

Eggshell Powder as An Adsorbent for Removal of Cu (II) and Cd (II) from Aqueous Solution: Equilibrium, Kinetic and Thermodynamic Studies

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Abstract

The adsorption of Cu (II) and Cd (II) ions onto eggshell was investigated. The effects of contact time, initial pH of solution, adsorbent dosage, initial metal concentration, agitation speed, and temperature were studied in batch experiments. The maximum adsorption capacities for Cu(II) and Cd(II) were 8.4 and 7.01 mg/g, respectively. Thermodynamic parameters such as Gibbs free energy change, Enthalpy change and Entropy change have been calculated. These thermodynamic parameters indicated that the adsorption process was thermodynamically spontaneous under natural conditions and the adsorption was endothermic in nature. Experimental data were also tested in terms of adsorption kinetics, the results illustrated that the adsorption process was following pseudo second-order kinetics. In addition Fourier transfer infrared spectroscopy (FTIR) analysis suggests that Cu (II) and Cd (II) interact with metal oxides and -OH functional group present in eggshell powder.

Keywords: Eggshell, Copper ions, Cadmium ions, Adsorption, Kinetic.

1. Introduction

The presence of inorganic pollutants such as heavy metals in aquatic systems is of considerable concern because of their non-biodegradability, mobility and toxicity. Heavy metals can accumulate in the human body over time, causing serious health effects. Accordingly, the removal of heavy metals from wastewater has received a great deal of attention in recent years [1]. Several conventional physical and chemical wastewater treatment technologies including coagulation, filtration, evaporation recovery, precipitation, oxidation/reduction, electrochemical treatment, ion exchange and reverse osmosis have been used to remove heavy metals from aqueous systems. However, these methods are economically unfavorable or technically complicated, and are only used in special cases of wastewater treatment [2]. Moreover, most of these processes are only effective when there are high levels of metals in aqueous solution. There has recently been increased interest in the development of new

materials capable of removing toxic heavy metals from contaminated water [3]. The sorption process, which includes metal immobilization in contaminated water, has gained attention in the past few decades because it offers advantages such as high efficiency and low operating cost, and because it is eco-friendly when compared to conventional treatment methods [4]. Adsorbents such as activated carbons prepared from some agricultural by-products, some cellulosic wastes and their carbonization products, bituminous coal and commercial activated carbons were used [5]. Due to the high cost of the activation process, there is a need to adopt cheaper and readily available materials which can be used economically on a large scale [6]. Domestic egg consumption is high and continues to increase with the rapid increase of population, waste eggshells contain high contents of calcium carbonate (85–95%); therefore, their recycling or reuse has the potential to reduce environmental pollution while acting as a cost effective material for the immobilization of heavy metals in wastewater and soil [7]. Therefore, this study was conducted to evaluate the effectiveness of waste eggshells on the removal of heavy metals Cu (II) and Cd (II) from aqueous solutions.

2. Experimental Work and Methods

2.1. Preparation of Adsorbent

Chicken eggshells were used as the adsorbent in this study. The chicken eggshells were collected from kitchen waste and washed by deionized water for several times to remove the dirt particles. The eggshells were then air-dried and incubated in hot air oven at 40 °C for 35 minutes (protein component in eggshell can denature at high temperature; > 40 °C) [8]. Eggshells were ground to a powder in a grinder, and sieved to obtain between 60-100 mesh mesh to obtain (0.25-0.104 mm) size particles. The eggshell powder comprises of 94 % calcium carbonate, with small amounts of magnesium carbonate, calcium phosphate and other organic matter including protein [9].

2.2. Preparation of Simulated Wastewater

Stock solutions (1000 mg/L) of single Cu(II) and Cd(II) were prepared by dissolving an appropriate weight of pure salt $\text{Cu}(\text{NO}_3)_2$ and $\text{Cd}(\text{NO}_3)_2$ (manufactured by BDH, England) in the desired volume of de-ionized water. The stock solution was successively diluted with de-ionized water to obtain the desired test concentration of metal ions. Concentration of Cu(II) and Cd(II) were measured using atomic adsorption spectrophotometer (AAS) (Shimadzu, Japan).

