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The use of eucalyptus leaves as adsorbent for copper ion removal

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ABSTRACT

The possibility of using eucalyptus leaves as a locally available adsorbent for the removal of copper ions from aqueous solutions was studied. A series of batch experiments were carried out at different operating conditions. The influence of initial concentration, adsorbent concentration, contact time, temperature, pH, and presence of other ions such as Na^+ and K^+ on the adsorption process were investigated. Adsorptions of metal ions were pH dependent and the optimum pH for the removal of Cu^{2+} was found to be 6.5. The adsorption capacity of copper from aqueous solution by eucalyptus leaves was increased by increasing the contact time. At initial concentration of 100 mg/l, the maximum adsorption capacity was 1.16 mg of copper ion per gram of adsorbent. Isotherm studies showed that the data were best fitted to Langmuir isotherm model. The monolayer adsorption capacity was 1.92 mg/g. The values of R_L and "n" have indicated the favorability of copper adsorption onto eucalyptus leaves. The results reveal that the adsorption process was spontaneous and exothermic process. Physical activation of eucalyptus leaves influenced the copper removal positively. Addition of salts to the metal solution affected the copper uptake negatively.

Keywords: Eucalyptus leaves; Adsorption; Activation; Isotherm models

1. Introduction

In recent years, the presence and removal of toxic and polluting heavy metals in wastewaters from industrial effluents, water supplies, and mine waters has received much attention. It has been established that a serious of health hazards may result from dissolved heavy metals escaping into the environment. They tend to accumulate in living tissues throughout the food chain, and consequently multiplying its danger [1]. Thus, it is necessary to eliminate heavy metals from water and wastewater to protect public health. Different methods have been used to remove heavy metals from wastewater such as chemical precipitation, ion exchange, membrane separation, and adsorption. Among them, adsorption was found to be the most commonly used method for eliminating these contaminants, especially at low concentrations. Different adsorbents are developed from available natural materials and used for heavy metals removal such as activated carbon [2,3], pine bark [4], charcoal [5] banana peel [6], tar sands [7], modified rice husk [8,9], zeolites [10], and moss peat [11]. Many low-cost agricultural by products, such as almond shells, olive stones, and peach stones were used for the removal of Zn^{2+}, Cd^{2+}, and Cu^{2+} [12]. Several studies have shown that low-cost natural adsorbents are efficient for copper ions removal [13–17]. In this study, the adsorption of copper ions using eucalyptus leaves was investigated. The amount of copper uptake by eucalyptus leaves was investigated at different conditions, such as contact time, pH, temperature, adsorbate concentration, sorbent concentrations, and presence of ions such as Na^+ and K^+ on activated and nonactivated eucalyptus leaves.

2. Material and methods

2.1. Materials

Stock solution of copper (1,000 mg/l) was prepared in 1 L of deionized water by dissolving 3.929 g of copper sulfate ($CuSO_4.5H_2O$) and used for all experiments with required dilution. The eucalyptus leaves were collected and used for whole experiments.

2.2. Methods

2.2.1. Preparation of adsorbent

Eucalyptus leaves were washed, sun dried for few days, mechanically crushed and sieved to a particle size ranging from 0.064 to 1.0 mm before being used in the adsorption processes. All experiments were performed using eucalyptus leaves having a particle size of 0.064 mm. Most of the experiments were carried out on an activated eucalyptus leaves. Some of the experiments were carried out on the dried (or fresh) eucalyptus leaves without any further activation. Physical pre-activation was accomplished by heating the eucalyptus to the activation temperature (105˚C) for 1 h and keeping it at the required activation temperature for 2 h and 105˚C; it is familiar that the activation should remove volatile components and thus would possibly increase the surface area of adsorption.

2.2.2. Effect of adsorbent doses

The effect of adsorbent doses on the equilibrium adsorption of copper was investigated with eucalyptus leaves of 5, 10, 15, and 20 mg/ml in four set of 20 ml water which each contained 10, 20, 30, 40, 50, and 100 mg/l of copper concentration. The glass vials were shaken for 24 h with 100 rpm at room temperature (25˚C). The water samples were then filtered and analyzed in terms of Cu^{2+} by flame atomic absorption spectrophotometer (SPECTRO AA 10).

