Algerian Journal of Environmental Science and Technology
September edition. Vol.9. Nº3. (2023)
ISSN : 2437-1114
www.aljest.org

Equilibrium, kinetic and thermodynamic studies of the biosorption of copper onto Luffa Cylindrica Cords

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ARTICLE INFO

Article History :

Received **: *21/02/2021***
Accepted **: *13/07/2022***

Key Words:

Luffa cylindrica cords; Copper; Isotherm; Thermodynamics; Kinetics.

ABSTRACT/RESUME

Abstract: *This study explores the possibility of using Luffa Cylindrica Cords to remove Copper from aqueous effluents. The removal of copper was investigated in both batch and continuous processes. Batch mode adsorption studies were performed by varying parameters such as contact time, pH, initial copper concentration and temperature. The different adsorption models Langmuir, Freundlich, Elivoch and Temkin were used for the mathematical description of the adsorption equilibrium, and it was found that the very well - equipped experimental data for the Langmuir model (R^2=0.961), the pseudo - first - order and pseudo - second - order kinetic models were applied to the experimental data. The experimental data fitted very well the pseudo-second-order kinetic model (R^2= 0.965) and also followed the model of intra particle diffusion for concentrations between 5 and 50 mg/L), whereas diffusion is not the only rate - control step. Finally the thermodynamic parameters for the adsorption of copper onto cords were also determined. The negative sign of ΔS° shows the decreased randomness at the solid - solution interface during adsorption, and the negative sign of ΔH° indicates that the adsorption process is exothermic. The negative sign of ΔG° confirms the spontaneous nature of the adsorption process, physical reaction.*

I. Introduction

Heavy metals are placed among the most important environmental pollutants that have changed into a global concern due to their toxicity and carcinogenetic properties. These metals, even in trace amounts, accumulate in the body of creatures and their food cycle [1]. Examples of heavy metals include copper, lead, mercury and zinc [2]. Generally, heavy metals have densities above 5g/cm3. As the heavy metals cannot be degraded or destroyed, they are persistent in all parts of the environment. Human activity affects the natural geological and biological redistribution of heavy metals through pollution of the air, water [3]. Different methods and processes such as coagulation and flocculation, biological treatment, adsorption, membrane processes, etc could be used to remove dyes and organic matters from wastewaters [4]. Biosorption process in comparison with other methods is a better procedure in terms of cost, simplicity in design and operation, availability, effectiveness and lack of sensitivity to toxic substances [5]. Biosorption is an alternative technique for removing heavy metals from aqueous solutions, based on the property of a certain class of biomass to accumulate this kind of pollutant through mechanisms, such as physical adsorption, complexing and ion exchange. The most commonly used natural biosorbents are totally renewable, their cost is low and their use and handling involve no additional risks; they may also be specifically selective for different pollutants and are generally disposed of by incineration [6]. Many materials, such as waste red mud and fly ash, tea-industry waste, calcite and hydroxyapatite, waste materials as refuse concrete, waste paper and charcoal, and natural indigenous rocks as andesite, granite and olive stone waste have been tested for copper removal from water systems by various researchers [7]. Luffa cylindrica cords, mainly consists of cellulose, hemicelluloses and lignin; of composition (60%, 22% and 10.6 % by weight, respectively) [8]. Because of its unique structure, Luffa cylindrica has been used as an efficient adsorbent or as a carrier for immobilization of some microalgal cells for the removal of water pollutants [9]. For the present study, a batch - contact - time method was used, and the equilibrium of Cu (II) adsorption onto Luffa cylindrica cords were investigated with the attempts to fit the data to Langmuir, Freundlich, Temkin and Elovich equations. The uptake of Cu (II) on Luffa cylindrica cords were examined as a function of temperature, initial copper concentration, adsorbent dose, particles size, pH, and contact time. The biosorption kinetic data of the biomaterial were tested by the pseudo - first - order and the second - order kinetic models. The thermodynamics of the biosorption was also evaluated.

II. Materials and methods

II.1. Preparation of biosorbent

Luffa cylindrica is an annual herbaceous plant of the family Cucurbitaceae grows north of Algiers (W Chlef). It gives a fruit of cylindrical shape slightly angular, straight and curved, of very variable size. Its sponge-type structure, formed of fibrous cords consolidated between them. Luffa cylindrica cords (LCC) were washed with boiling distilled water for 30 min. These cords were then placed in a solution of NaOH (12%) for 15 min and washed again with tap water. They are whitened with bleach 12% for 3 h at ambient temperature after which the cords are rinsed with distilled water several times. The cords of LCC were oven dried at 378 K for 120 min, then crushed and sifted. Samples are ready for adsorption.

