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Removal of Selected Metal Ions from Aqueous Solutions Using Chitosan Flakes

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ABSTRACT

Commercially available chitosan's potential in the adsorption of heavy metals like zinc, copper, cadmium, and lead from aqueous solutions under variable physicochemical conditions was investigated. The results obtained from equilibrium and kinetic studies showed that there was significant uptake of these metal ions by chitosan and that chitosan flakes had a maximum sorption capacity for copper ions. The order of metal ion adsorption by chitosan decreased from Cu^{2+} to Zn^{2+} as follows: copper $>$ lead $>$ cadmium $>$ zinc. There was a considerable increase in sorption capacity with an increase in chitosan amount; however, this parallelism diminished when the chitosan mass exceeded 0.24 g in 25 mL of metal solution. The sorption of metal ions from various salt solutions by chitosan flakes was not improved by agitation. The heavy metal uptake by chitosan was found to be pH-dependent, with a maximum at pH 6.0 and 7.0. Sorption equilibrium studies were conducted with a constant sorbent weight and varying initial concentration of metal ions. The experimental data of adsorption from solutions containing metal ions were found to correlate well with the Langmuir isotherm equation.

Key Words. Heavy metals; Chitosan; Adsorption; Kinetics; Equilibrium

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INTRODUCTION

The hazards associated with the pollution of water bodies caused by heavy metals have led to the development of various wastewater reclamation technologies. The traditional treatment processes, including filtration, ion exchange, precipitation with carbonate (1) or hydroxide (2), and a variety of sorbents such as peat (3) or Chelex 100 resin and amberlite (4), have been developed and practiced, along with their inherent merits and demerits, to clean heavy metal contaminated waters. For example, filtration and chemical precipitation have been considered as low cost technologies but neither of them is capable of removing trace levels of metal ions (5). Similarly, the effectiveness of ion exchange, which is capable of reducing metal ion concentrations to parts per million levels, in removing metal ions from wastewaters is decreased in the presence of large quantities of competing mono- and divalent cations.

The use of chelation ion exchange for wastewater remediation has been gaining attention in the recent past. Chelation ion exchange, in contrast to simple ion exchange, takes advantage of removing only toxic metal ions while the harmless ions move on into the environment (5). Some of the best chelation ion-exchange materials consist of different biopolymers and their derivatives because of the variety of functional groups, like —OH and —NH_2, with which other chemical moieties, e.g., metal ions, can easily reacted or be bound. These biopolymers, in fact, have remarkable capabilities of lowering metal ion concentrations to parts per billion levels (5–7), thus making them environmentally safe. Such materials include cellulosics, alginates, proteins, chitin, and chitin derivatives. Several investigators have reported the heavy metal binding capacities of chitin and chitosan (8–13). The latter is produced commercially by deacetylation of chitin with sodium hydroxide at high temperature.

Chitin (poly [β-(1–4)-2-acetamido-2-deoxy-D-glucopyranose]), also called *N*-acetyl-D-glucosamine, one of the most abundant organic materials produced annually by biosynthesis, occurs in animals, particularly in crustacea, molluscs, and insects, and in certain fungi. Its derivative chitosan (poly [β-(1–4)-2-amino-2-deoxy-D-glucopyranose]), also called D-glucosamine, has reactive amino groups which are responsible for complex formation between metal ion and the polymer. Chitosan is an heteropolymer made of D-glucosamine and a small fraction of *N*-acetyl-D-glucosamine residues. Likewise, polymer chains of chitin contain a proportion of D-glucosamine residue in addition to *N*-acetyl-D-glucosamine residue (14). Therefore, the adsorption ability of chitosan is found to be much higher than that of chitin, which has relatively fewer free amino groups. Chitosan also has many other useful features, for example, hydrophilicity, biocompatibility, biodegradability, antibacterial property, and a remarkable affinity for many proteins (15).

Muzzarelli and Tubertini originally described chitosan in terms of a natural chelating polymer (16). The ability of chitosan to form complexes with metal ions, particularly transition metal and posttransition metal ions, is well documented (14, 17, 18), and it is therefore considered as a promising adsorbent for removing heavy metals from polluted waters. Most previous studies have dealt with examining the capabilities of chitosan to adsorb metal ions from solutions containing a single solute, such as copper (8), cadmium (19), mercury (20), vanadium (17), nickel (21), lead (5), etc. However, wastewaters usually contain a wide variety of metal ions which might show a different adsorption pattern on chitosan flakes than if they were present alone. To the best of our knowledge, no attempts have been made to understand the physicochemical characteristics of the adsorption process by chitosan when various metal ions are present simultaneously in solution. In this paper the effects of various physiocochemical parameters, including chitosan amount, reaction time (with or without shaking), pH, and initial metal ion concentration, on the simultaneous adsorption of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+} by chitosan flakes have been investigated.