2.3. Batch Experiments

Series of batch adsorption tests were conducted to determine the effects of contact time, initial pH, initial concentration of pollutants, adsorbent dosage, and operating temperature on adsorption performance of eggshells used as adsorbent materials. Therefore, various adsorbent dosages of 0.05, 0.1, 0.25, 0.3, 0.4, 0.5, 1, and 2 g were introduced into 250 mL flasks with 50 mL solution containing 50 mg/L of single Cu(II) and Cd(II). The flasks were then placed in an orbital shaker (Edmund Buhler SM25, German) and agitated up to a total contact time of 180 min at a fixed agitation speed of 250 rpm. Samples were taken at predetermined time intervals (10, 15, 20, 30, 35, 40, 50, 60, 80, 90, 120 and 180 minute) and then separated by filtration (filter paper type: Whatman 542, England). Contaminants concentration in the samples were analyzed by atomic absorption spectrophotometer (AAS) (Shimadzu, Japan).

Batch tests were carried out in a pH range of 3–7 to determine the effect of initial pH on adsorption because the precipitation of Cu(II) and Cd(II) is at pH greater than 9.7 [10]. The pH of the aqueous solution was adjusted by adding 0.1 M HNO_3 or 0.1 M NaOH as required. Effects of various operating temperatures ranging from 20 to 50 °C were also investigated in the batch studies. Temperature adjustments were conducted in the same orbital shaker. The efficiency of adsorption (%) was calculated as follows:

$$R = [(C_o - C_e)/C_o] \times 100 \quad \dots (1)$$

Where C_o and C_e are the initial and the equilibrium contaminant concentrations respectively. R is the removal efficiency (%).

2.4. Contact Time

Batch adsorption tests were carried out at different contact time intervals (0 - 180 minute) at initial contaminants concentration of 50 mg/L. This was done by contacting 0.25g of adsorbent with 50 mL of single contaminant solution at the pH 4.

2.5. PH Variation

The pH value of the solution is an important controlling parameter in the adsorption process, and the initial pH value of the solution has more influence than the final pH, which influences both the adsorbent surface metal binding sites and the metal chemistry in water [11]. The effect of pH on the single adsorption of Cu(II) and Cd(II) using eggshells was carried out within the range that would not be influenced by the metal precipitated [9]. It was reported that the suitable pH range for the adsorption of Cu(II) and Cd(II) was (3 - 7). This experiment was conducted at 20 °C to study the effect of initial pH value of solution on the adsorption of Cu(II) and Cd(II) by contacting 0.25 g of the adsorbent (eggshells) with 50 mL of 50 mg/L contaminant solution in a volumetric flasks.

2.6. Adsorbent Dosage

The solution of metal ion of 50 mL with concentration of 50 mg/L was added to various amount of the several adsorbents (0.05-2 g) in 250 mL volumetric flasks and agitated for 90 minute on a shaker, then the solution was filtered and analyzed.

2.7. Initial Metal Concentration

The initial metal concentration provides an important driving force to overcome all mass transfer resistance of metal between the aqueous and solid phases [12]. The initial concentrations of Cu (II) and Cd (II) were ranged from 50 to 100 mg/L.

2.8. Agitation Speed

The effect of agitation of the sorbent/sorbate system for Cu (II) and Cd (II) adsorption was monitored at (0, 50, 100, 150, 200 and 250 rpm).

2.9. Temperature Variation

The batch adsorption process was studied at different temperatures of 20, 30, 40, and 50 °C in order to investigate the effect of temperature on the adsorption process using eggshells. This was achieved by contacting 0.25 g of adsorbents with 50 mL of 50 mg/L single contaminant solution at pH 6 for 90 minute with agitation speed of 250 rpm by using shaker incubator. The results were used to investigate the thermodynamics of the adsorption process.