2.2.3. Effect of pH

The effect of pH for copper adsorption onto eucalyptus leaves was investigated at different pH values ranging from 2.5 to 8, time (24 h), and speed (100 rpm) for 100 ppm copper concentration in 20 ml water glass vials containing 0.1 g eucalyptus leaves. Acidic and basic pHs were adjusted by adding either 1 N phosphoric acid or sodium hydroxide solution. The glass vials were shaken for 24 h with 100 rpm at room temperature.

2.2.4. Effect of temperature

The effect of temperature on copper uptake was investigated at different temperature (25, 35, and 45˚C), time (24 h), and speed (100 rpm) for 100 ppm copper concentration in 20 ml water containing 5 mg/ml of eucalyptus leaves. The equilibrium data were fitted with Langmuir and Freundlich isotherm models.

2.2.5. Effect of contact time

Contact time experiments were conducted with 100 mg/l copper concentration in 20 ml water containing 5 mg/ml of eucalyptus leaves at room temperature (25˚C). Water was agitated at 100 rpm for 3 h. At different time intervals, water samples were withdrawn and filtered for analysis.

2.2.6. Effect of the presence of other ions

The effect of salt addition on the adsorption of Cu^{2+} ions was studied for solutions containing 100 mg/l copper concentrations and 0.0, 0.1, 0.5, and 1 M Na^+ ions or K^+ in the form of sodium chloride (NaCl) or potassium chloride (KCl), respectively. In each sets of experiments, 5 mg/ml of dried (or fresh) eucalyptus leaves were added and shaken for 24 h with 100 rpm at room temperature without pH adjustment. Another set of experiments were carried out on an activated (or heated) eucalyptus leaves.

2.3. Analysis

The collected water samples from different experiments were filtered using a 0.45 μm filter paper. The filtrate was analyzed using an atomic absorption spectrophotometer.

2.4. Calculation

The amount of copper adsorbed by adsorbent (q) was calculated by the following equation:

$$Q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where V is the volume of solution (l), m is the amount of adsorbent (g), and C_0 and C_e (mg/l) are the initial and equilibrium metal concentrations in the water, respectively.

3. Results and discussion

3.1. Effect of contact time

Fig. 1 shows the effect of contact time on copper uptake by eucalyptus leaves. The rate of copper removal was very rapid during the first 5 min, and thereafter, the rate of copper removal remained constant. There was no considerable increase in adsorption after about 10 min. The copper uptake increases with time and equilibrium was reached after 15 min. The higher adsorption rate at the initial period (first 5 min) may be attributed to an increased number of free sites on the adsorbent available at the initial stage; consequently there exist increased concentration gradients between adsorbate in solution and adsorbate on adsorbent surface [18,19]. Furthermore, at the initial stage of adsorption the mesopores become saturated with the adsorbed metal ions. Thus, a decrease in the mass transfer driving force between liquid and solid phase in the adsorption system occurred as time elapsed.

3.2. Effect of doses

The effect of doses was investigated with six copper concentration sets (10–100 mg/l) in 20 ml water by adding four doses for each set (Fig. 2). The copper uptake was found to increase with a decrease in the mass of adsorbent. The highest copper uptake was 1.16 for the initial copper concentration of 100 ppm at the adsorbent dose of 5 mg/ml. Subsequently, the copper uptake started to decline with increasing the adsorbent mass for all six sets. This may be attributed to the partial aggregation among the available active binding sites that may act for small increase in copper removal at high doses [6]. Thus, 5 mg/ml of eucalyptus leaves and 100 mg/l of copper concentration were chosen for the rest of experiments.

3.3. Effect of pH

Fig. 3 illustrates the effect of pH on uptake of Cu^{2+} for initial copper concentration of 100 ppm, eucalyptus leaves concentration of 5 mg/ml and shaking time of 24 h. It is evident that at pH values between 2.6 and 6.5, the copper uptake increases sharply and reached a maximum value (1.16 mg/g) at pH 6.5 (which was slightly acidic). The speciation diagram [20] has confirmed that at pH below 6.5, the Cu^{2+} is the main free species which involved in true adsorption. Lower adsorption capacity at low pH may be attributed to the competition between the Cu^{2+} with the H_3O^+ ions for binding on adsorbent sites [21,22]. At higher pH, the uptake was also low compared to the optimum condition. This may be attributed to the fact that, when pH is higher than 6.5, Cu^{2+} ions would precipitate as $Cu(OH)^{2+}$, therefore the uptake was not completely by adsorption [20].