II.2. Preparation of solution

Stock solution of Cu (II) ions with a concentration of 1000 mg/L was prepared by dissolving $CuSO_4$ (Merck, Germany) in distilled water. The desired Cu (II) concentrations were prepared from the stock solution by making fresh dilutions for each sorption experiment and the experimental solutions were obtained by diluting the stock solution to the required concentrations (5 to 50 mg/L).

II.3. Adsorption experiments

The method used for the establishment of the adsorption isotherm method is to put in a water bath fitted with a stirrer, a series of flasks with which we introduce a volume solution of the adsorbate Cu (II)) known concentration (5, 25 and 50 mg/L) and a mass of the LCC at different temperatures with pH 8.5. For a given contact time corresponding to equilibrium, we recover the filtrate solution for passing the UV-visible spectrophotometer.

For these tests, 1 g of LCC were added to 200 mL Cu (II) solutions with different initial concentrations. The mixture was agitated on an electromagnetic stirrer at 350 rpm. At predetermined time intervals (0 - 160 min).

The copper concentration analysis of filtrate solution was immediately measured with UV-visible spectrophotometer ((Unicam 8625, France)). The adsorption amount at time t, qt (mg/g) was calculated by the following equation:

$$q_t = \frac{\left(C_0 - C_t\right) V}{m} \qquad (1)$$

Where q_t is the amount of Cu(II) taken up by the adsorbent (mg/g); C_t (mg/L) is the concentration of Cu (II) solution at time, t (min), C_0 (mg/L) is the initial concentration of Cu(II). V and m are the volume of the solution (L) and the mass of the adsorbent (g), respectively.

III. Results and discussion

III.1. Effect of pH on the removal of copper

Many studies suggest that pH is an important factor in the biosorption process **[10]**, variations in pH could change the characteristics and availability of metal ions in solution as well as the chemical status of the functional groups responsible for biosorption. Some experiments were therefore performed at 343 K with 50 mg/L solutions to study the Cu (II) adsorption on LCC as a function of solution pH.

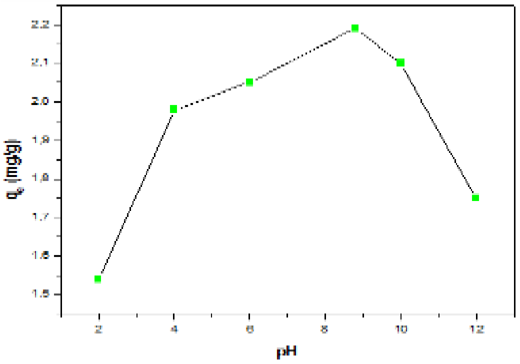

Figure 1. *Effect of pH on the adsorption of Cu (II) onto LCC :m =1 g, V=200ml, T*=343 K.

III.2. Effect of adsorbent dose

The effect of the adsorbent dose on the amount adsorbed of Cu (II) was studied at 343K and *p*H of 8.5 by varying the adsorbent amounts from 0.125 to 7.5 g/L. For all these runs, initial concentration of copper was fixed at 50 mg/L. The analysis showed that the adsorption of Cu (II) increased as the adsorbent dosage increased from 0.125 to 7.5 g/L due to the limited availability of the adsorbing species number for a relatively larger number of surface sites on the adsorbent.

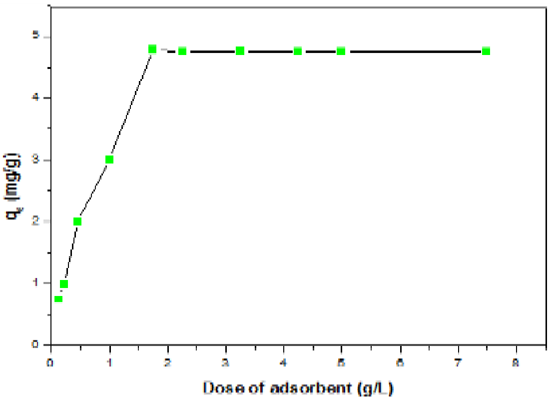

Figure 2. *Effect of adsorbent dosage on Cu (II)/LCC: V=200 ml, T=*343K.

III.3. Effect of contact time

The adsorption rate of inorganic pollutants by the adsorbents is relatively rapid and the adsorption equilibrium reaches in 60 min. The figure 3 shows the effect of contact time on the adsorption of the Cu (II) on LCC. The adsorption equilibrium of Cu (II) was achieved after 60 min (qe = 9.98mg/g) and no remarkable changes were observed for the longer contact time, which also indicated that the adsorption rate of Cu (II) on LCC were much faster than of some other natural adsorbents for .heavy metal [11].

