SEPARATION SCIENCE AND TECHNOLOGY
Vol. 39, No. 14, pp. 3331–3351, 2004

Removal of Heavy Metals from Wastewater by Adsorption on Coir Pith Activated Carbon

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ABSTRACT

The activated carbon prepared from coconut coir pith by potassium hydroxide activation was found to exhibit remarkable adsorption capacity for cadmium, copper, and zinc. Batch equilibrium tests showed that the extent of metal removal was found to be dependent on initial concentration, contact time, pH, and carbon dose. It was found that at pH values below 3, the adsorption of metal ions was very less and was effective above pH 6. The metal ion sorption was found to follow the Freundlich model. The kinetics of adsorption of metal ions followed Ist order. The agglomerated coir pith carbon was evaluated for the effect of flow rate and bed depth in column experiments. Linear relationships between bed depth and service time were obtained for all the metal ions by performing bed-depth service time (BDST) analysis. The adsorbed metal ions could be quantitatively recovered by using

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DOI: 10.1081/SS-200036561 0149-6395 (Print); 1520-5754 (Online)

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1.0 M HCl, and the capacity of carbon remained unaffected when put to repeated use for the removal of metal ions from aqueous solutions. The efficiency of carbon when applied to industrial effluents was found to be superior in removal of metal ions compared with the commercial ones.

Key Words: Coir pith; Activated carbon; Heavy metals; Adsorption; Batch studies; Freundlich isotherm; Kinetics; Column studies; BDST; Effluent treatment.

INTRODUCTION

The increasing contamination of urban and industrial wastewaters by toxic metal ions is a serious environmental problem. These inorganic micropollutants are of considerable concern because they are non-biodegradable, highly toxic, and have a probable carcinogenic effect.[1] The traditional techniques for the removal of metal ions from aqueous effluents are incapable of reducing concentration to the levels required by law (process of reduction or lime precipitation) or prohibitively expensive (process of ion exchange and electrolytic removal).[2–4] Adsorption appears to be one of the most promising techniques for the treatment of waste streams contaminated by metal ions, because it offers the advantage of recovering the adsorbed metals for recycling or reuse.[5] Activated carbon has long been recognized as an effective adsorbent for this purpose.

According to a review by Huang[6] metals that activated carbons are capable of adsorbing include Cr(III,VI), Cd(II), Hg(II), Cu(II), Fe(II,III), Zn(II), Ni(II), V(IV,V), Au(I), and Ag(I). The best metal adsorbing activated carbons are reported to be made from “soft,” low-density precursors, such as rice hulls and sugarcane bagasse in contrast to “hard” density precursors.[7] Coir pith, an agricultural by-product generated in billions of kilograms annually, is used as precursor material for this study. Since granular activated carbons (GAC) are better suited than powdered activated carbons (PAC) for fluid flow where fixed bed systems are used, where the decrease in column pressure drop is minimum, the activated carbon can be easily regenerated for reuse.[8] The activated carbon obtained from coir pith was agglomerated with polyvinylacetate (PVAC) to granular form. The objectives of this study were to (i) determine the efficiency of coir pith activated carbon (CPC) to remove Cu, Cd, and Zn from aqueous solution and industrial effluents and (ii) compare the effectiveness of the prepared CPC with that of commercial activated carbon.

EXPERIMENTAL

Preparation of Chemically Activated Carbon

Coconut coir pith was sieved and was washed several times with distilled water and was dried at 110°C for 24 hr. Chemically activated coir pith carbon was prepared by treating 50 g of coir pith with 50 mL of 10% KOH solution and was subjected to activation at 700–750°C for 30 min. The carbon obtained was repeatedly washed with distilled water and then with 10% hydrochloric acid. The carbon was washed again with distilled water to remove the free acid and dried at 110°C. The carbon was ground to a finer size by using mortar and pestle, and particle size of 0.2–0.3 mm, which was retained to 80% was used for further studies. The powdered CPC thus obtained was treated with an emulsion of readily available synthetic polymer—PVAC. Three parts by weight of powdered CPC was mixed with one part by weight of PVAC to form a semisolid mass. The agglomerated product was pressed between porcelain tiles into a thin sheet of 2–3-mm thickness and then cut to 2-mm size range. The granular CPC obtained (GCPC) was washed with water, dried at 110°C, and was used for further investigation in column operations.

Batch Studies

Batch studies were carried out by adding a known amount of CPC to a series of flasks containing 100 mL of solution of concentration 50 mg/L of Cu(II), Cd(II), and Zn(II). The solutions were equilibrated in a rotary mechanical shaker at 150 rpm and 25°C. The progress of adsorption during the course of the experiment was determined by taking aliquots after desired contact time, by filtering through Whatman No.1 filter paper, and by assessing the metal concentration. The amount of metal ion adsorbed was computed as

$$\text{Adsorbate uptake } (\%) = \frac{(C_0 - C_t) \times 100}{C_0} \qquad (1)$$

where C_0 and C_t are the initial and at time (t) of the adsorbate concentration, respectively.

