# Adsorption of acetic acid from aqueous solutions by using tea waste as low-cost biosorbent: Equilibrium, kinetic and thermodynamic studies

O. Khelifi[1,2], A. Djadallah[1], Kh. Khalili[1], F. Khanfoussi[1], H. Laksaci[3], M. Nacef[2], M.L. Chelaghmia[2]; A.M. Affoune[2]

[1]Département d'hydrocarbures et énergies renouvelables, Faculté des sciences et de la technologie, Université Ahmed Draia Adrar, Algeria.
[2]Laboratoire d'Analyses Industrielles et Génie des Matériaux (LAIGM), Département de génie des procédés, Faculté des sciences et de la technologie, Université 8 Mai 1945 Guelma, Algérie.
[3]Laboratoire d'études physico-chimiques des matériaux et application à l'environnement (LEPCMAE), USTHB, Algiers, Algeria.

*Corresponding author: khelifi.omar@univ-adrar.dz; Tel.: +213 6 62 52 43 09; Fax: +213 37 20 72 68

## ARTICLE INFO

## ABSTRACT/RESUME

*Abstract: This study aims to evaluate the preparation of a low-cost biosorbent from tea wastes were for use as a natural carrier for the removal of acetic acid from aqueous solutions. In order to clarify the adsorption process, batch experiments were carried out to study operating parameters effect on the adsorption process such as: contact time (5-60 min); initial concentration of acetic acid (0.01-0.1 mol $L^{-1}$) and temperature (15-35 °C). The experimental data were analyzed by Langmuir and Freundlich isotherm. The equilibrium is perfectly described by the Langmuir model ($R^2>0.99$) with a maximum monolayer adsorption capacity of 20.16 mg $g^{-1}$ and the calculated specific surface area is equal to 42.489 $m^2$ $g^{-1}$. Kinetic study shows that the pseudo-second order well fitted the kinetic data. Thermodynamic parameters indicate that the adsorption process is feasible, spontaneous and exothermic. The results of the present study show that tea waste can be advantageously used as a low cost biosorbent for the removal of an acid from aqueous solutions.*

## I. Introduction

Many environmental issues are growing day by day and are threatening the survival of mankind on earth. Water pollution is one of them. With rapid increase in population and growth of industrialization, quality of both surface and ground water is changing day by day [1]. Water pollution from industrial waste is a serious problem in many countries. This pollution may cause adverse effects on the environment and on human health [2]. Discharge of industrial wastewater has increased. There is the need to find cheap and efficient method for the treatment of industrial wastewater prior to disposal into natural water. Acetic acid is one of the most important chemicals in the chemical industry and belongs to the top 50 chemicals in amount produced [3]. On an industrial scale acetic acid is produced via the petrochemical route: Carbonylation of methanol, liquid-phase oxidation of hydrocarbons, and oxidation of acetaldehyde [4]. Acetic acid is used in the manufacture of vinyl acetate, acetic anhydride, acetate esters, monochloroacetic acid, and as a solvent in the production of dimethyl terephthalate and terephthalic acid [5]. The increasing demand for acetic acid has led to an increase in the acetic acid amount in the industrial wastewaters. Acids in wastewater are classified as priority pollutants and they must be recycled, diluted or treated chemically and then degraded biologically. European countries require neutralization or biological degradation of these waste streams [6]. Several processes have been employed for the removal of pollutants from water and wastewater including chemical precipitation, ion-exchange, membrane filtration, electrochemical treatment and adsorption [7].

However, most of these methods have several disadvantages such as high operating costs, low selectivity, incomplete removal, and production of large quantities of wastes. Therefore, physicochemical approach by using adsorption method has been proposed to solve this problem [8]. Adsorption is preferred for the removal of heavy metals from wastewater due to its simplicity, lower cost and high efficiency [7]. Activated carbon is the most widely used adsorbent because of its excellent adsorption efficiency. Commercially available activated carbons are very expensive and restrict its use in developing countries [1]. During the last two decades, many researchers have focused on the preparation of some biosorbents from natural waste of chicken eggshells [8], date stones [9], Stipa tenacissima L Alfa fibers [10], mango seed kernels [11], rices hulls [12], cocoa shells [13], Turkish fly ash [14], banana peel [15], bagasse fly ash [16], rice husk [17] and washingtonia seed [18]. These biosorbents from agricultural waste materials being economic and eco-friendly due to their unique chemical composition, availability in abundance, renewablity, low cost and more efficiency seem to be a viable option for heavy metal remediation [11].