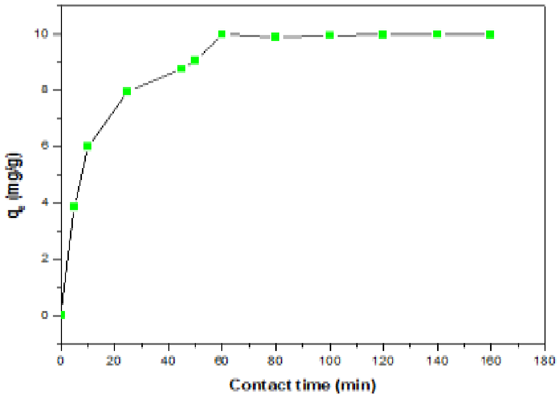

Figure 3. *Effect of Contact time on Cu(II) removal by LCC at given conditions: pH= 8.8, C_0 = 50 mg/L, m = 1 g, V=200 ml at 343 K.*

III.4. Effect of initial Cu (II) concentration on temperature-dependent adsorption

The initial Cu (II) concentration provides an important driving force to overcome all mass transfer limitations of Cu (II) between the aqueous and solid phases. Therefore, a higher initial Cu (II) concentration will enhance the adsorption process. Figure 4 show the effect of initial Cu (II) concentration on the equilibrium adsorption capacity (q_e) of Cu (II) on LCC at different temperatures. I was clear to see that the q_e values increase with the increase in the initial Cu (II) concentrations or the solution temperatures. The maximum equilibrium q_e values were determined as 2.75, 6.75 and 9.98 mg/g for 50mg/L initial Cu (II) concentrations at 296, 313 and 343 K, respectively.

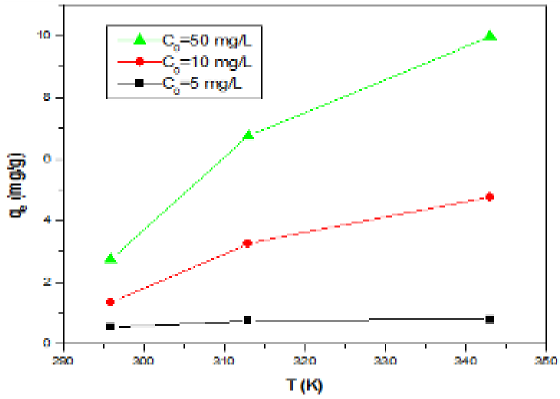

Figure 4. *Effect of temperature on the amount adsorbed by the Cu (II) / LCC:* pH= 8.8, m = 1 g, d_p(µm) ≤ 150 and V = 200ml.

III.5. Adsorption isotherms

Adsorption isotherm models are widely used to describe the adsorption process and investigate mechanisms of adsorption. Adsorption isotherms were calculated based on the well-known Langmuir, Freundlich models. The Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites, using uniform strategies of adsorption [12]. The linear form of the Langmuir isotherm equation is given as:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} \qquad (2)$$

where Ce is the equilibrium concentration of the biosorbate (mg/L), qe (mg/g), K_L is the Langmuir adsorption constant (L/mg), and qm is the theoretical maximum adsorption capacity (mg/g).
The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter **[13]**, RL, defined as:

$$R_L = \frac{1}{1 + K_L C_0} \qquad (3)$$

The value of R_L indicates the shape of isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$) [14].

The Freundlich isotherm, on the other hand, assumes heterogeneous surface energies, in which the energy term in the Langmuir equation varies as a function of the surface coverage [11]. The well-known logarithmic form of the Freundlich isotherm [15] is given by the following equation:

$$Log(q_e) = Log(k_F) + \frac{1}{n_F} Log(C_e) \qquad (4)$$

where K_F (mg/g (L/mg)$^{1/n}$) and 1/n are the adsorption constants of Freundlich model generally related to the strength of interaction between adsorbate and sorbent.

Temkin's model rests on the assumption that during the adsorption gas phase, the heat of adsorption due to interactions with the adsorbate decreases linearly with the recovery rate θ. From qe plotted as a function of Ln Ce, B_T, and K_T values can be determined [16] :

$$q_e = \frac{RT}{B_T} Ln\left(K_T . C_e\right) \qquad (5)$$

Where R is the perfect gas constant (8.314 J/(mol K)), T is the absolute temperature (K), B_T is the variation in energy of adsorption (J/mol), and K_T is the constant of Temkin (L/mg).