3. Kinetic Equations

The kinetics of Cu(II) and Cd(II) adsorption on eggshells were analyzed using pseudo-first-order [13] and Pseudo-second-order models [14], The pseudo-first order kinetic rate equation is:

$$(dq_t/dt) = k_1 (q_e - q_t) \quad \dots (2)$$

Where k_1 is the rate constant of pseudo first order adsorption (min^{-1}), q_e is the amount of contaminant sorbed at equilibrium (mg/g) and q_t the amount of contaminant sorbed at time t

(mg/g). Integrating equation (2) for the boundary conditions $t = 0$ to $t=t$ and $q_t=0$ to $q_t= q_e$ and rearranging yields the linear time-dependent function.

$$\text{Log}(q_e - q_t) = \text{Log} q_e - (kt/2.303) \quad \dots (3)$$

The intercept of the straight-line plots of $\text{log}(q_e - q_t)$ against t equal $\text{log} q_e$. However, if the intercept does not equal q_e , then the reaction is not likely to be first-order, irrespective of the magnitude of the correlation coefficient. The pseudo- second- order kinetic rate equation is:

$$(dq_t/dt) = k_2 (q_e - q_t)^2 \quad \dots (4)$$

Where k_2 is the rate constant of pseudo second order adsorption ($\text{mg} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$). Taking into account, the boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_e$. The integrated form of Equation (4) can be rearranged to obtain Equation (5):

$$(t/q_t) = (1/k_2 q_e^2) + (t/q_e) \quad \dots (5)$$

The plot of (t/q_t) and t of Equation (5) gives a linear relationship, q_e and k_2 can be determined from the slope and intercept of the plot, respectively.

4. Result and Discussion

4.1. Effect of Contact Time

The effect of contact time on the removal percent of Cu(II) and Cd(II) from the solution is shown in Figure (1).

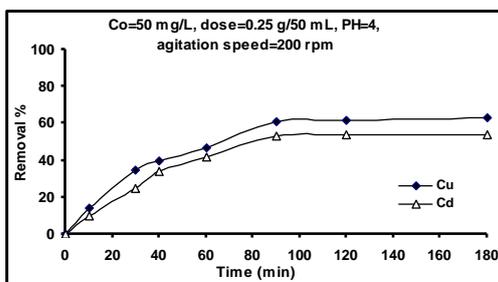


Figure 1: The effect of contact time on the removal percent.

It was observed that the removal of Cu(II) and Cd(II) by the eggshells increased with increase in contact time. The adsorption of Cu(II) was rapid for the first 80 minute as a result of available binding sites on the eggshells, while the adsorption of Cd (II) was less than Cu(II). The adsorption reached equilibrium within 90 minute where the removal percent reached 61% and 53% for Cu(II) and Cd(II), respectively. The period of 90 minute was therefore used for the adsorption of Cu (II) and Cd (II) by eggshells. The initial fast removal occurs due to surface adsorption on the adsorbent. The subsequent slow phase occurs due

to diffusion of the metal ions into the inner part of the eggshells.

4.2. Effect of pH on Adsorption

The pH of solution was examined at different pH values, covering a range of 3-7. The maximum adsorption was obtained at pH 6 for both contaminants. Removal of Cu (II) and Cd (II) by eggshells was pH dependent as shown in Figure (2).

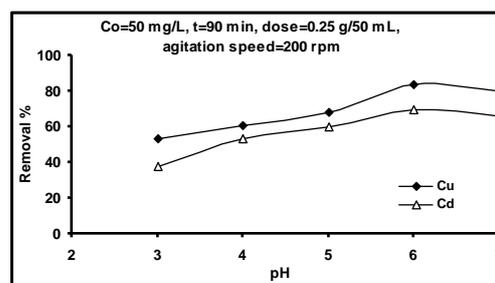


Figure 2: Effect of pH on the removal percent

At $\text{pH} < 3.0$, H^+ ions compete with Cu (II) and Cd (II) ions for the surface of the adsorbent which would hinder metal ions from reaching the binding sites of the sorbet caused by the repulsive forces. At $\text{pH} > 7$, the Cu (II) and Cd (II) will precipitate due to hydroxide anions forming copper and cadmium hydroxide precipitate. For this reason the maximum pH value was to be 6. At $\text{pH} = 6$ the highest removal efficiency was observed 84% and 70% for Cu (II) and Cd (II), respectively.

4.3. Effect of Eggshells Dosage

The adsorption of ions was observed to increase as the amount of adsorbent increased gradually from 0.05 to 2 g, as shown in Figure(3).