The influence of experimental variables, such as contact time, solution concentration, pH, and carbon dose was evaluated. The pH of solution was adjusted by using 0.1 N NaOH or 0.1 N HCl. The concentration of metal ions were determined by atomic absorption spectrophotometer, Analytic Jena (AJ) Vario 6.

Column Studies

Because the volume of wastewater to be treated is usually large, continuous flow systems are preferred over batch process. Column experiments were performed with GCPC in a single fixed bed down flow column of 3 cm internal diameter and 60 cm height. The influence of bed height and flow rate were evaluated by column studies to establish the capacity of carbon for the removal of Cu(II), Cd(II), and Zn(II) from solutions. For these experiments, the initial concentration was kept at 20 mg/L and the initial pH were adjusted to 6.0. The flow rates were maintained constant as required by experimental conditions. The flow rates were maintained constant by checking at intervals of 10 min. Aliquots were collected at 10 min intervals and were analyzed for residual metal-ion concentration. Percolation of solution was stopped as soon as the concentration of Cu(II), Cd(II), and Zn(II) reached 3.0, 2.0, and 5.0 mg/L, respectively.

Desorption Studies

Desorption experiments were carried out by using 1.0 M HCl. After complete desorption of the metal ion, the carbon bed was repeatedly washed with water to remove the free acid. Then 50 mL of 0.5 M NaOH was percolated through the carbon bed at a flow rate of 5 mL/min to remove any residual acidity. The carbon bed was finally washed with water, and the next cycle of operation of removal of metal ion from solution was performed. The process of adsorption of metal ion and regeneration of carbon bed was repeated until the carbon was exhausted completely.

RESULTS AND DISCUSSION

The physicochemical characteristics of powder and agglomerated CPC are summarized in Table 1. The high surface area and good ion-exchange capacity reveal that the carbon is suitable for the removal of heavy metals from water and wastewater. The agglomerated carbon GCPC possesses similar properties as that of the corresponding powder form. Because it possesses high density, it was evaluated for its applicability in column operations.

Batch Studies

Effect of Initial Concentration and Contact Time

The effect of initial concentration and contact time on the removal efficiency of Cu(II) by CPC are shown in Fig. 1. It is evident from the figure

Table 1. Physicochemical characteristics of CPC.

S. no.	Characteristics	CPC	GCPC
1	Moisture content (%)	8.9	8.4
2	Ash content (%)	2.04	2.17
3	Matter soluble in water (%)	1.26	0.98
4	Matter soluble in acid (%)	3.04	2.18
5	pH	8.2	8.5
6	Decolorizing power (mg/g)	30	26
7	Ion exchange capacity (meq/g)	2.34	2.27
8	Iron content (%)	0.048	0.026
9	Phenol number	134	149
10	Apparent density (g/cm^3)	0.19	0.52
11	Surface area (m^2/g)	877	826

that the removal of metal ion is found to increase with decrease in its initial concentration. Increase in contact time increases the uptake of metal ions. Initially, the rate of uptake is very fast. However, equilibrium is attained in 3 hr in the case of Cu(II). Similar studies done with Cd(II) and Zn(II) also showed an equilibrium time of 3 hr.

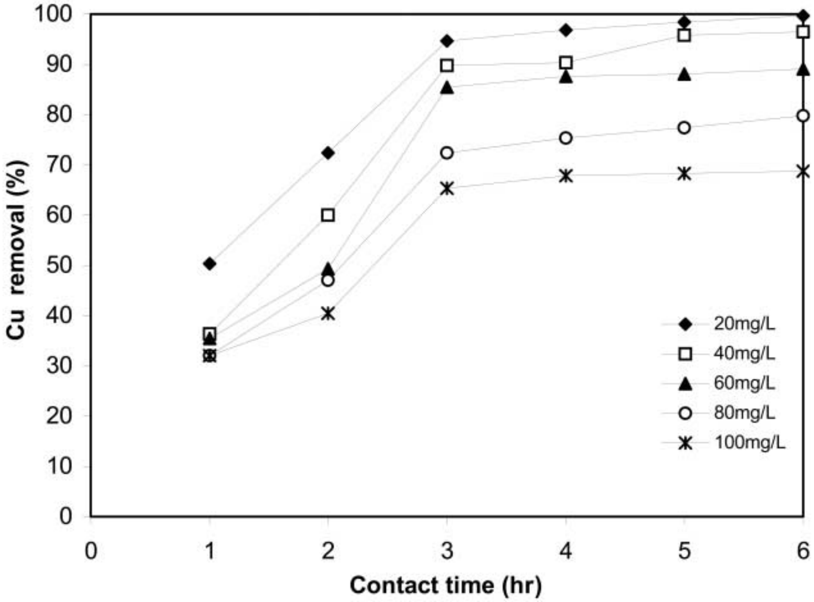

Figure 1. Effect of initial concentration and contact time on adsorption of Cu(II) by CPC.