MATERIALS AND METHODS

Chitosan (with 85% deacetylation) in the form of flakes was purchased from Sigma Chemical Co., St. Louis, MO, and was used in this form. Metal ion solutions containing the desired concentrations of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+} together were prepared from analytical grade standard solutions (1000 $mg{\cdot}L^{-1}$). The effects of the following different physicochemical parameters on the adsorption potential of chitosan were studied.

Chitosan Amount

Different amounts of chitosan (ranging from 0.06 to 0.6 g) and 25 mL of the salt solution mixture containing 10 $mg{\cdot}L^{-1}$ of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+} were shaken at 150 rpm for 24 hours at ambient temperature and centrifuged at 2500*g* for 20 minutes. The concentrations of the metal ions present in the supernatant were analyzed using AAS (Perkin-Elmer 2380). The chitosan amount showing maximum adsorption of metal ions was used for further experiments.

Contact Time

Chitosan (0.250 g) was placed with 25 mL of the salt solution mixture containing 10 $mg{\cdot}L^{-1}$ of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+}. The flasks were shaken at 150 rpm for 72 hours at ambient temperature. To assess the need of shaking, a control set of experiment without shaking was also performed.

The metal ions–chitosan mixtures from both sets of experiments were collected at 0, 3, 6, 12, 24, 48, and 72 hours, centrifuged at 2500*g* for 20 minutes, and the amounts of metal ions in the supernatant were analyzed using AAS. The 0-hour observation was used to compare the adsorption of metal ions by chitosan at various contact times. The results obtained in this step were applied in further studies.

pH of Salt Solutions

The salt solution mixture, containing Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+} (10 $mg{\cdot}L^{-1}$ each), was prepared in standard buffers having pH values of 4 and 5, 6 to 8, and 9 obtained, respectively, with acetate (0.1 M), phosphate (0.1 M), and carbonate (0.1 M) buffers. The pH of the buffer solution was maintained using acidic and basic components of the buffer. The pH of acetate buffer was adjusted using acetic acid and sodium acetate. The pH of phosphate buffer was regulated using potassium dihydrogen phosphate and sodium hydroxide. The pH of carbonate buffer was corrected using sodium bicarbonate and sodium hydroxide. Changes occurring in the pH of the buffers on addition of metal solution were adjusted with appropriate bases. Flasks having 0.250 g of chitosan and 25 mL of the solution, containing metal ions, were equilibrated (without shaking) for 12 hours at ambient temperature. Subsequently, each of the mixtures was centrifuged at 2500*g* for 20 minutes and the amounts of metal ions present in the supernatant were estimated by AAS. The pH value exhibiting maximum adsorption of metal ions was selected for further experiments.

Concentration of Metal Ions

Chitosan (0.250 g) was mixed with 25 mL of the solutions containing 0 to 20 $mg{\cdot}L^{-1}$ metal ions, i.e., Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+}, prepared in 0.1 M acetate buffer, pH 6.0, for 12 hours at ambient temperature. After 12 hours of contact time the mixtures were centrifuged at 2500*g* for 20 minutes and the concentrations of the metal ions in the supernatant were estimated using AAS.

The adsorption capacity of chitosan (mg $metal{\cdot}g^{-1}$ chitosan) was calculated by the following equation (15, 17):

$$q = (C_0 - C_e)V/m$$

where q = metal ions adsorbed by chitosan ($mg{\cdot}g^{-1}$)
C_0 = initial metal concentration ($mg{\cdot}L^{-1}$)
C_e = metal concentration at equilibrium ($mg{\cdot}L^{-1}$)
V = volume of metal solution (L)
m = mass of chitosan (g)

The experiments were conducted in triplicate and the data presented represent the average of three values. The deviations from the central mean value are represented by standard deviation.