According to the best of our knowledge, there are not any reports in the literature about the equilibrium, kinetic and thermodynamic studies of acetic acid adsorption onto low-cost biosorbent prepared from tea wastes. In the present study, batch adsorption of acetic acid from aqueous solutions was investigated by using low-cost biosorbent. Operating parameters effects such as acetic acid initial concentration, contact time and temperature have been investigated. Thereafter, equilibrium and kinetic data of the adsorption process were then analyzed to study the adsorption isotherms, kinetics and mechanism of acetic acid adsorption on the prepared biosorbent. Thermodynamic studies were also carried out to estimate the standard Gibbs free energy change ($\Delta G^0$), standard enthalpy change ($\Delta H^0$) and standard entropy change ($\Delta S^0$).

## II. Materials and methods

### II.1. Chemical reagents

All the chemical reagents used in this study were of analytical reagent grade including acetic acid (CH$_3$COOH, M=60.05 g mol$^{-1}$, 99.5 %, d=1.049) was used as adsorbate and sodium hydroxide (NaOH, ≥ 97%). Deionized distilled water was used for the experimental procedure. A stock solution of acetic acid was prepared (0.2 mol L$^{-1}$) and was diluted with distilled water to obtain desired concentration ranging from 0.01 to 0.1 mol L$^{-1}$.

### II.2. Biosorbent Preparation

The effects of experimental parameters: bios Tea wastes (TW) were collected and were washed with tap water for several times and afterwards with distilled water for three times. Then, they were dried at 105 °C for 24 h to achieve constant weight, and then were crushed and milled. Thereafter, they were sieved into a uniform size and kept in dessicator until adsorption experiments. Some properties of prepared biosorbent are showed in Table 1.

***Table 1.*** *Some physico-chemical properties of the prepared biosorbent.*

| Physico-chemical properties | Value |
|---|---|
| pH | 6.74 |
| Conductivity (µS cm$^{-1}$) | 465.2 |
| Humidity (%) | 2.68 |
| Ash content (%) | 10.52 |
| Particle size (µm) | 0.05-0.5 |

### II.3. Batch Adsorption Experiments

The effects of experimental parameters: initial acetic acid concentration (0.01-0.1 mol L$^{-1}$), contact time (5-60 min) and temperature (15-35 °C) on the adsorptive removal of acetic acid were studied in a batch mode using 25 mL of each acetic acid solution at a shaking speed of 150 rpm. After adsorption process, the biosorbent was separated by filtration. After filtration through Watman filter paper, the residual concentrations in the solution at equilibrium were determined by volumetrically titrating it with 0.1 N NaOH solutions and using of phenolphthalein like as colored indicator.

The removal rate R (%) and the equilibrium amount of acetic acid adsorbed per unit mass of biosorbent qe (mol g$^{-1}$) and instant adsorption capacity qt (mol g$^{-1}$) were, respectively, calculated as follow:

$$R\ (\%) = \frac{(C_0 - C_e)}{C_0} * 100 \tag{1}$$

$$q_e = \frac{(C_0 - C_e)}{m} * V \tag{2}$$

$$q_t = \frac{(C_0 - C_t)}{m} * V \tag{3}$$

Where $C_0$ (mol L$^{-1}$) is the initial acetic acid concentration; $C_e$ (mol L$^{-1}$) is the equilibrium acetic acid concentration; $C_t$ (mol L$^{-1}$) is the acetic acid concentration at instant $t$; $V$ (L) is the volume of the solution; and $m$ (g) is the mass of the biosorbent.

### II.4. Equilibrium study

For adsorption equilibrium, acetic acid solutions with concentrations varying from 0.01 to 0.1 mol L$^{-1}$ were mixed with 0.5 g biosorbent dose into 50

mL beaker under magnetic stirring at 300 rpm for 30 min at room temperature.