Effect of pH

Figure 2 represents the effect of pH on the adsorption of Cu(II), Cd(II), and Zn(II) on CPC. It is evident, from the figure, that sorption is very low at acidic pH and increases with increasing pH. As the pH of the metal ion solution increases from 3.0 to 9.0, the removal of copper, cadmium, and zinc increases from 70.8% to 97.6%, 68% to 93.2%, and 54.1% to 83.1%, respectively. The results also demonstrate that adsorption is the major removal step for metal ions, at least in the less than neutral pH region. In the alkaline region, although the extent of metal removal overlaps that by chemical precipitation, it is not certain if precipitation is the major metal removal mechanism. Since chemical precipitation always results in a removal at the multilayer quantities, the results appear to imply adsorption as the sole removal mechanism, even at the alkaline pH region.[9] But generally metals precipitate at higher alkaline pH range of 8–10. So, for the present investigation, an initial pH of 6.0 was selected for further studies. As the pH increases, the adsorbent surface becomes more and more negatively charged and the adsorption of M^{2+} and MOH^{+} species are favored.

The metal removal is assumed to be due to formation of surface–metal complexes[10] of $SO–M^{2+}$ or $SO–MOH^{+}$ suggesting electrostatic attraction mechanism during adsorption. Corapcioglu and Huang[9] studied the

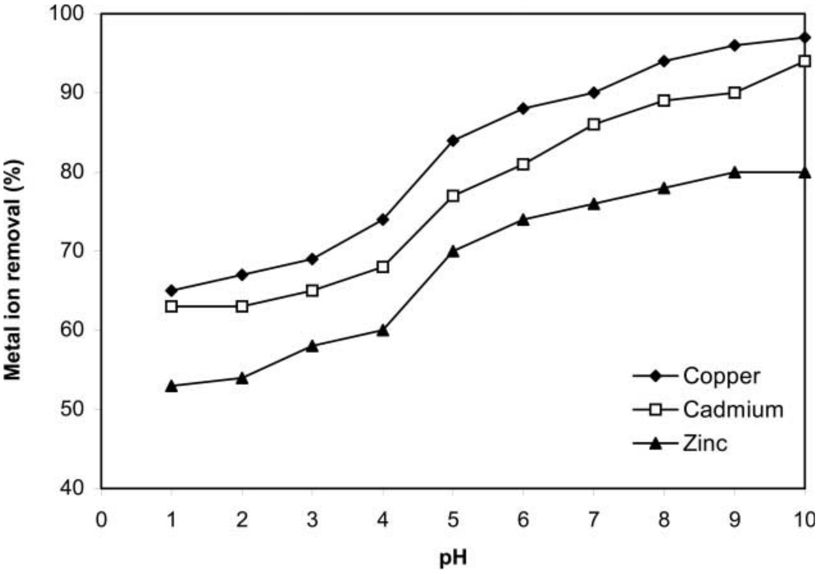

Figure 2. Effect of pH on adsorption of metal ions by CPC.

adsorption of several metal ions by the activated carbon from Nuchar, Filtrasorb, and Darco. It was reported that the adsorption increased with the solution pH. Copper adsorption increased significantly from pH 3 to 5. Similarly, it was observed by Marzel et al.[11] that sorption of cadmium and zinc on activated carbon increased from 0% to 100% over a narrow pH range (4.5–8.0). As the surface charge density decreases with an increase in the solution pH, the electrostatic repulsion between the positively charged metal ions and the surfaces of the activated carbon is reduced, which causes more adsorption. More importantly, higher pH can result in the higher adsorption of positively charged metal ions due to the formation of surface complex reactions.[10]

Effect of Carbon Dose

Figure 3 shows the effect of carbon dosage on Cu(II), Cd(II), and Zn(II) removal from their aqueous solutions. In all the cases, the removal of metal ions increased with an increase in carbon dose and attained complete removal after a particular level that differed with each metal. Thus, 0.4, 0.6, and 0.8 g/L are required for the quantitative removal Cu(II), Cd(II), and Zn(II), respectively. The increase in removal efficiency is due to the availability of more surface functional groups at higher carbon doses.