3.4. Effect of salt addition

The effect of salt concentration on the removal of Cu^{2+} from aqueous solutions using activated and nonactivated eucalyptus leaves was investigated for solutions containing copper concentration of 100 ppm

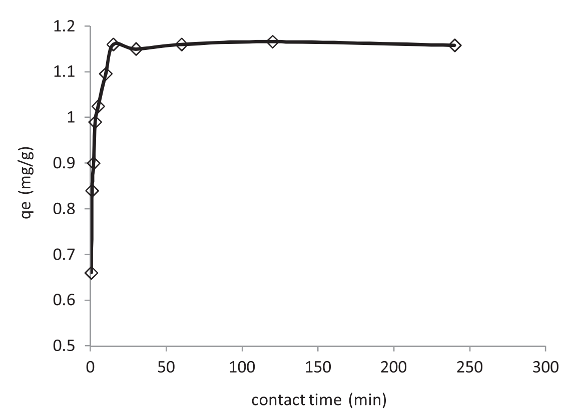

Fig. 1. Adsorption kinetics of copper on eucalyptus leaves (rpm: 100; pH: 6.5; T: 25°C, C_0: 100 mg/l; dose: 5 mg/ml; activated eucalyptus leaves).

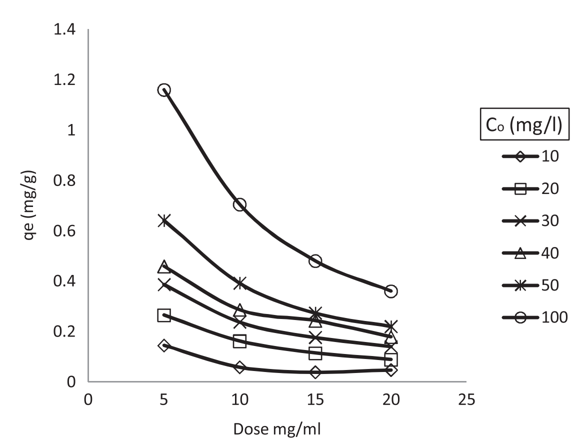

Fig. 2. Effect of adsorbent doses on the uptake of Cu^{2+} ions (rpm: 100; pH: 6.5; T: 25°C, C_0: 10–100 mg/l; activated eucalyptus leaves).

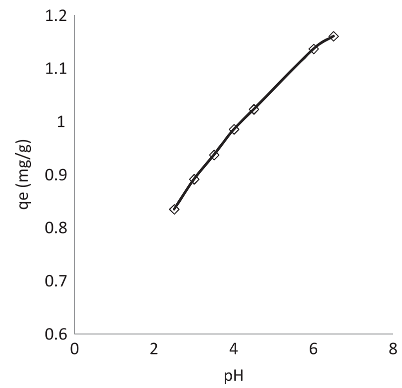

Fig. 3. Effect of initial pH on the uptake of Cu^{2+} ions by eucalyptus leaves (rpm: 100; pH: 6.5; *T*: 25°C, C_0: 100 mg/l; dose: 5 mg/ml; activated eucalyptus leaves).

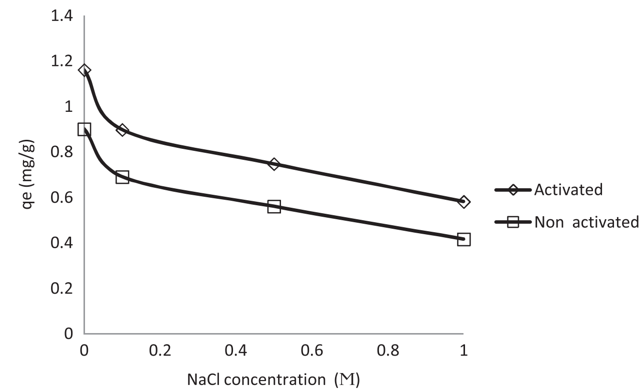

Fig. 5. Effect of NaCl addition on the uptake of Cu^{2+} ions by eucalyptus leaves (rpm: 100; pH: 6.5; T: 25°C; C_0: 100 mg/l; dose: 5 mg/ml).