The Langmuir and Freundlich adsorption isotherms, often used to describe the adsorption of solutes from a liquid phase, were applied to our experimental results.

The Langmuir isotherm is one of the models that describe monolayer coverage. It assumes a homogenous adsorption surface with binding sites having equal energies. Linear form of the Langmuir isotherm equation can be expressed as follows:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_{max}} + \frac{1}{K_L \, Q_{max}} \qquad (4)$$

Where $K_L$ (L mol$^{-1}$) is the Langmuir constant, $Q_{max}$ (mol g$^{-1}$) represents the maximum adsorption capacity under the experimental conditions. $Q_{max}$ and $K_L$ are determined from the slope and intercept of plotting $C_e/q_e$ versus $C_e$, respectively.

The essential characteristic of the Langmuir isotherm can be expressed by a dimensionless factor ($R_L$), also called the separation factor. It is calculated according to the following equation:

$$R_L = \frac{1}{1 + K_L \, C_0} \qquad (5)$$

The $R_L$ value indicates the adsorption favorability, hence: the adsorption conditions are favorable when $0 < R_L < 1$, the conditions are unfavorable if $R_L > 1$, linear for $R_L = 1$ and irreversible when $R_L = 0$.

Freundlich isotherm is an empirical equation based on an exponential distribution of adsorption sites and energies. The linearized Freundlich isotherm equation is represented by the following equation:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \qquad (6)$$

Where $K_F$ (mol g$^{-1}$) (L g$^{-1}$)$^{1/n}$ is the Freundlich constant related to the bonding energy. $n$ is the heterogeneity factor and n is a measure of the deviation from linearity of adsorption. $K_F$ and $n$ are, respectively, determined from the intercept and slope of plotting ln $q_e$ versus ln $C_e$.

Temkin isotherm assumes that the adsorption is characterized by a uniform distribution of the binding energies up to some maximum binding energy; it has been applied in the following form:

$$q_e = \left(\frac{RT}{B}\right) \ln A + \left(\frac{RT}{B}\right) \ln C_e \qquad (7)$$

Where $A$ (L g$^{-1}$) and $B$ (J mol$^{-1}$) are the Temkin constants. T is the absolute temperature in Kelvin and R is the universal gas constant (8.314 J mol$^{-1}$ K$^{-1}$). $A$ and $B$ are determined from the intercept and slope of plotting $q_e$ versus ln $C_e$, respectively.

## II.5. Kinetic study

To investigate the controlling mechanism of biosorption process, the pseudo-first order and pseudo-second order were applied to model the kinetic study of the acetic acid adsorption onto prepared biosorbent.

The linear form of the pseudo- first order of Lagergren is giving by the following equation:

$$\ln(q_e - q_t) = \ln \, q_e + k_1 \, t \qquad (8)$$

Where $q_t$ is the adsorption capacity of acetic acid at time $t$, $k_1$ (min$^{-1}$) is the rate constant of the pseudo-first order. $k_1$ and $q_e$ were calculated from the slope and intercept by plotting $\ln(q_e - q_t)$ versus $t$, respectively.

The pseudo-second order is expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 \, q_e^2} + \frac{1}{q_e} t \qquad (9)$$

Where $k_2$ (g/mmol.min) is the rate constant of the pseudo-second order. $q_e$ and $k_2$ were determined from the slope and intercept by plotting $(t/q_t)$ versus t, respectively.

## II.6. Thermodynamic study

Thermodynamic parameters: standard Gibbs free energy change ($\Delta G^0$), standard enthalpy change ($\Delta H^0$) and standard entropy change ($\Delta S^0$) were calculated to evaluate the feasibility and the nature of the adsorption process.