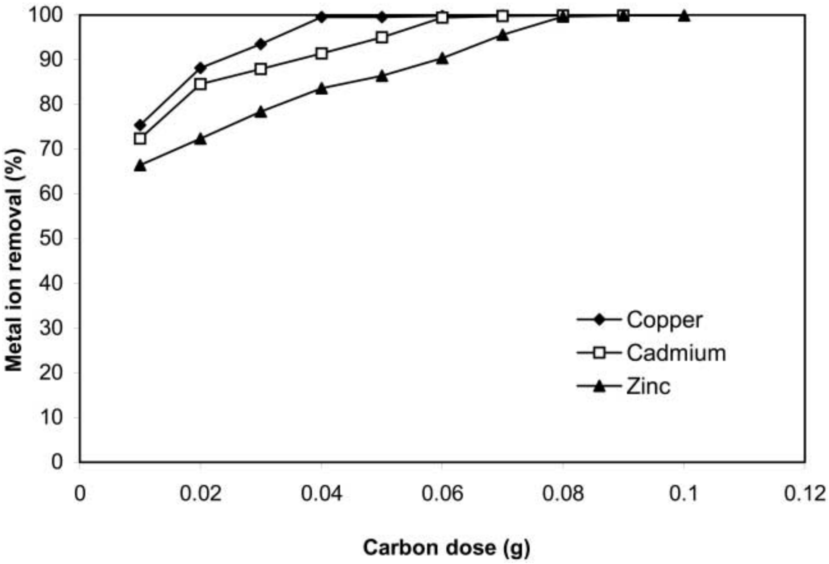

Figure 3. Effect of carbon dose on adsorption of metal ions by CPC.

Freundlich Adsorption Isotherm

Inspection of an adsorption isotherm is a relatively simple method of assessing the feasibility of using activated carbon for a particular application. The uptake of metal ions by CPC has been evaluated in accordance with the linearized form of Freundlich isotherm equation[12]

$$\log X/M = \log K + \frac{1}{n \log C_e} \tag{2}$$

where X is the amount of adsorbate adsorbed, M is the weight of carbon, X/M is the concentration in the adsorbed phase, i.e., the amount of adsorbate adsorbed per unit weight of carbon, C_e is the equilibrium concentration of adsorbate in solution after adsorption, K and n are constants. The straight-line nature of Fig. 4 for Cu(II) indicates that the adsorption process follows the Freundlich type. Similar straight-line plots were obtained for Cd(II) and Zn(II) also. The "*k*" values and sorption intensities ($1/n$) established from the intercept and slope are furnished in Table 2. In any case, isotherms can

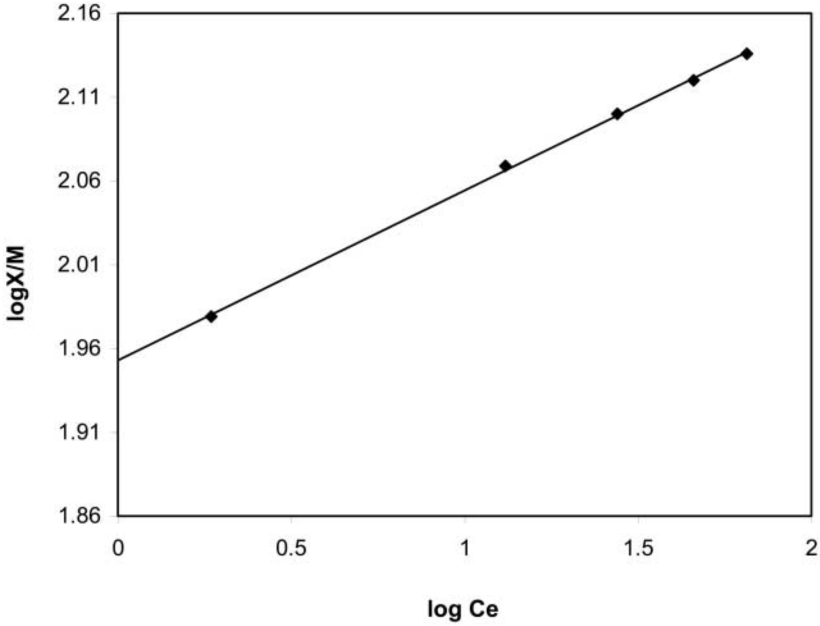

Figure 4. Freundlich isotherm for adsorption of Cu(II) on CPC.

Table 2. Intercept and slope values of Freundlich isotherm for CPC.

S. no.	Metal ion	Slope $1/n$	Intercept k
1	Cu(II)	0.10	89.7
2	Cd(II)	0.08	58.8
3	Zn(II)	0.08	42.6

serve only as a guideline and cannot give accurate scale-up information for the treatment when using columns due to existence of nonequilibrium conditions.

Kinetics of Adsorption

There are essentially three consecutive mass transport steps associated with the adsorption of a solute from solution by porous adsorbents. The first step, bulk transport of solute in the solution phase, usually is rapid because of mixing and convective flow. The second step, film transport involves diffusion of the solute through a hypothetical "film" or hydrodynamic boundary layer. The third step, termed intraparticle diffusion or pore diffusion, involves the transport of the adsorbate from the particle surface into the interior sites by diffusion within the pore-filled liquid and migration along the solid surface of the pore. Film and pore transport are, thus, the major factors controlling rates of adsorption from solution by porous adsorbents.

Kinetic studies for the removal of metal ions by CPC were carried out by using the Lagergren model.[13]

$$\log(Q_e - Q) = \log Q_e - \left(\frac{K_{ad}}{2.303}\right)t \tag{3}$$

where Q and Q_e are the amounts of metal ion adsorbed (mg/g) on the carbon at time, t (min) and at equilibrium time respectively, and K_{ad} is the rate constant of adsorption (min^{-1}). Figure 5 shows the plot of $\log(Q_e - Q)$ against contact time for Cu(II).