and 0.0, 0.1, 0.5, and 1 M K^+ ions in the form of potassium chloride (KCl). Fig. 4 shows that for both activated and nonactivated eucalyptus leaves, the copper uptake was decreased in the presence of K^+ ions in the solution, and that an increase in salt concentration led to a further decrease in Cu^{2+} ion uptake. In the case of Na^+ ions, a more pronounced effect was obtained for this decrease in metal uptake with the increase in the salt concentration up to 1 M (Fig. 5). This may be attributed to the competition between Na^+ and Cu^{2+} ions on the vacant active sites of eucalyptus leaves surface. In such a case, Na^+ ions hinder Cu^{2+} ions from reaching the sorbent-active sites due to the repulsive forces. Furthermore, it was clear that the Cu^{2+} uptake by activated eucalyptus was higher than that by nonactivated, which showed that the adsorbent capacity was positively influenced upon activation. The same results were obtained by Deshkar and others [23], where the presence of soft and hard ions resulted in a negative influence of the sorption of Hg^{2+} by Hardwickia binata.

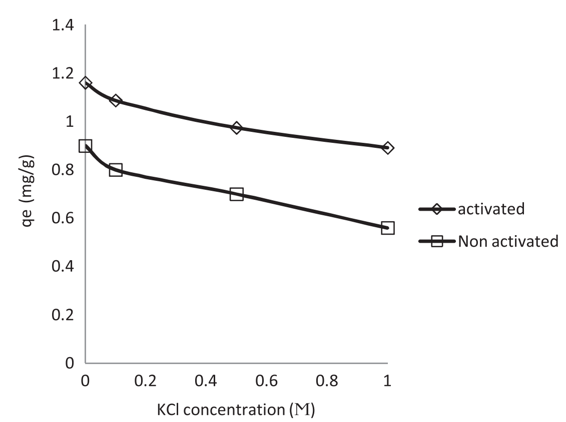

Fig. 4. Effect of KCl addition on the uptake of Cu^{2+} ions by eucalyptus leaves (rpm: 100; pH: 6.5; T: 25°C; C_0: 100 mg/l; dose: 5 mg/ml).

3.5. Adsorption isotherms

Adsorption isotherms are characterized by certain constant values, which describes the surface properties and the adsorbent affinity and can as well be applied to compare the adsorptive capacities of the adsorbent for different pollutants. The linearized form of the Freundlich and Langmuir relations was used to fit the equilibrium data. The correlation coefficients (R^2) were used to judge the applicability of the adsorption isotherm. Using Langmuir equation:

$$\frac{1}{Q_e} = \frac{1}{K_L} + \frac{1}{C} \cdot \frac{1}{bK_L} \tag{2}$$

where C is the equilibrium concentration (mg/l), K_L is the Langmuir constant related to the adsorption capacity (mg/g), and b is the Langmuir constant associated with the free adsorption energy (l/mg). Values of the calculated parameters along with regression coefficients at different temperature are presented in Table 1. The linear plot of Langmuir isotherm for copper adsorption is shown in Fig. 6. The maximum adsorption capacity, K_L, for complete monolayer coverage are found 1.92, 1.64, and 1.14 mg/g for 25, 35, and 45°C adsorption temperature, respectively. It was noticed that as the temperature increased from 25 to 45°C, the value of K_L (the maximum adsorption capacity) decreased from 1.92 to 1.14. R^2 values close to one, clearly suggest that the adsorption isotherms fit well with Langmuir isotherm model. Experimental results were also applied to the Freundlich adsorption

Table 1
Langmuir constants related to adsorption of Cu^{2+} from aqueous solution by eucalyptus leaves

Adsorption temp. (°C)	Langmuir isotherm		
	b	K_L	R^2
25	0.0099	1.919	0.995
35	0.0093	1.639	0.981
45	0.0081	1.136	0.980

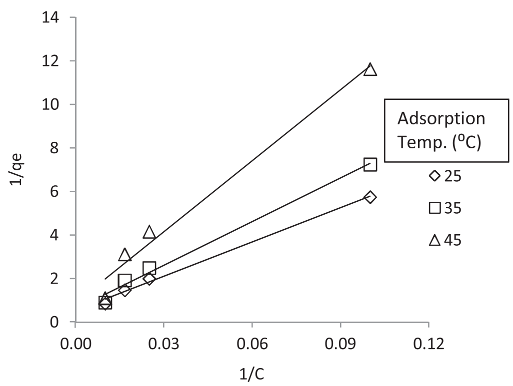

Fig. 6. Langmuir plot for the adsorption of Cu^{2+} by eucalyptus leaves (rpm: 100; pH: 6.5; C_0: 10–100 mg/l; dose: 5 mg/ml; activated eucalyptus leaves).

isotherm, but the linear state of the Freundlich adsorption isotherm was not obtained.