The standard Gibbs free energy change ($\Delta G^0$) is determiner from the following equation:

$$\Delta G^0 = -RT \ln K_c \qquad (10)$$

Where $R$ is the universal gas constant (8.314 J mol$^{-1}$ K$^{-1}$), $T$ is the absolute temperature (K) and $K_c$ is the thermodynamic equilibrium constant. Relation between standard Gibbs free energy change ($\Delta G^0$), standard enthalpy change ($\Delta H^0$) and standard entropy change ($\Delta S^0$) is expressed by the following equation:

$$\ln K_c = \frac{-\Delta G^0}{RT} = \frac{-\Delta H^0}{RT} + \frac{\Delta S^0}{R} \qquad (11)$$

Where $\Delta H^0$ is the standard change in enthalpy (kJ mol$^{-1}$), $\Delta S^0$ is the standard change in entropy (kJ mol$^{-1}$ K$^{-1}$). The values of $\Delta H^0$ and $\Delta S^0$ were calculated from the slope and intercept of the plot of ln $K_c$ versus $1/T$, respectively.

## III. Results and discussion

### III.1. Effect of acetic acid initial concentration

The results of acetic acid initial concentration effect of on the adsorption capacity are show in Fig. 1. We note that this latter increases along with increase in initial acetic acid concentration. The equilibrium adsorption capacity from 0.1 to 0.875 mmol g$^{-1}$ could be attributed to the ratio of the available adsorption active sites to the initial number of acetic acid moles.
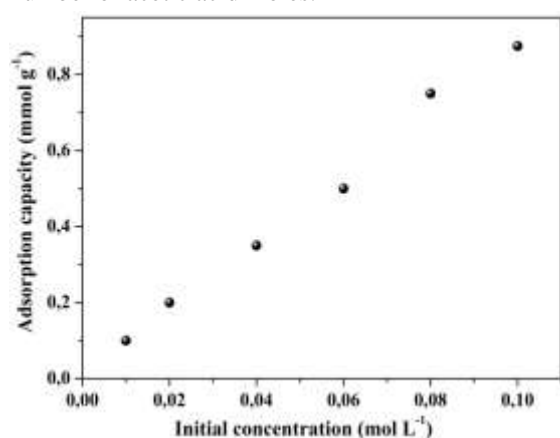


*Figure 1. Effect of initial acetic acid concentration on the adsorption capacity.*

### III.2. Effect of contact time

The effect of contact time on the acetic acid adsorption was determined and the corresponding variation of adsorption capacity is shown in Fig. 2. The obtained results indicate that the adsorption capacity of acetic acid by the prepared biosorbent increased with time up to 30 min and thereafter remains almost constant. The adsorption capacity of acetic acid increases from 0.24 mmol g$^{-1}$ at 5 min to 0.75 mmol g$^{-1}$ at 60 min. According to the results, the equilibrium reached at 30 min was taken as the optimal contact time for the subsequent experiments. The initial adsorption capacity could be due to the abundance of active binding sites on the biosorbent, and with gradual occupancy of these sites, the adsorption becomes less efficient in the later stage.
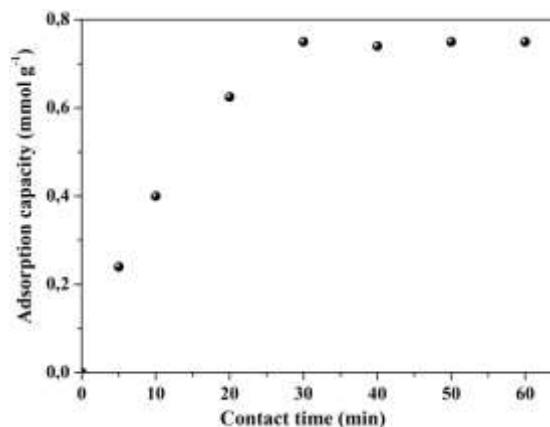


*Figure 2. Effect of contact time on the adsorption capacity of acetic acid by prepared biosorbent.*

### III.3. Effect of temperature

Fig. 3 shows that the equilibrium adsorption capacity of acetic acid molecules on prepared biosorbent increases with the increase in temperature. The equilibrium adsorption capacity increases from 0.36 to 0.65 mmol g$^{-1}$ in the temperature range 15-35 °C. The increase in equilibrium adsorption capacity of acetic acid could be due to the intraparticle diffusion rate of acetic acid into the pores of prepared biosorbent which is accelerated at higher temperatures. The results indicate that this adsorption is an endothermic process.
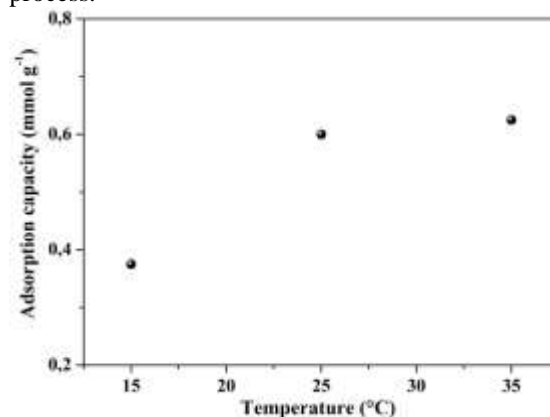