The straight line nature of the plot confirms the adherence to Lagergren equation. Similar straight line plots for Cd(II) and Zn(II) suggest that the removal of metal ions by adsorption follows the first-order kinetics. The rate constants K_{ad} calculated from the slope of the linear plots for the metal ions studied are presented in Table 3. These values are comparable with those reported in the literature.[14,15]

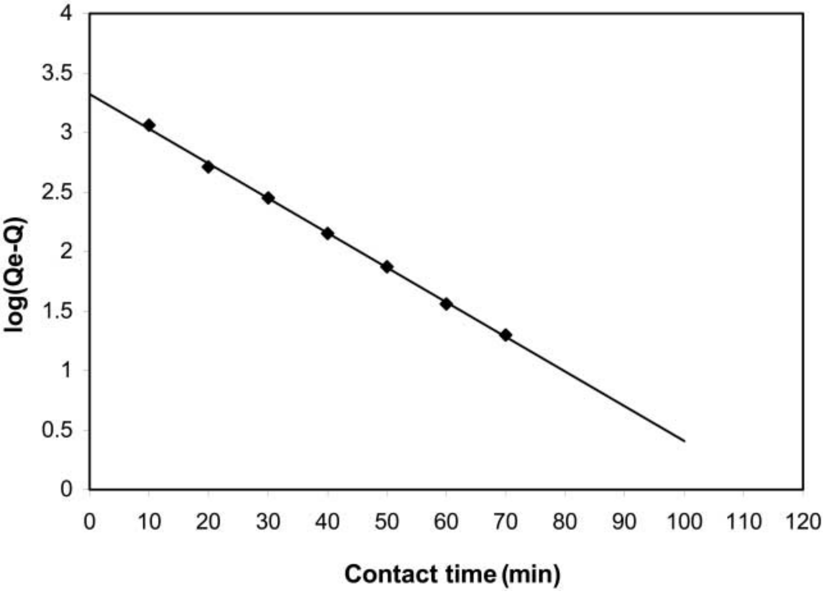

Figure 5. Lagergren plot for the adsorption of Cu(II) on CPC.

Effect of Anions

The influence of chloride and sulfate ions on the adsorption of Cu(II) is shown in Fig. 6. It is evident from the figure that there is no significant influence of both the anions up to a concentration of 3000 mg/L. However, there is slight reduction in adsorption efficiency of CPC when the concentrations of the anions were raised to 5000 mg/L. The effect was more pronounced in the case of Cd(II) and Zn(II) adsorption. Chloride appears to retard the adsorption efficiency much more than sulfate in all the cases. This may be

Table 3. Lagergren rate constant for adsorption of metal ions.

S. no.	Metal ion	Adsorption rate constant K_{ad} (min^{-1})
1	Cu(II)	2.914×10^{-2}
2	Cd(II)	2.704×10^{-2}
3	Zn(II)	2.482×10^{-2}

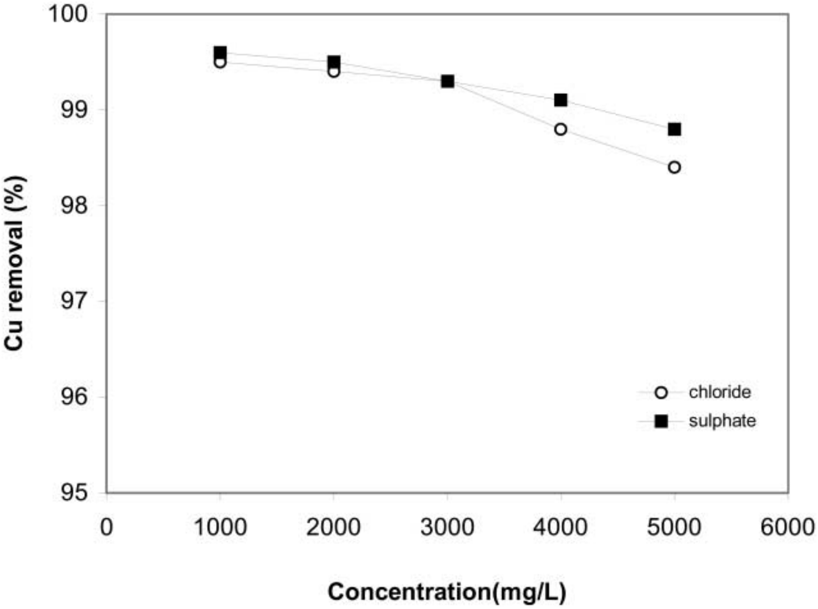

Figure 6. Effect of anions on Cu(II) adsorption by CPC.

due to the formation of stable complexes of metals with anions such as those of metal–chloro complexes, which probably inhibit the adsorption process. The formation of metal complexes would result in the accumulation of negative charges, which induces repulsion for the adsorption of metal–ion pairs.[16]

Column Studies

To evaluate the efficiency of GCPC for treating large volumes of water, column studies were performed. The optimum conditions obtained from batch studies were applied for the treatment of metal ions in the column studies. Thus, the pH was maintained at 6.0 for the metal–ion solutions.