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RESULTS AND DISCUSSION

Chitosan flakes exhibited a tremendous capability in accumulating Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+} from the aqueous solutions (Figs. 1–5). The degree of absorption, however, varied with chitosan mass, reaction time, pH, as well as the metal ion identity and its initial concentration. The order found for metal ion adsorption by chitosan is as follows: $Cu > Pb > Cd > Zn$. The solutions containing 10 $mg{\cdot}L^{-1}$ of each metal ion showed a reduction of 95, 84, 48, and 44% in Cu^{2+}, Pb^{2+}, Cd^{2+}, and Zn^{2+} levels, respectively. The present observations showing maximum adsorption in the case of copper are consistent with those reported earlier (5, 20). The percentage reduction in metal ion contents found here is also similar to the one observed by Deans and Dixon (5) and Mitani et al. (9). For example, chitosan mixed with solutions containing 10 $mg{\cdot}L^{-1}$ of Cu or Pb resulted in a reduction of 92 and 83%, respectively (5). Despite the fact that an absolute comparison of results with other reported findings is difficult due to varying techniques, conditions, and the physical states of chitosan used in the experiments, attempts have been made to discuss our results in the light of previous ones. However, in contrast to other studies that involved a single type of metal ion (8, 19, 20, 22), the results presented take into account the presence and/or interactions of other metal ions on the adsorption capacity of chitosan under different physicochemical conditions.

Various processes such as adsorption, ion exchange, and chelation dominate the mechanisms responsible for complex formation between chitosan and metal ions. For calcium, ion exchange is the dominant process, whereas for other metals, such as Cu and Pb, adsorption with some chelation is more important (13). Also, the interactions of metal ions with chitosan are believed to be influenced by the degree of polymerization and deacetylation, and the distribution of acetyl groups along the polymer chains (23). The evidence currently available supports the concept that chitosan–metal ion complex formation occurs primarily through the amino groups functioning as ligands (14, 21, 24). Further, an increase in sorption performances due to the grafting of more amino groups has confirmed their involvement in complex formation (25). By definition, a chelate formation requires the involvement of two or more complexing groups from the same molecule. Therefore, the metal ion seeks two or more amino groups from one molecule of chitosan. The two -NH_2 groups could come from two different glucosamine residues of the same molecule, or from two different molecules. The amino group (NH_2) on the chitosan has been suggested as the active site for metal ion coordination because the lone pair of electrons present on the nitrogen can establish dative bonds with transition metal ions like Zn^{2+}, Cu^{2+}, etc. Kurita et al. (7) also demonstrated a positive correlation between amino groups and the adsorption rate of mercury and copper. Nevertheless, the sorption of metal ions by various other nitrogen-containing polymers is well docu-

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mented, but chitosan has been reported to have a higher nitrogen content (7.2%) than the other substances (chitin, amberlite, etc.) and is thus much superior as a metal adsorbent (8). However, the involvement of ligands other than amino groups in complex formation can not be ignored as the concentration of adsorbed metal ions in some cases was found to be greater than the total concentration of amino groups in the sample (14).

Figure 1 shows the relationship between metal ions adsorbed by chitosan flakes and the chitosan mass. As is evident, the different amounts of chitosan used resulted in a significant uptake of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+}, varying with metal species. The parallelism between an increase in chitosan amount and adsorption of metal ions by chitosan was found until 0.24 g chitosan was used, beyond which the sorption of metals became constant. Thus, 0.24 g of chitosan was sufficient to reach the adsorption equilibrium between chitosan and 25 mL of metal solution containing 10 $mg \cdot L^{-1}$ of each metal ion. Based on these results, it can be speculated that for higher metal concentrations and larger volumes of metal solutions, more chitosan amount would be required and vice versa.

Figure 2 shows that the amount of chitosan used at all reaction times exhibited a considerable uptake of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+}. However, varying the length of the contact time between the chitosan and metal ions seemed to have little effect on the adsorption capacity of chitosan (Fig. 2). The increase in the adsorbed metal ion contents leveled off after 6 hours of exposure to chitosan, indicating the attainment of adsorption equilibrium. Longer con-