*Figure 3. Effect of temperature on the adsorption capacity of acetic acid by prepared biosorbent.*

### III.4. Adsorption isotherm study

The Langmuir, Freundlich and Temkin adsorption isotherms, often used to describe the adsorption of solutes from a liquid phase, were applied to our experimental results. The adsorption isotherms plot of Langmuir, Freundlich and Temkin models are graphically illustrated in Fig. 4 (a), (b) and (c).
The Langmuir, Freundlich and Temkin adsorption constants evaluated from the isotherms with the correlation coefficient ($R^2$) values for the both models are listed in Table 2.

As it can be seen from the results, Langmuir isotherm model has the highest value of regression coefficient compared to the Freundlich and Temkin isotherm, which indicate that this model describes very well the adsorption process and suggests that the adsorption on the surface of prepared biosorbent was a monolayer adsorption. According to the Langmuir equation, the maximum uptake capacity for acetic acid was 0.336 mmol $g^{-1}$, which is equivalent to 20.16 mg $g^{-1}$. The value of $R_L$ indicates that the adsorption process is favorable.
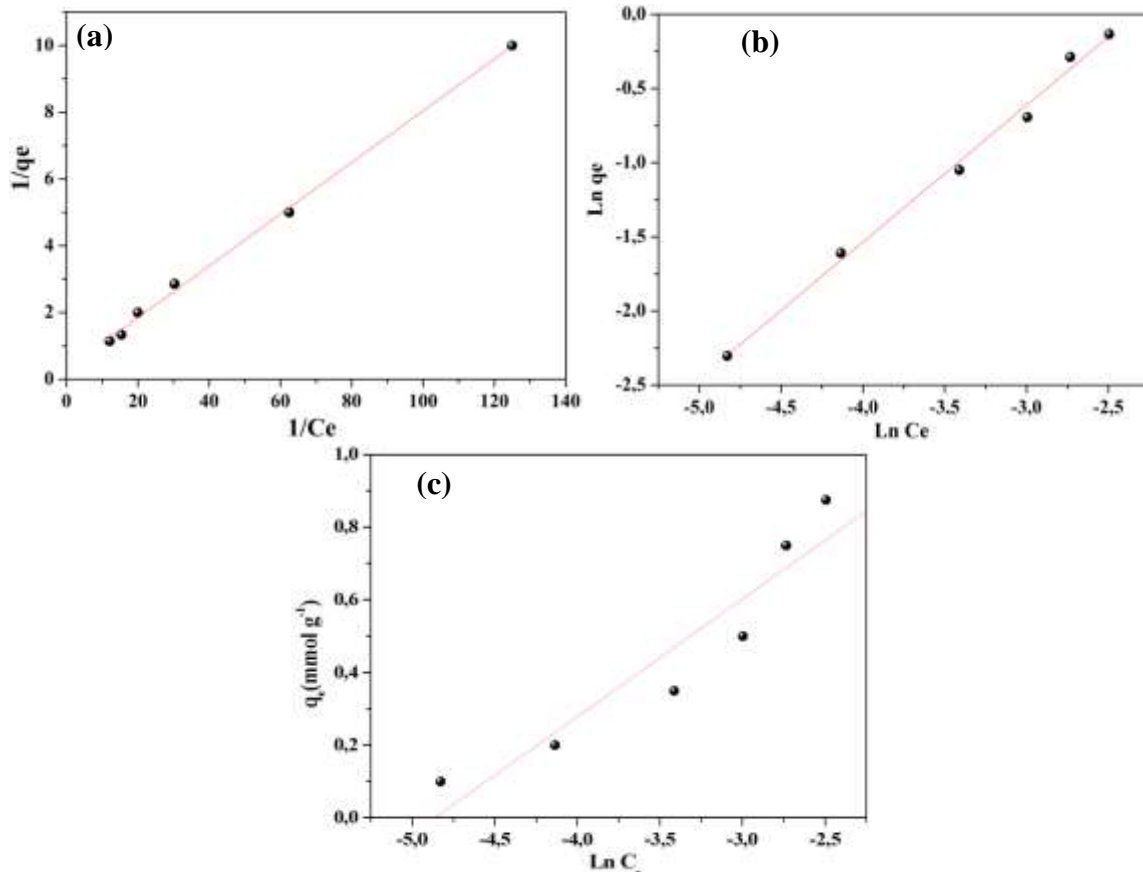