Effect of Flow Rate

Experiments were conducted by using metal ion concentration of 20 mg/L, percolating at different flow rates of 15, 20, and 25 mL/min at a constant bed depth of 15 cm. Aliquots were collected at 10 min intervals and were analyzed for residual metal–ion content. The breakthrough plot of

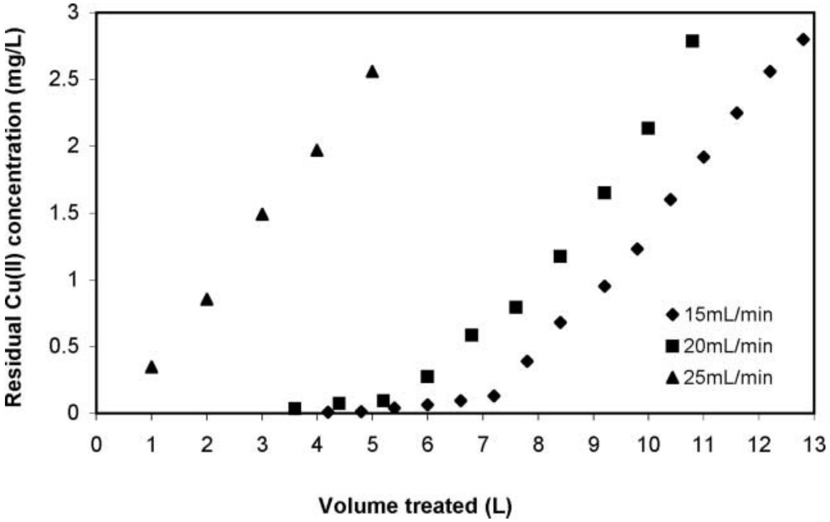

Figure 7. Breakthrough curves for Cu(II) adsorption on CPC at different flow rates and constant bed depth.

Cu(II) obtained at different flow rates is shown in Fig. 7. It can be seen from the plots that as the flow rate increases, the service times are shortened and, hence, could treat less volume. This is due to the decreased retention time of the metal ions percolating through the column at higher flow rates. Similar results were obtained for experiments with Cd(II) and Zn(II) solutions also.

Effect of Bed Depth

Experiments were conducted by using metal–ion solutions with initial concentration of 20 mg/L, percolating through bed depths of 10, 15, 20, and 25 cm at a flow rate of 20 mL/min. Each 200 mL fraction of the effluent was collected separately and was analyzed for the metal–ion content. The breakthrough plots for Cu(II) are presented in Fig. 8. From the figure, it is noted that as the bed depth increases the volume of water treated also increases. When considering the optimal flow rate of 20 mL/min and a bed height of 20 cm, the uptake of metal ions by 56.7 g GCPC was found to be 264 mg of Cu(II), 192 mg of Cd(II), and 176 mg of Zn. This corresponds to removal of 4.65 mg/g, 3.38 mg/g, and 3.1 mg/g of Cu(II), Cd(II), and Zn(II), respectively.

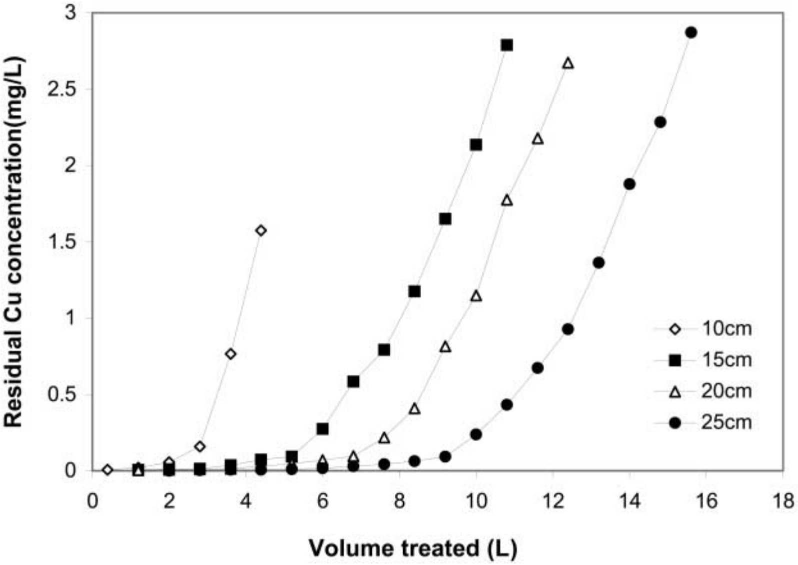

Figure 8. Breakthrough curves for Cu(II) adsorption on CPC at different bed depths and constant flow rate.