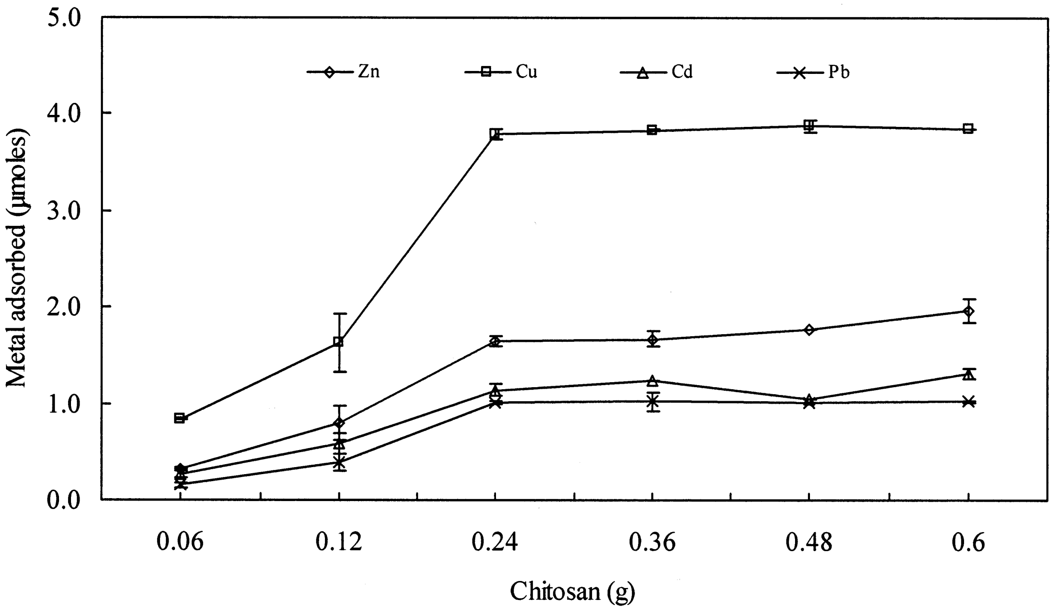

FIG. 1 Heavy metal adsorption on chitosan flakes as a function of chitosan mass.

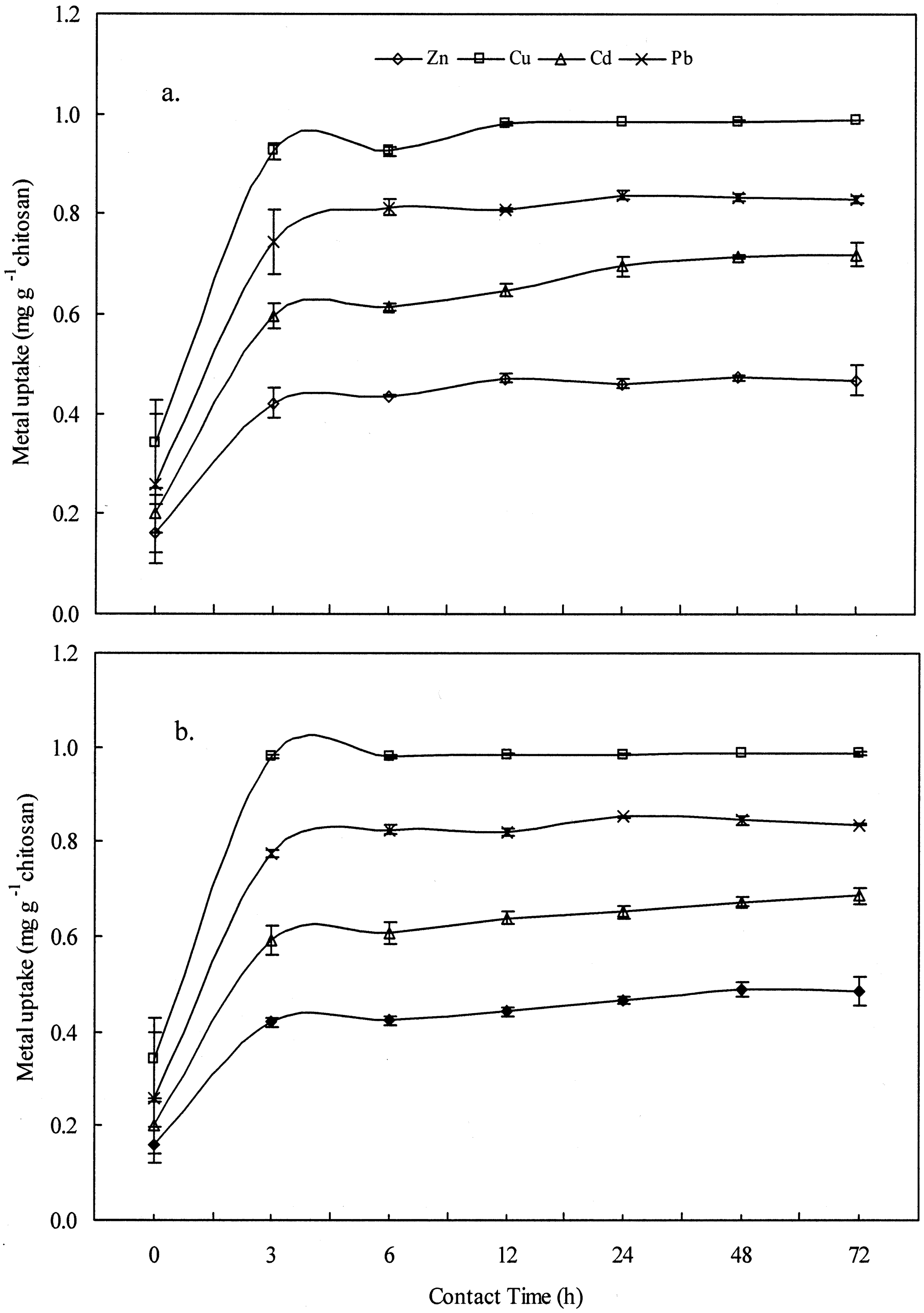

FIG. 2 Time course of heavy metal uptake by chitosan flakes without (a) and with shaking (b).