*Figure 4. Plot of Langmuir model (a), Freundlich model (b) and Temkin model (c).*

*Table 2. Isotherms parameters for acetic acid adsorption by TW biosorbent.*

| Langmuir model | | | | Freundlich model | | | Temkin model | | |
|---|---|---|---|---|---|---|---|---|---|
| $Q_{max}$ (mol $g^{-1}$) | $K_L$ (L mg$^{-1}$) | $R_L$ | $R^2$ | $K_F$ (mol $g^{-1}$) (L $g^{-1}$)$^{1/n}$ | $n$ | $R^2$ | $A$ (L $g^{-1}$) | $B$ (J mol$^{-1}$) | $R^2$ |
| 0. 3363 | 2.2980 | 0.0043 | 0.9929 | 6.0099 | 1.0846 | 0.9829 | 4.8643 | 7027.003 | 0.9451 |

The maximum adsorption capacitiy ($Q_{max}$) of acetic acid found in the present study was compared with those of other carbonaceous adsorbent reported in the literature and are presented in Table 3.

*Table 3. Maximum adsorption capacities for acetic acid adsorption by some carbonaceous adsorbents.*

| Biosorbents | $Q_{max}$ (mg $g^{-1}$) | References |
|---|---|---|
| Brassica nigra | 0.96 | [5] |
| Polyalthia longifolia | 48.07 | [1] |
| Activated carbon | 132 | [19] |
| Triplochiton Scléroxylon | 76.8 | [20] |
| Terminalia    Superba | 12.8 | [20] |
| Animal AC | 55.2 | [21] |
| Tea wastes leaves | 20.16 | Our study |

From Table 3, the value of monolayer maximum adsorption capacity of tea wastes leaves is lower than that of activated carbon. On the other hand, this value is higher or considerably greater than other reported biosorbents. The comparison shows that the tea wastes leaves used in this research shows a favorable adsorption capacity for acetic acid if compared to the other biosorbents. The different performances of acetic acid uptake could be explained in term of the following factors: (i) the heterogeneous nature and composition of biosorbents; (ii) the textural and surface properties of each biosorbent such as structure, functional groups and their surface area.

The monolayer maximum adsorption capacity ($Q_{max}$) calculated from the Langmuir isotherm model was used to calculate the specific surface area ($S_{SA}$) as follows [6]:

$$S_{SA} = A_{ads} \cdot N_A \cdot Q_{max} \qquad (12)$$

Where: $A_{ads}$ is the cross-sectional area of the adsorbate molecule; $N_A$ is Avogadro's number ($6.023 \times 10^{23}$). Assuming that the acid molecules are adsorbed vertically, with the aliphatic chain up and the carboxyl group down attached to the adsorbent surface, the cross-sectional area of a straight-chain acid was taken to be about $21 \times 10^{-20}$ m$^2$ [22].
From the monolayer maximum adsorption capacity, the calculated specific surface area is equal to 42.49 m$^2$ g$^{-1}$.

### III.5. kinetic study

The experimental results pertaining to the adsorption kinetics are compared to both models, allowing determining the parameters of these models (Table 4). Analysis of the correlation coefficients shows that the pseudo-second order gives better fits if compared to the pseudo-first order.