Bed-Depth Service Time Analysis

The procedure described by Hutchins[17] for the use of breakthrough curves in the design of activated carbon columns is designated as bed-depth service time (BDST) analysis.

According to the BDST model described by Hutchins[17]

$$\ln[(C_0 - C_B) - 1] = \left(\frac{KN_0Z}{V}\right) - KC_0t \tag{4}$$

where t is the service time (hr), the time required for the effluent to reach a specific breakthrough concentration C_B, K is the adsorption rate constant ($m^3/kg\,hr$), Z is the depth of adsorbent (m), V is the hydraulic loading rate (or) linear flow velocity of wastewater passing through the adsorbent (m/hr), C is the concentration of adsorbate in wastewater, N is the residual adsorbing capacity per unit volume of bed (kg/m^3). At $t = 0$, $N = N_0$, and at $Z = 0$, $C = C_0$ (influent concentration).

Equation (4) can be expressed as

$$t = aZ + c \tag{5}$$

where t is the service time in minute up to breakthrough concentration, a is the slope of BDST graph $a = N_0/C_0V$, Z is the bed depth (cm), and c is the ordinate intercept

$$c = \frac{-1}{C_0K} \ln\left[\left(\frac{C_0}{C_B}\right) - 1\right] \tag{6}$$

Many experimental factors can influence the numerical values of a and c. As such, bench scale experiments typically are conducted to define the experimental conditions under which the most favorable values of a and c are obtained. Thereafter, pilot scale experiments are conducted to define a BDST equation, which can be used in actual design calculations. After a BDST curve has been established, Eq. (5) can be modified for changes in flow rate and feed concentration.

The plot obtained by performing BDST analysis for Cu(II) is represented in Fig. 9. Similar straight-line plots were obtained for Cd(II) and Zn(II) also. The linear relationship indicates that when all the other parameters were maintained constant, the volume of effluent treated up to breakthrough is a direct function of bed depth (depth of carbon). This technique provides carbon

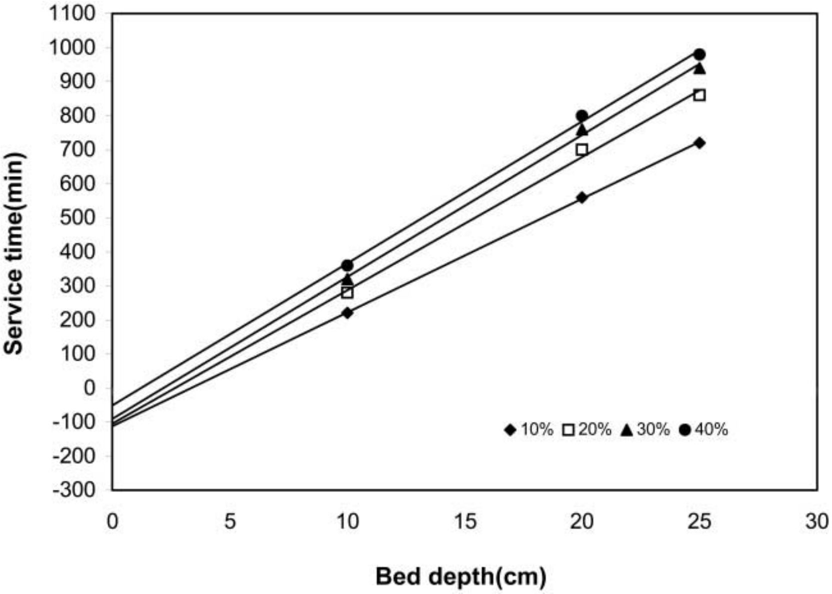

Figure 9. BDST plot for Cu(II) adsorption on CPC.

dosage based on a single fixed bed. The actual carbon usage rate will be lower for beds in series.

Desorption Studies

When activated carbon becomes exhausted or the effluent from the activated carbon bed reaches the maximum level imposed, the spent carbon must be processed to remove the adsorbate, thereby regenerating the carbon for subsequent use. To assess the practical utility of the adsorbent, studies were undertaken on column operation. The results of the desorption studies of Cu(II) is shown in Fig. 10. It is observed that 1000, 1200, and 900 mL of 1.0 M HCl was sufficient for the complete desorption of Cu(II), Cd(II), and Zn(II) from GCPC, respectively. The first aliquot of 100 mL elutes almost 50% of the adsorbed metal ion, and the rest gets desorbed progressively in the following aliquots. The process of adsorption, desorption of metal ion, and regeneration of carbon bed was repeated above until the carbon was exhausted completely. The results of the recycling studies is shown in Fig. 11, for Cu(II). It was found that the capacity of GCPC remains unaffected, up to five cycles of operation for the metal ions. The elution of metal ions with