tact times (>3 hours) between chitosan and metal solutions have already been reported not to result in an increase in metal ion uptake (5, 12, 20). Furthermore, Udaybhaskar et al. (24) studied the removal of chromium(VI) in the 1–5 $\mathrm{mg \cdot L^{-1}}$ range by chitosan as a function of time and found that the removal of Cr was very rapid initially, with more than 80% of total removal being within 1 hour at all concentrations used. An initial rapid uptake of cadmium was also observed by Jha et al. (19). However, Mitani et al. (9), while studying the metal ion uptake by chitosan beads, agitated the metal–chitosan mixture for 48 hours. There are other reports describing that continuous stirring for 7 days achieve equilibrium (8).

Surprisingly, not much difference in the chitosan-induced adsorption of metal ions was shown by the metal ion samples tested in this study, with and without shaking (Fig. 2). Metal ion sorption seems to be uncontrolled by the shaking of the metal–chitosan mixture. This is possibly due to the metal ion uptake being limited to the surface of chitosan, as shown by Jansson-Charrier et al. (17). They observed a very restricted effect of stirring speed on the overall sorption rate, concluding that the control of metal ion adsorption onto chitosan was mainly due to intraparticle diffusion. In general, the adsorption of a material from the solution on adsorbent has been suggested to occur in three consecutive steps, (a) sorption at the surface of the adsorbent, (b) diffusion of adsorbate into the pore of the adsorbent, and (c) sorption on the internal surface of the adsorbent. The third step is assumed to be rapid while Steps (a) and (b) are the rate-determining steps, either singly or in combination (8). Helfferich (26) stated the external transport as the rate-limiting step in systems which have poor mixing, dilute concentration of the sorbate, small particle size of the sorbent, and high affinity between the sorbent and sorbate. Peniche-Covas et al. (20), however, confirmed the intraparticle diffusion as the rate-determining step. Nevertheless, the required contact time for reaching maximal metal ion adsorption was 6 hours, but to ensure complete achievement of adsorption equilibrium, it was extended to 12 hours in subsequent studies.

Generally, the adsorption of metal ions by chitosan has been found to be pH sensitive (14). Adsorption of Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+} on chitosan flakes was observed at all levels of pH studied (Fig. 3). The uptake of metal ions by chitosan showed a rise as the pH increased from 4 to 7. This could be due to the greater availability of amino groups at higher pH values. The ESR spectra of chitosan membranes complexed with Cu ions revealed the involvement of a higher number of amino groups with an increase in pH of the complex solution (14). The reduced adsorption of metal ions at acidic pH values could be attributed to the fact that at a lower pH the metal ions that would coordinate with the lone pair of nitrogen would have to compete with H_3O^+ for an active site (19). Muzzarelli and Tubertini (16) observed a decrease in uptake capacity for many metal ions by chitin and chitosan when solution pH was de-