Elovich equation is also used successfully to describe second order kinetic assuming that the actual solid surfaces are energetically heterogeneous, but the equation does not propose any definite mechanism for adsorbate-adsorbent [17].

$$q_t = \frac{1}{\beta_{El}} Ln(\alpha_{El}\, \beta_{El}) + \frac{1}{\beta_{El}} Ln(t) \qquad (6)$$

Where α_{El} is the initial adsorption rate (mg/g min), and the parameter be is related to the extent of surface coverage and activation energy for chemisorption (g/mg).

Table 1 summarizes the constants of the Langmuir, Freundlich, Elovich and Temkin isotherms obtained from the slope and the interception of the plots of each isotherm at different temperatures. The values of R^2 exceed 0.9 for the three models of Langmuir, Freundlich, Temkin and Elovich, suggesting that the four models with near experimental results.

Table 1. *Parameters of different adsorption models for Cu (II) / LCC*

Isotherm model	**Parameter**	**Temperature (K)**		
		296	**313**	**343**
Freundlich	K_F (mg$^{1+1/n}$/g.L$^{1/n}$)	7.685	5.901	5.82
	$1/n_F$	1.75	2.907	3.061
	R^2	0.909	0.985	0.978
Langmuir	q_m (mg/g)	0.580	1.219	3.077
	K_L(L/g)	0.158	0.125	0.099
	R^2	0.978	0.999	0.907
	R_L	0.112	0.138	0.168
Temkin	K_T (L/g)	3.419	7.844	60.779
	B_T (J/mol)	6342.63	3306.58	2912.8
	R^2	0.808	0.977	0.876
Elovich	α_{El} (mg/min.g)	0.308	1.113	2.982
	β_{El} ([g/mg)	1.115	0.620	0.461
	R^2	0.966	0.917	0.980

The adsorption isotherm constants of Cu (II) adsorption process on the LCC are listed in Table 1. From Table 1, the values of 1/n were found to be great than 1. The values of R^2 for Freundlich model (0.909, 0.985, and 0.978 at 296, 313, and 343 K, respectively) were high, indicating that this model can be used to characterize the equilibrium adsorption. However, Freundlich model was not the best model in this study. Langmuir models were fitting well with the isotherm data at various temperatures with highest R^2 values, compared to Freundlich model. The Langmuir fit is consiste with strong monolayer sorption onto specific sites.

III.6. Adsorption Kinetics

The rate constant for surface adsorption of Cu (II) ions onto LCC are also studied under the light of the pseudo-first-order rate expression of Lagergren model **[18]**:

$$Log(q_e - q_t) = Log q_e - \frac{k_1}{2.303} t \qquad (7)$$

Where q_e and q_t are the amounts adsorbed at equilibrium and at time, t (min), and k_1 is the rate constant of the pseudo-first-order adsorption (1/min).
The pseudo-second-order kinetic model (8) can be represented in the following form **[19]:**

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (8)$$

Where k_2 is the rate constant of the pseudo-second order adsorption (g/mg min).
The kinetics of adsorption of the adsorbate on the adsorbent was verified at different initial concentrations (5, 25, and 50 mg/L).

Most adsorption reactions take place through multi-step mechanism comprising (i) external film diffusion, (ii) intraparticle diffusion, and (iii) interaction between adsorbate and active site. Since the first step is excluded by shaking the solution, the rate-determining step is one of the other two steps. Weber and Morris **[20]** described the intraparticle uptake of the adsorption process to be proportional to the half power of time:

$$q_t = K_{dif}\, t^{1/2} \qquad (9)$$

With: k_{dif}: rate constant intraparticle diffusion (mg/g.min$^{1/2}$).

The results of Table 2 show that the second - order rate constant k_2 decreased with increasing concentrations. The correlation coefficients for the second-order kinetic model were between 0.919 and 0.998. Moreover, the experimental q_e (exp) values agree well with the calculated ones. On the other hand, the correlation coefficients for the pseudo-first-order kinetics (0.910 $<R_2$ <0.994) were lower than that from pseudo second-order one. These results indicate that the adsorption system of copper obeyed a pseudo-second-order kinetic model. The pseudo - first - order and pseudo - second - order kinetic models could not identify the diffusion mechanism.
We observe that the diffusion rate constants increase in the same direction as the initial concentration of Cu (II) (Table 2). Indeed for initial concentrations between 5 and 50 mg/L, the values of K_{dif} vary from 0.103 to 1.404 mg/(g min$^{1/2}$) for the Cu (II) / LCC system.

