_{Se}} \quad (11)$$

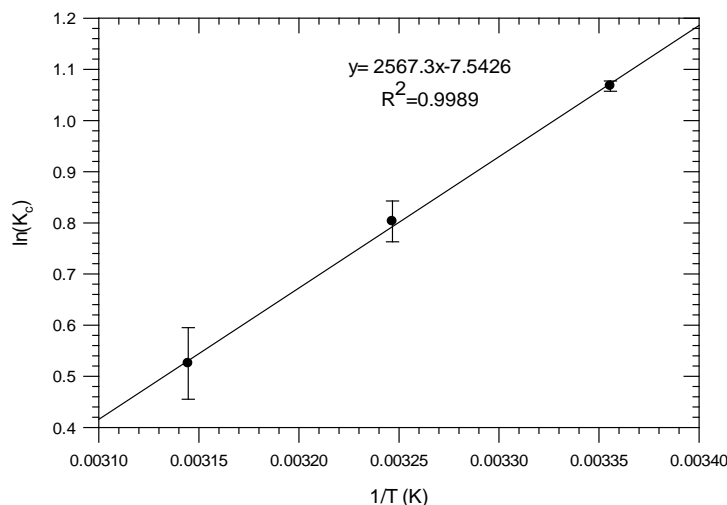
where:  $C_{Ae}$ : the equilibrium Hg(II) concentration adsorbed on ACROL at (mg/L) and  $C_{Se}$ : the equilibrium Hg(II) concentration in solution (mg/L). The change in standard free energy ( $\Delta G^\circ$ ), enthalpy ( $\Delta H^\circ$ ) and entropy ( $\Delta S^\circ$ ) of adsorption were calculated from the following equation:

$$\Delta G^\circ = -RT \ln K_c \quad (12)$$

Thermodynamic parameters were estimated from van't Hoff. A plot of  $\ln K_c$  versus  $1/T$  (see **Figure 5**) was used to determine  $\Delta H^\circ$  (= -slope.  $R$ ) and  $\Delta S^\circ$  (= intercept.  $R$ ) from slope and intercept by following equation:

$$\ln K_c = \frac{-\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (13)$$

The calculated thermodynamic parameters are listed in **Table 4**. The negative values of  $\Delta G^\circ$  at all temperature studied indicate the spontaneous nature of Hg(II) adsorption by ACROL. Also, the negative of  $\Delta G^\circ$  values, less than -15 kJ/mol, are connected with physical interactions between ACROL sites and Hg(II) ions. The experimental data show the adsorption capacities at equilibrium were decreased with an increase in temperature indicating that the adsorption process of Hg(II) onto ACROL follows exothermic path and the negative value of  $\Delta H^\circ$  confirms the exothermic nature of Hg(II)-ACROL adsorption process. These results confirm the previous findings which obtained in the adsorption isotherm study. Also, the negative values for both  $\Delta H^\circ$  and  $\Delta S^\circ$  suggest



**Figure 5.** Relationship between Gibbs free energy calculated from different isotherm models and adsorption process temperature (K). 40 mg of ACROL was added to varied Hg(II) concentrations from 100 to 400 mg/L Hg(II) at constant pH 2.4, 200 rpm and T = 298, 308 and 318 K.



**Table 3.** Kinetic parameters for Hg(II) adsorption by ACROL at various temperatures.

T (K)	Pseudo-First order					Pseudo-Second order kinetic			
	$q_{eexp}$ (mg/g)	$q_{ecal}$ (mg/g)	$k_1 \times 10^{-2}$ (min <sup>-1</sup> )	$R^2$	SSE	$q_{e2cal}$ (mg/g)	$k_2 \times 10^{-4}$ (g/mg·min)	$R^2$	SSE
298	294.1	5.554	3.777	0.7502	283.1	100.0	200.0	1.000	128.1
308	454.6	177.9	6.080	0.8543		125.0	3.710	0.9711	
318	588.2	13210	3.224	0.7536		625.0	5.565	0.9992	

ACROL (40 mg) was added to 100 ml of 300 mg/L of Hg(II), pH 2.4 and at 298, 308 and 318 K.

**Table 4.** Thermodynamic parameters for sorption of Hg(II) on ACROL.

Initial ion (mg/L)	$\Delta G^\circ$ (KJ/mol) 298 K	$\Delta G^\circ$ (KJ/mol) 308 K	$\Delta G^\circ$ (KJ/mol) 318 K	$\Delta H^\circ$ (J/mol)	$\Delta S^\circ$ (J/mol)
100.8	-0.4694	-0.8480	-0.6080	-308.8	-0.9072
263.8	-2.322	-2.142	-2.001		
284.0	-2.319	-2.0967	-1.594		
391.08	-2.645	-2.056	-1.389		

Optimum ACROL (40 mg) was added to 100 ml of varied Hg(II) concentrations from 400 mg/L Hg(II) at different temperature (298, 308 and 318 K) and at constant pH 2.4 and 200 rpm for 75 min.

that the adsorption is physical in nature with a weak forces of attraction and a decrease in randomness at ACROL/Hg(II) interface during the adsorption process [16].

#### 4. Conclusion

The findings in this research revealed that ACROL can be used as adsorbent for Hg(II) ions. The isotherm, kinetic and thermodynamic studies confirmed the experimental results. The equilibrium was the best described by Langmuir isotherm at high temperature, while the kinetic of the adsorption process was followed by pseudo-second-order model. The biosorption mechanism is chemisorption process. The negative values of  $\Delta H^\circ$  and  $\Delta S^\circ$  show that the Hg(II) adsorption process onto ACROL is exothermic in nature.

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