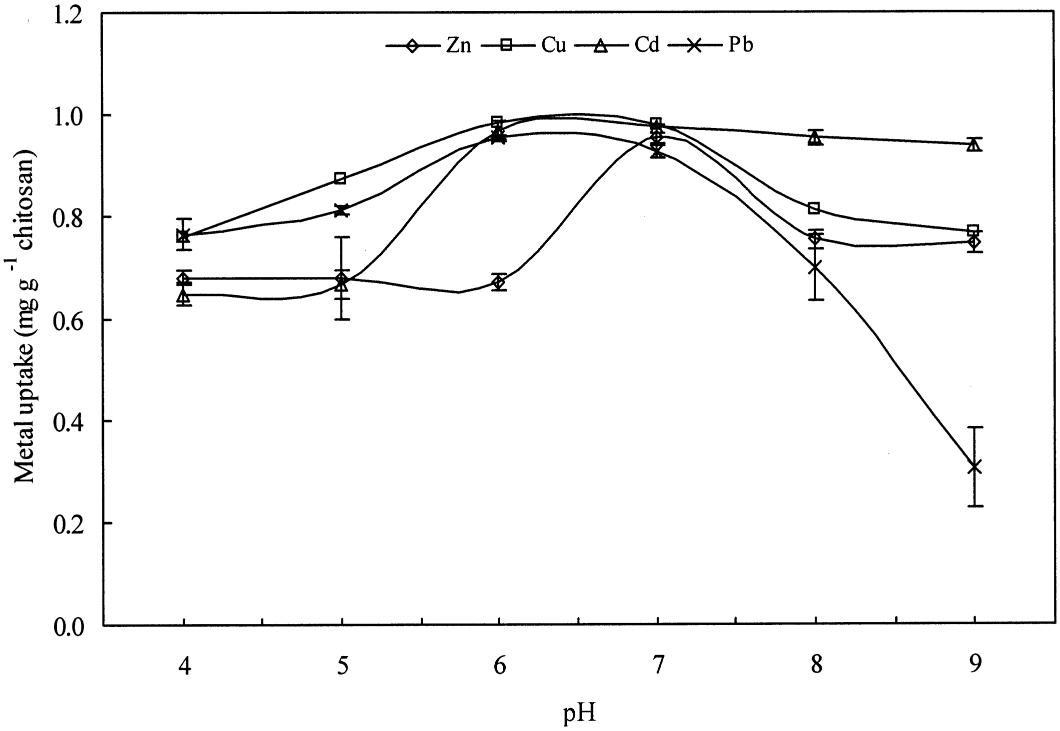

FIG. 3 Effect of pH on the adsorption potential of chitosan flakes.

creased from 7 to 2.5 whereas hexavalent chromium uptake by chitosan decreased as the pH increased from 3 to 5 (24). The correspondence between rising pH and adsorption of metal ions was not found above pH 7. The experimentally observed decrease in metal removal at higher pH values may be explained by rapid changes in protonated and unprotonated forms of chitosan (24). The maximum decrease in the uptake of metal ions was found in the metal solution having pH values of 6.0 and 7.0.

The adsorption capacity of chitosan flakes for metal ions from solutions having initial concentrations ranging from 2 to 20 mg·L^{-1} was studied (Fig. 4). The pattern of sorption of metal ions reduction in metal ion contents was virtually irregular, but the maximum reduction was obtained in metal ion solutions containing 16 mg·L^{-1} of heavy metals. These observations are in close agreement with those obtained by Peniche-Covas et al. (20), who concluded that the adsorption capacity of chitosan is increased by augmenting mercuric chloride concentrations until an equilibrium concentration is reached. A significantly reduced adsorption efficiency of chitosan by raising the initial concentrations of copper and lead from 10 to 100 ppm was also found (5). Increasing metal ion concentration in the solutions seems to reduce the external diffusion of the adsorbate and enhances intraparticular diffusion (17). In the present studies the adsorption equilibrium was attained with solutions having

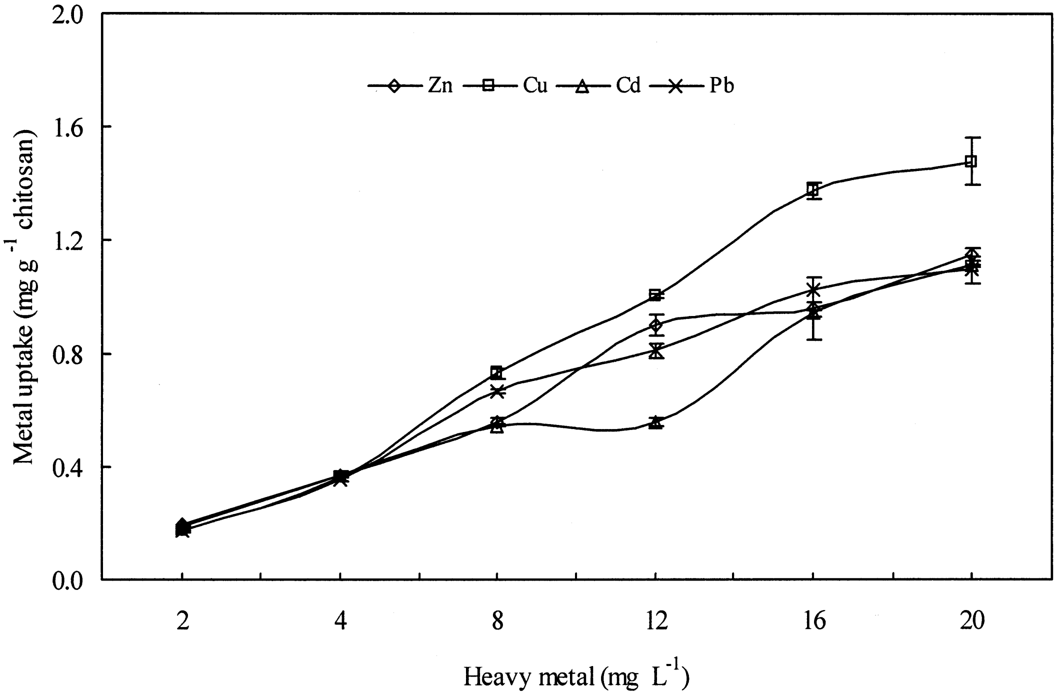

FIG. 4 Effect of initial metal ion concentration on the adsorption potential of chitosan flakes.