Table 2. *Application Results of adsorption models for the system (Cu (II) / LCC).*

Kinetic model	Cu (II) (mg/L)	5	25	50
Pseudo - first - order kinetic model	$q_{e,\,exp}$ (mg/g)	0.79	4.75	9.98
	$q_{e,\,cal}$ (mg/g)	0.17	9.2	10.34
	K_1 (min^{-1})	0.025	0.037	0.046
	R^2	0.994	0.910	0.950
Pseudo - second - order kinetic model	$q_{e,\,cal}$ (mg/g)	1.293	6.135	1.124
	K_2 (g/mg.min)	0.016	0.007	0.077
	R^2	0.919	0.979	0.998
Intraparticle diffusion	K_{dif} (mg/g.min)	0.103	0.616	1.404
	R^2	0.868	0.949	0.715

For the first - order kinetic model, the obtained R^2 values were relatively low, ranging from 0.910 to 0.950, and the calculated $q_{e(cal)}$ were much larger than the experimental $q_{e(exp)}$. The pseudo - second - order rate constant k_2 and the corresponding linear regression correlation coefficient values, R^2, are given in Table 2. The applicability of the kinetic model is compared by judging the correlation coefficients R^2 and the agreement between the calculated and the experimental q_e values. In a view of these both considerations, we may conclude that the pseudo second-order mechanism is predominant R^2 (0.965).

III.7. Thermodynamic studies

The original concepts of thermodynamics assumed that in an isolated system, where energy cannot be gained or lost, the entropy change is the driving force. In environmental engineering practice, both energy and entropy factors must be considered in order to determine what processes will occur spontaneously. The Gibbs free energy change, ΔG°, is the fundamental criterion of spontaneity. Reactions occur spontaneously at a given temperature if $\Delta G°$ is a negative quantity. The value of $\Delta G°$ can be determined from the following equation:

$$\Delta G^0 = -R\,T\,Ln(k_L) \qquad (15)$$

Where K_L is the adsorption equilibrium constant, R is the gas constant (8.314 J/mol K), and T is the absolute temperature.

A convenient form of the Van't Hoff equation then relates K_L to the standard enthalpy and entropy changes of adsorption, ΔH^o and ΔS^o, respectively **[21]**:

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \qquad (16)$$

(16) can be written as:

$$Ln\,(k_L) = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{R}\cdot\left(\frac{1}{T}\right) = -\frac{\Delta G^0}{R}\cdot\frac{1}{T} \qquad (17)$$

The values of $\Delta G°$, $\Delta H°$, and $\Delta S°$ for the biosorption of Cu (II) onto LCC at different temperatures (296, 313, and 343 K) are given in Table 3.

Table 3. *Thermodynamic parameters for the adsorption of Cu (II) onto LCC different temperatures*

T (K)	**ΔG^0 (kJ/mol)**	**ΔH^0 (kJ/mol)**	**ΔS^0 (J/mol.K)**	**R^2**
296	- 0.37	-2.442	-0.007	0.823
313	-251			
343	-0.041			

The negative (ΔG°) values of Gibb's free energy represent that the adsorption undergoes thermodynamically feasible reaction and also spontaneous in nature [22]. The negative ΔH° value (-2.442 KJ / mol) shows that the nature of adsorption is exothermic. The negative ΔS° value (- 0.007 KJ / mol.K) suggests a decrease in the randomness at the Solid/solution interface during adsorption of copper on to LCC **[23]**. The ΔG° values obtained in this study for the copper ion are < - 10 kJ/mol, which indicates that physical adsorption was the predominant mechanism in the sorption process **[24]**.

IV. Conclusions

Equilibrium, kinetic, and thermodynamic studies were made for the adsorption of Cu (II) from aqueous solution onto Luffa cylindrica cords powder at pH 8.5 Equilibrium data were fitted to Langmuir, Freundlich, Elivoch and Temkin isotherms, and the equilibrium data were best described by the Langmuir isotherm model (R^2 = 0.961), with a maximum monolayer adsorption capacity of 9.98 mg/g at 60 min. The adsorption kinetics is best described by the pseudo - second - order (R^2 = 0.965).

The thermodynamic parameters for the adsorption of Cu (II) onto cords were also determined. The negative sign of ΔS° shows the decreased randomness at the solid - solution interface during adsorption, and the negative sign of ΔH° indicates that the adsorption process is exothermic. The negative sign of ΔG° confirms the spontaneous nature of the adsorption process, physical reaction.

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Please cite this Article as:

Laidani Y., Henini G., Hanini S., Fekaouni A., Equilibrium, kinetic and thermodynamic studies of the biosorption of copper onto Luffa Cylindrica Cords. ***Algerian J. Env. Sc. Technology*, 9:3 (2023) 3265-3271**