**Table 4.** *Kinetic parameters of models for acetic acid biosorption.*

| Kinetic model | Parameters | Values |
|---|---|---|
| **Pseudo-first order** | $q_e$ (cal.) (mmol g$^{-1}$) | 0.072 |
| | $k_1$ (min$^{-1}$) | 1.451 |
| | $R^2$ | 0.955 |
| **Pseudo-second order** | $q_e$ (cal.) (mmol g$^{-1}$) | 1.806 |
| | $k_2$ (g mmol$^{-1}$ min$^{-1}$) | 0.017 |
| | $R^2$ | 0.999 |

Analysis of the correlation coefficients shows that the pseudo-second order gave better fits compared to the pseudo-first order. These findings are in agreement with the majority of reported works. It can be concluded that the rate-controlling step was

the sharing or exchange of electrons between the acetic acid and the prepared biosorbent. The value of rate constant ($k_2$) is small, which indicates that the kinetic of the adsorption process is slow. It could be attributed to the nature and properties of the biosorbent.

### III.6. Thermodynamic study

The value of $\Delta H^0$ and $\Delta S^0$ were calculated from the slope and intercept by plotting ln $K_c$ versus $1/T$, respectively (Fig. 5).
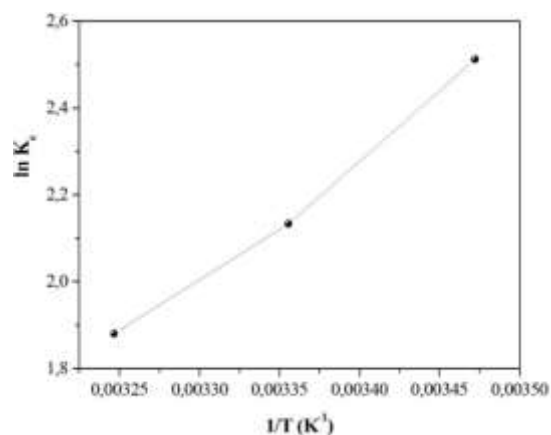


**Figure 5.** *Thermodynamic plot for acetic acid adsorption by prepared biosorbent.*

The values of obtained thermodynamic parameters are presented in Table 5. The negative values of $\Delta G^0$ indicate that the mechanism of acetic acid adsorption onto prepared biosorbent is feasible and show the spontaneous nature of the process. The negative value of $\Delta H^0$ confirms the exothermic nature of acetic acid adsorption onto the prepared biosorbent and the positive value of $\Delta S^0$ reflects the affinity of prepared biosorbent for acetic acid and shows the increasing randomness at the solid-liquid interface during the adsorption process.

**Table 5.** *Thermodynamic parameters of acetic acid adsorption on prepared biosorbent.*

| T (K) | $\Delta G^0$ (kJ mol$^{-1}$) | $\Delta H^0$ (kJ mol$^{-1}$) | $\Delta S^0$ (kJ mol$^{-1}$ K$^{-1}$) |
|---|---|---|---|
| 288 | -6.0155 | | |
| 298 | -5.1519 | - 23.3224 | 0.06023 |
| 308 | -4.8166 | | |

### IV. Conclusion

Batch adsorption studies for the removal of acetic acid from aqueous solutions have been carried out using tea wastes leaves as low-cost biosorbent. Batch studies demonstrated that under laboratory conditions, a contact time of 30 min and temperature of 25 °C for achieving an adsorption capacity more than 35 % of acetic acid from

synthetic solution containing 0.1 mol $L^{-1}$ of initial concentration. Equilibrium isotherm data were in good agreement with Langmuir than Freundlich isotherm model. The maximum monolayer adsorption capacity, $Q_{max}$, of acetic acid calculated from Langmuir model was found to be 20.16 mg $g^{-1}$ at 25 °C and . Adsorption kinetics studies revealed that the pseudo-second order kinetic model better described the adsorption data. The thermodynamic parameters $\Delta G^0$, $\Delta H^0$ and $\Delta S^0$ showed a favored, spontaneous, endothermic and physicochemical adsorption process. Based on the results obtained from this study, it can be concluded that the tea wastes leaves is an easily, locally available and low-cost biosorbent; it may be treated as a cost effective and a potential biosorbent for the removal of acetic acid from aqueous solutions.

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