16 mg·L^{-1} of metal ions, from which an adsorption of 0.95, 1.37, 0.94, and 1.02 mg/g chitosan was observed for Zn^{2+}, Cu^{2+}, Cd^{2+}, and Pb^{2+}, respectively.

The equilibrium of a solute separated between two phases can be determined from the partition of the solute concentration in the solid phase as a function of the solute concentration in the liquid phase. These sorption curves or isotherms were studied by varying the initial concentrations of metal ions considered. Various models of sorption isotherms, such as the Langmuir and Freundlich models, have commonly been used to find the sorption potential of an adsorbent. In this study the average values from Fig. 4 were fitted to the linear form of the Langmuir equation, which assumes uniform distribution of sorption sites and sorption energies without interactions between the sorbed molecules (27). The Langmuir equation is represented by the following expression:

$$q = q_{\mathrm{m}} \frac{bC_{\mathrm{e}}}{1 + bC_{\mathrm{e}}}$$

where q = metal uptake or adsorption capacity (mg·L^{-1})
C_{e} = equilibrium concentration or final concentration of metal ion (mg·L^{-1})

q_m = maximum metal uptake or maximum adsorption capacity ($mg \cdot g^{-1}$)
b = adsorption equilibrium constant ($L \cdot mg^{-1}$)

The values of Langmuir's coefficients q_m and b can be derived from a straight-line equation that was obtained by plotting C_e/q versus C_e. These coefficients were determined separately for each metal ion considered and are presented in Table 1. It may be mentioned that though the experiments were carried out using a mixture of metal ions and chitosan, the data for each metal ion was used to calculate the coefficients. The adsorption coefficient q_m is the solid-phase concentration corresponding to a condition in which all available sites are filled, i.e., the maximum adsorption capacity. Thus, the isothermal data can be used to calculate the ultimate sorption capacity of chitosan by substituting the required equilibrium concentrations in Langmuir's equation. The coefficient b is related primarily to the net enthalpy of adsorption. The highest value of q_m obtained for copper after applying the experimental results to the sorption model clearly indicates the greater adsorption capacity of chitosan flakes for copper ions (Table 1). The experimental data on the adsorption of other metal ions, such as zinc, cadmium, and lead ions, produced almost similar values of q_m.

The values of q for the metal ions considered, calculated by using the sorption model, were plotted against C_e (Fig. 5). The data showed a very good correlation between the calculated and the experimentally measured values of metal ion concentrations in the solid phase. However, increasing the initial metal ion concentration may not increase the equilibrium concentration in the solid phase as the plateau is reached in all the isotherms. Although a correlation exists between the measured and calculated values, the latter were considerably lower than the values previously reported by using the same sorption model for certain metal ions. For example, the maximum adsorption of mercury calculated from Langmuir model was 430 mg Hg^{2+}/g chitosan (20). Sim-

TABLE 1
Langmuir Constants for Adsorption of Metal Ions on Chitosan Flakes

Heavy metals	Langmuir constants	
	q_m ($mg \cdot g^{-1}$)	b ($L \cdot mg^{-1}$)
Zinc	11.66	0.97
Copper	20.92	0.53
Cadmium	9.93	0.79
Lead	12.61	0.66