The dimensionless constant separation factor or equilibrium parameter, R_L [24] is given by:

$$R_L = \frac{1}{(1 + bC_0)} \quad (3)$$

where b is the Langmuir constant and C_0 is the initial concentration of Cu^{2+}. At adsorption temperature of 25°C, the values of R_L were found to be 0.9, 0.8, 0.7,

Table 2
The separation factor constants related to adsorption of Cu^{2+} from aqueous solution by eucalyptus leaves at different adsorption temperature

Initial Cu^{2+} conc. (ppm)	R_L		
	25°C	35°C	45°C
10	0.9	0.9	0.9
40	0.8	0.7	0.7
60	0.7	0.6	0.6
100	0.6	0.5	0.5

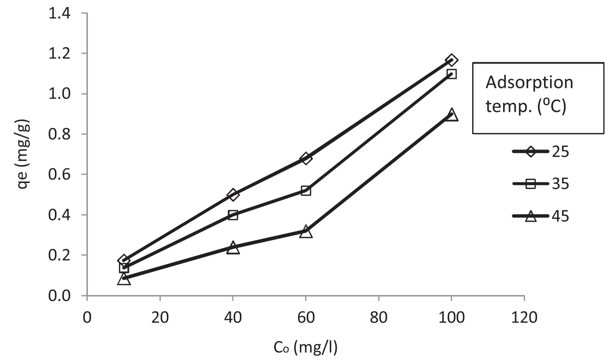

Fig. 7. Effect of solution temperature on the uptake of Cu^{2+} ions by eucalyptus leaves (rpm: 100; pH: 6.5; C_0: 10–100 mg/l; dose: 5 mg/ml; activated eucalyptus leaves).

and 0.6 for initial concentrations 10, 30, 40, 60, and 100 ppm, respectively. This indicates favorable adsorption of Cu^{2+} on eucalyptus leaves because all R_L values between 0 and 1. Similar results have been obtained when the adsorption temperature was increased to 35 and 45°C (Table 2).

3.6. Effect of temperature

To determine the thermodynamic properties and thermal effects on the adsorption, the temperature variation experiments were conducted at 25, 25, and 45°C, with an initial copper concentration between 10–100 mg/l and 5 mg/ml eucalyptus leaves. The experimental data show that the copper uptake was decreased with temperature (Fig. 7). It seems obvious that higher temperature has negative effect on copper adsorption, indicating that the process was exothermic in nature. To evaluate the nature of the adsorption process, the thermodynamic parameters such as Gibbs free energy ($\Delta G°$), enthalpy change ($\Delta H°$), and entropy

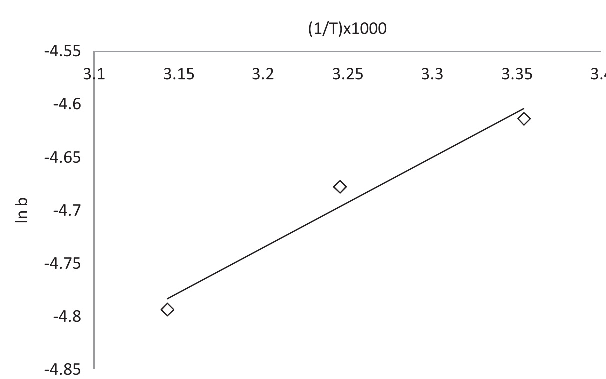

Fig. 8. Variation of ln b with reciprocal of temperature (rpm: 100; pH: 6.5; dose: 5 mg/ml; activated eucalyptus leaves).

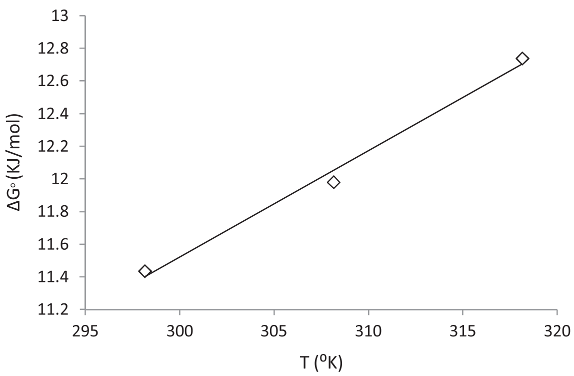

Fig. 9. Variation of ΔG° with temperature (rpm: 100; pH: 6.5; dose: 5 mg/ml; activated eucalyptus leaves).