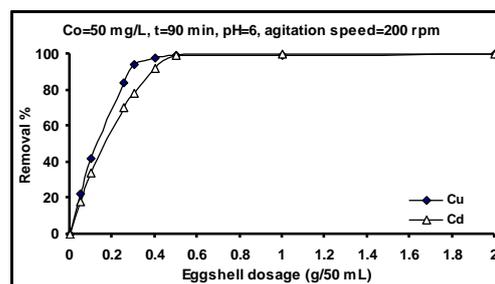


Figure 3: Effect of eggshells dosage on the removal percent

The maximum removal was obtained at the adsorbent dose of 0.4 and 0.5 g for Cu (II) and Cd (II), respectively. Where further increase in the quantity of the adsorbent more than (0.4 and 0.5)g had no more effect on the adsorption rate for Cu (II) and Cd (II), respectively. Therefore, 0.4 and 0.5 g of the adsorbent was sufficient to adsorb

maximum quantity of ions and also the percent removal of Cu (II) was more than that of Cd (II). Also the removal efficiency is associated with the adsorbent dose due to the availability of more adsorbing sites at higher doses.

4.4. Effect of Initial Metal Concentration

The effect of initial Cu (II) and Cd (II) concentration on the adsorption of these contaminants onto eggshells is shown in Figure 4. It could be seen that the removal percent of Cu (II) decreased from 98% to 76% with the increase in initial concentration from 50 to 100 mg/L, while this value decreased from 95% to 69% with the same range of increase in the value of initial concentration for Cd (II). This means that the amount of these contaminants sorbed per unit mass of adsorbent decreased with the increase in initial concentration. This plateau represents saturation of the active sites available on the eggshells samples for interaction with contaminants, indicating that less favorable sites became involved in the process with increasing concentration [15].

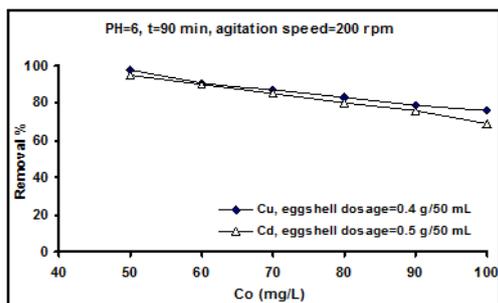


Figure 4: Effect of initial metal concentration on the removal percent.

4.5. Effect of agitation speed

The effect of agitation speed on the removal percent of Cu (II) and Cd (II) was studied by varying the speed of agitation from 0 to 250 rpm and keeping other parameters equal to the best obtained from the previous steps. Figure 5 shows that about 7.5% and 5% of the Cu (II) and Cd (II), respectively, were removed before shaking and the uptake increases with the increase in shaking rate. However, there was an increase in contaminants uptake when agitation speed was increased from zero to 250 rpm at which about 100% of Cu (II) and Cd (II) were removed. These results can be associated to the fact that the increase in the agitation speed improves the diffusion of contaminant towards the surface of the reactive media (eggshells). Thus, proper contact is developed between contaminants in the solution and the binding sites on the eggshells,

which promotes effective transfer of contaminants to the eggshells sites.

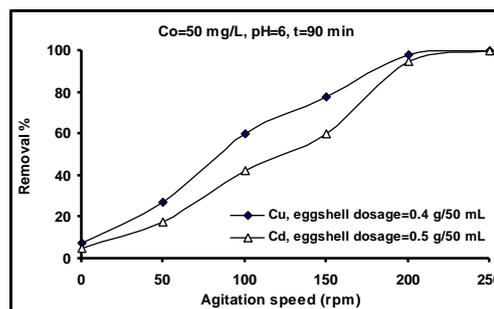


Figure 5: Effect of agitation speed on the removal percent.

4.6. Effect of temperature

The extent of Cu (II) and Cd (II) adsorption on the eggshells at various temperatures is shown in Figures (6) and (7).