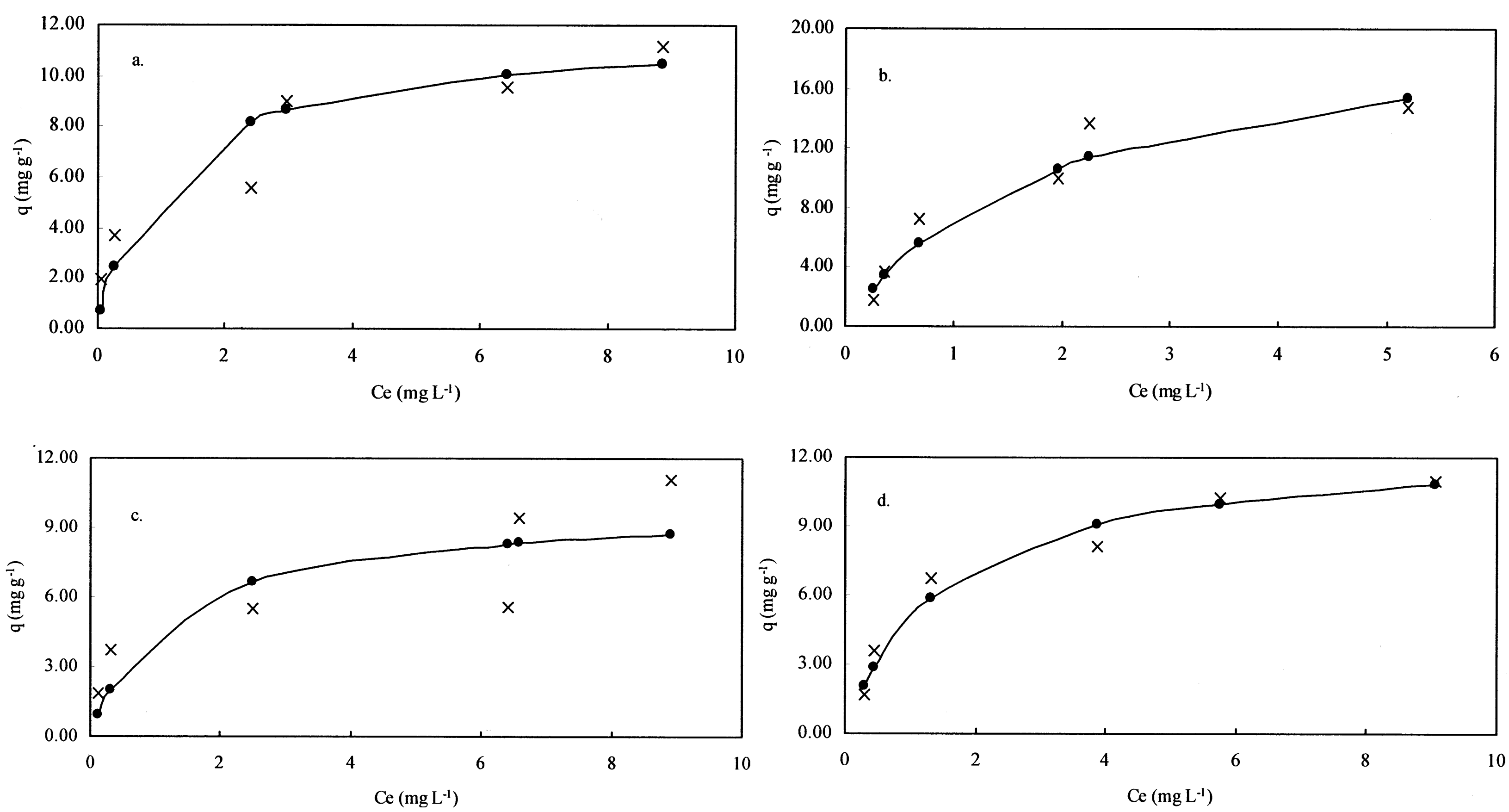

FIG. 5 Adsorption isotherms for zinc (a), copper (b), cadmium (c), and lead (d) on chitosan flakes (– • – calculated; × observed).

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ilarly, values of the adsorption coefficient ranging from 305 to 877 mg$\cdot$g^{-1} chitosan were calculated for vanadium (17). The difference in the values of maximum adsorption could be due to variations in metal ion identity and concentration, and also to the experimental conditions.

CONCLUSIONS

The present findings indicate the suitability of applying chitosan flakes in programs aimed at removing heavy metals like zinc, copper, cadmium, and lead from wastewaters. This process, in fact, generates a concentrated heavy metal stream from which precious metals can be recovered by washing the metal-laden chitosan flakes with some acidic solutions. This study has shown that the adsorption of metal ions by chitosan flakes is largely affected by the metal ion identity, the initial concentration of metal ions, the pH of the solution, the reaction time, and the adsorbent mass. Of the metal ions studied, maximum adsorption by chitosan was observed for copper ions. The sorption of metal ions by chitosan flakes was optimum at pH 6.0 and 7.0. The adsorption of metal ions by chitosan was initially rapid up to 3 hours and then remained constant. Shaking of the chitosan flakes–metal ion mixture did not appear to affect the sorption capacity of chitosan for the metal ions. Equilibrium isotherm studies reveal that the sorption of zinc, copper, cadmium, and lead onto chitosan can be described Langmuir’s equation.

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