Table 3
Calculated values of thermodynamic parameters for the equilibrium adsorption of copper onto eucalyptus leaves

Adsorption temp. (℃)	K_L (mg/g)	ΔG° (kJ/mol)	ΔS° (kJ/mol K)	ΔH (kJ/mol)
25	1.919	−11.44	−0.0651	−7.069
35	1.639	−11.98		
45	1.136	−12.74		

change (ΔS°) for copper ions adsorption by eucalyptus leaves were calculated using the following equations. The enthalpy change of adsorption (ΔH) (Eq. 4) as calculated from the slope of ln(b) vs. $1/T$ is shown in Fig. 8.

$$\text{In } b = \text{In } b' - \frac{\Delta H}{RT} \quad (4)$$

The magnitude of ΔH was determined as 7,069 J/mol. The negative value of ΔH° suggests that the adsorption reaction was exothermic in nature and strong affinity of the eucalyptus leaves for copper ions. The low value of ΔH (<62.7 kJ/mol) showed that the removal process was due to the physical adsorption mechanism [25]. The Gibbs free energy (ΔG°) and entropy change (ΔS°) were calculated to describe the nature of the adsorption process. At a certain temperature, a phenomenon is considered to be spontaneous if the ΔG° has a negative value. The value of ΔG° (kJ/mol) was calculated using the following equation:

$$\Delta G^\circ = -RT \text{ In } b \quad (5)$$

where R is universal gas constant, T is the absolute temperature (°K), and b is the adsorption equilibrium constant from Langmuir isotherm. ΔS° (kJ/mol.K) was calculated by the following equation:

$$\Delta G^\circ = \Delta H - T\Delta S^\circ \quad (6)$$

The plot of ΔG° vs. T was found to be linear (Fig. 9) and the value of ΔS°, was calculated from the intercept of the plot. The Langmuir and thermodynamic parameters calculated at different temperatures are shown in Table 3. The negative values of ΔG° for Langmuir isotherm have confirmed the feasibility of process and the spontaneous nature of adsorption. The values of ΔG° were increased from 11.44 to 12.74 kJ/mol as the temperature increased from 25 to 45℃. The magnitude of ΔS° calculated from Fig. 9 is given as 65.1 J/mol K. Furthermore, the negative value of ΔS° indicates that the adsorption reaction was enthalpy driven and spontaneous in nature [26].

4. Conclusions

Eucalyptus leaves is a high capacitate, economically viable, and low-cost adsorbent for copper removal. Adsorption of copper on eucalyptus leaves showed high association with Langmuir isotherm model. This study can conclude that eucalyptus leaves are a good alternative for copper removal from water. The adsorption capacity increased up to 1.16 mg of copper ion per gram of adsorbent when its initial concentration was 100 ppm. The copper uptake by activated eucalyptus leaves increased on increasing the metal concentration or the initial pH of the metal solution, but decreased on increasing the temperature of the solution. An increase in the eucalyptus leaves concentration in the suspension resulted in an increase of copper adsorption. Gibbs free energy values (ΔG) are negative and increases with an increase of temperature. The negative values of Gibbs free energy and entropy have confirmed the possibility of process and the spontaneous nature of adsorption. An increase in uptake of at least 29% was noted upon activation of the eucalyptus leaves.

Nomenclature

m	—	mass of the adsorbent (g)
C_0	—	initial concentration of adsorbate (mg/l)
C	—	concentration of adsorbate at equilibrium (mg/l)
R	—	the universal gas constant (J/mol K)
R^2		correlation coefficient
R_L	—	the dimensionless constant separation factor
q_e	—	mass of adsorbate adsorbed per unit mass of adsorbent at equilibrium (mg/g)

b — constant related to enthalpy (Langmuir model) (l/mg)
K_L — constant related to the maximum theoretical adsorption capacity (mg/g)
ΔH^o — enthalpy change (kJ/mol)
ΔG^o — Gibbs free energy (kJ/mol)
ΔS^o — entropy change (kJ/mol K)
b' — the adsorption energy constant

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