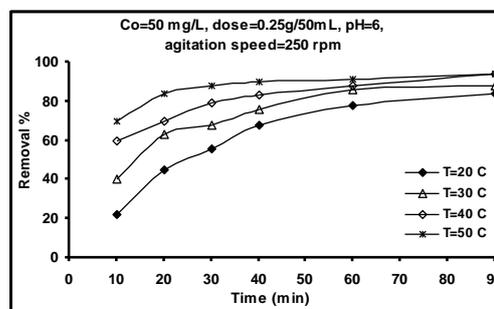


Figure 6: Effect of temperature on the removal percent of Cu (II).

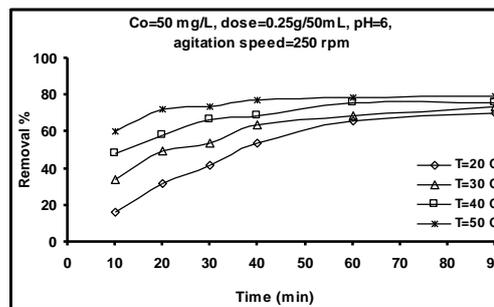


Figure 7: Effect of temperature on the removal percent of Cd (II).

As observed from the figures, temperature rises results in an increase in the removal a percentage of the metals ions. The enhancement of the adsorption capacity when temperature is increased is due to increased mobility and diffusion of the ionic species. The adsorption experiment could be regarded as a heterogeneous and reversible process at equilibrium. The apparent equilibrium constant for the process is shown to be:

$$K_c = (C_o - C_e)/C_e \quad \dots (6)$$

The Gibbs free energy of the adsorption process [16] is:

$$\Delta G^\circ = - RT \ln K_c \quad \dots (7)$$

where ΔG° is the standard Gibbs free energy change for the adsorption process (J mol^{-1}); R the universal gas constant ($8.314 \text{ J mol}^{-1}\text{K}^{-1}$), while T is the temperature (K). The effect of temperature on the adsorption of Cu (II) and Cd (II) by eggshell is listed in Table (1).

Table 1: Gibbs free energy values obtained from the adsorption of Cu (II) and Cd (II) using eggshell

Temperature (K)	$\Delta G^\circ (\text{KJ mol}^{-1}\text{K}^{-1})$	
	Cu (II)	Cd (II)
293	-4.03	-2.06
303	-5.02	-2.63
313	-7.16	-3.00
323	-7.39	-3.65

The free energy change (ΔG°) obtained for the adsorption of Cu (II) and Cd (II) at 293 K, initial concentration of 50 mg/L, and pH = 6 is (-4.03 and -2.06) kJ mol^{-1} for Cu (II) and Cd (II), respectively. The negative value of (ΔG°) obtained for the adsorption of Cu (II) and Cd (II) onto eggshells shows spontaneity of the adsorption process at that temperature. However, the result in Table (1) shows that the free energy values for the two contaminants decreases with increasing temperature. This implies that the spontaneity of the adsorption process increases with increasing the temperature. Consequently, the adsorption of the Cu (II) and Cd (II) using eggshells as adsorbent is endothermic hence the process is better carried out at kindly high temperature. From thermodynamics:

$$\Delta G^\circ = \Delta H - T\Delta S \quad \dots (8)$$

Or

$$\Delta G^\circ = - \Delta S (T) + \Delta H \quad \dots (9)$$

A plot of temperature against ΔG° gives a straight line with slope ΔS° and an intercept of ΔH . In (Figures 8 and 9) the slopes are (0.1222 and 0.0514) kJ/mol for Cu (II) and Cd (II), respectively. Therefore, the values of the entropy are (0.1222 and 0.0514) kJ/mol and enthalpy are (31.738 and 12.996) kJ/mol for Cu (II) and Cd (II), respectively.

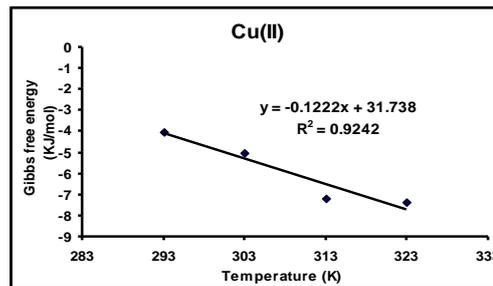


Figure 8: The Gibbs free energy plot for the adsorption of Cu(II) using eggshell.

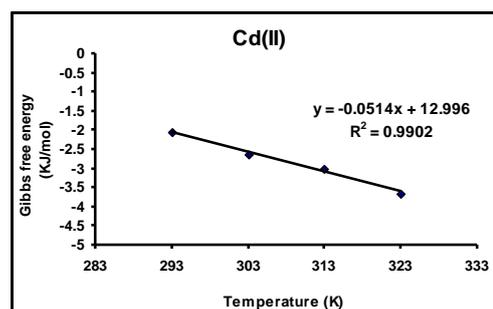


Figure 9: The Gibbs free energy plot for the adsorption of Cd(II) using eggshell.

4.7 Kinetic study

It is clear from Figures (10, 11, 12 and 13) and Table (2) that the reactions for Cu (II) and Cd (II) are not likely to be first order because of the value of q_e (experimental) does not equal to the value of q_e (theoretical), irrespective of the magnitude of the correlation coefficient, while the values of q_e (experimental) and q_e (theoretical) are very close with higher value of correlation coefficient in case of second - order, therefore the adsorption processes followed well pseudo second- order kinetics for the two contaminants.

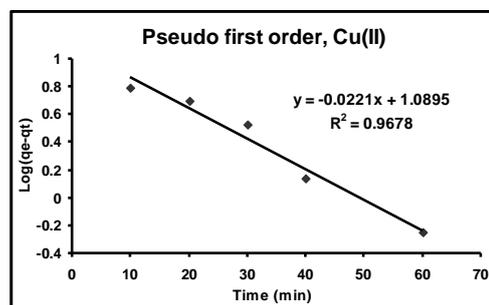


Figure 10: Pseudo-first order for Cu (II).

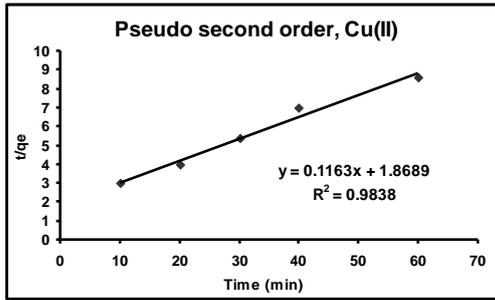


Figure 11: Pseudo-second order for Cu (II).

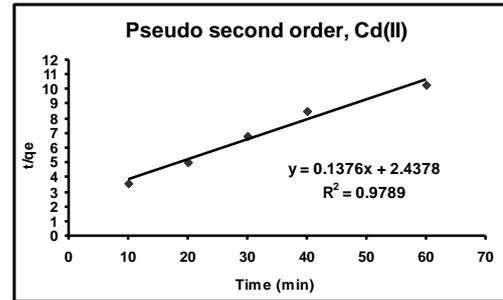


Figure 13: Pseudo-second order for Cd (II).

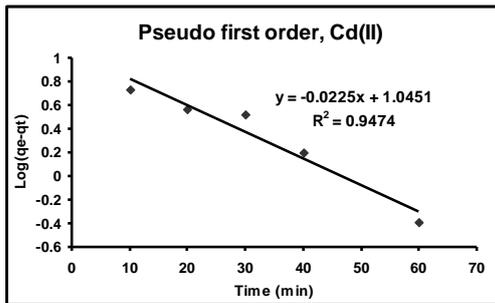


Figure 12: Pseudo-first order for Cd (II).

Table 2: Constants of pseudo-first and second order

Adsorbent	q _e (mg/g),Exp.	pseudo-first-order			pseudo-second- order	
Cu (II)	8.40	K ₁ , (min) ⁻¹	0.0508		K ₂ ,(mg.g ⁻¹ min ⁻¹)	0.0072
		q _e , (mg/g)	12.28		q _e , (mg/g)	8.59
		R ²		0.9678	R ²	0.9838
Cd (II)	7.01	K ₁ , (min) ⁻¹	0.0518	11.09	K ₂ ,(mg.g ⁻¹ min ⁻¹)	0.0077
		q _e , (mg/g)	0.9474		q _e , (mg/g)	7.2600
		R ²			R ²	0.9789

4.8 FTIR analysis of eggshells

The Fourier transfer infrared spectroscopy (FTIR) analysis has been considered as a kind of direct mean for identifying the characteristic functional groups on the surface of the eggshell, which are responsible for adsorption of metal ions [17]. The FTIR spectrum of eggshell powder was recorded to obtain the information regarding the stretching and bending vibrations of these functional groups. The FTIR spectra of eggshell powder before and after Cu (II) and Cd (II) adsorption are shown in Fig.14. The FTIR spectral analysis of eggshell powder shows distinct peak at 713.66, 875.68, 1419.61, 2515.18, 2912.5 and 3410.15 (1/cm). The peak observed at 3410.15 (1/cm) may be assigned to the presence of alcohol hydroxyl group (-OH) stretching. This result suggests that Cu (II) and Cd (II) interact with metal oxides and -OH functional group present in the eggshell powder.

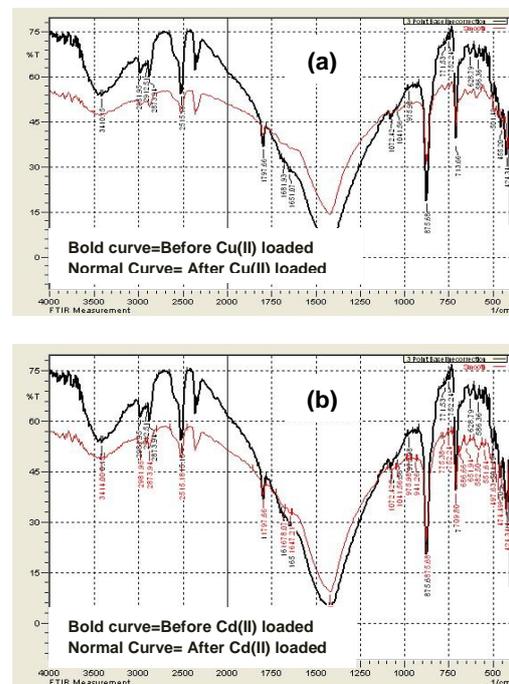


Figure 14: FTIR spectrum of eggshell powder before and after loaded of (a) Cu (II), and (b) Cd (II).

5. Conclusion

The potential use of eggshells as an adsorbent for Cu (II) and Cd (II) was studied. It was found that eggshell is more effective for removal of Cu (II) than Cd (II) from aqueous solution, the maximum adsorption capacities for Cu (II) and Cd (II) were 8.4 mg/g and 7.01mg/g, respectively. The percentage removal was strongly dependent on the contact time, initial pH of the solution, adsorbent dosage, and agitation speed. The study of the thermodynamic parameters indicated that the adsorption process was thermodynamically spontaneous under natural conditions and the adsorption is endothermic in nature. The process kinetics was found to follow the pseudo-second order rate equation.

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مسحوق قشر البيض كمادة ممتزة لأزالة أيونات النحاس والكاديوم من المحلول المائي: دراسة الموازنة، الحركية والديناميكية الحرارية

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كلية الهندسة/جامعة بغداد

الخلاصة:

تم التحقق في هذا البحث من استخدام قشور البيض كمادة ممتزة لامتزاز ايونات النحاس والكاديوم. وتم دراسة تأثير عدة عوامل منها زمن التوازن, الدالة الحامضية الابتدائية للمحلول, كمية المادة الممتزة, التركيز الابتدائي, سرعة الرج, بالإضافة الى درجة الحرارة. وكان الحد الاعلى لسعة الامتزاز (8.4mg/g) و(7.01mg/g) لايونات النحاس والكاديوم على التوالي. كذلك تم حساب بعض العوامل الترموديناميكية مثل Gibbs free energy Entropy, Enthalpy وقد اشارت هذه العوامل بان عملية الامتزاز هي عملية ترموديناميكيا تلقائية وماصه للحرارة . النتائج العملية اشارت ايضا بان عملية الازوريشن تتبع ل " pseudo second- order kinetics " . واخيرا تم اجراء فحص FTIR الذي اشار بوجود تداخل بين ايونات النحاس والكاديوم مع اكاسيد المعادن والمجموعه الفعالة (OH-) الموجوده في قشور البيض.