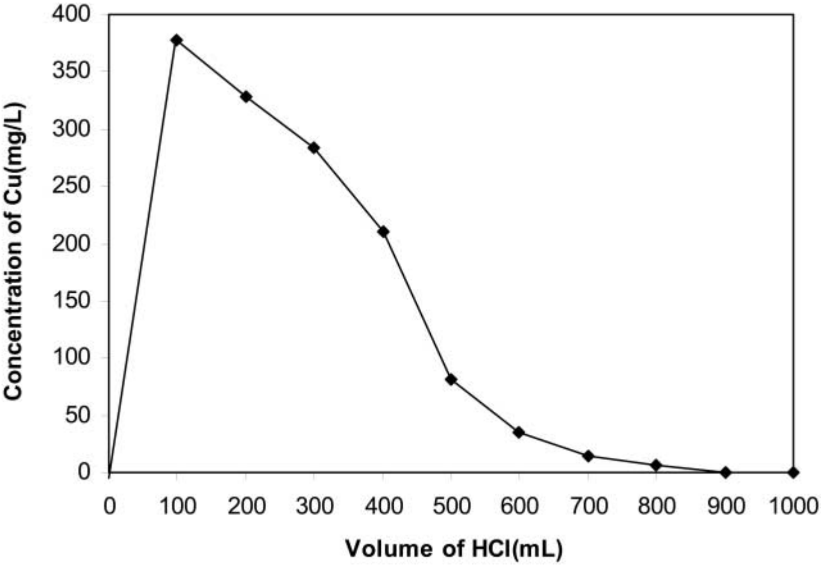

Figure 10. Desorption of Cu(II) from CPC by HCl.

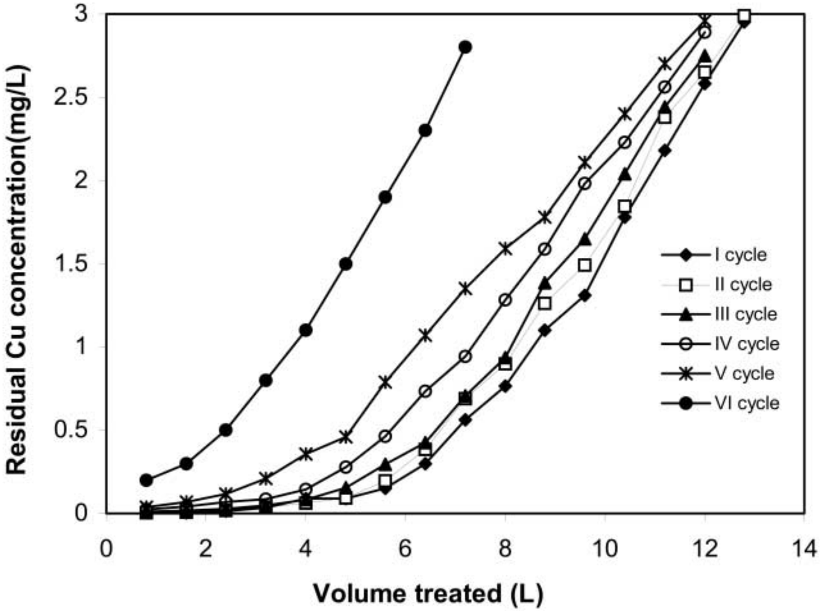

Figure 11. Regeneration cycles of CPC for adsorption of Cu(II).

HCl may be due to the lowering of pH, which would modify the polarity on the surface on the carbon, thereby reducing the force of attraction between the adsorbate and adsorbent.

Removal of Heavy Metals from Industrial Effluents

The suitability of CPC and GCPC for removing, copper, cadmium, and zinc from effluent of a metal-finishing workshop was examined. The composition of the effluent is given in Table 4.

Batch experiments were carried out by using 100 mL of the effluent adjusted to pH 6.0 and 0.5 g of carbon, and equilibrating for a period of 3 hr. At the end of equilibration, the concentration of Cu(II), Zn(II), and Cd(II) were established as described earlier. The results are presented in Table 5. It can be seen that the efficiencies of CPC and GCPC are comparable and very much superior with that of commercial carbons.

Column studies were performed with the effluent adjusted to pH 6.0 and by packing the column with GCPC to a bed depth of 35 cm. The effluent

Table 4. Characteristics of metal polishing industrial effluent.

S. no.	Parameter	Value
1	pH	1.1
2	Chloride	1,742
3	Nitrate	919
4	Phosphate	294
5	Sulfate	3,282
6	Nickel	1.27
7	Total chromium	179
8	Cadmium	204
9	Copper	501
10	Zinc	130

Note: All values are in mg/L, except pH.

was percolated through the carbon bed at a flow rate of 20 mL/min, and each 100 mL of the aliquot was collected separately and analyzed for the metal ions. The results of the column studies are presented in Fig. 12 for the removal of copper, cadmium, and zinc from the effluent. Cu(II) and Cd(II) are adsorbed preferentially over zinc. It is found that the volume of effluent treated is less compared with that of single aqueous metal ion solution. This is due to the high concentration of all metal ions in the effluent. The efficiency of the carbon treatment could be further improved by pretreatment of the effluent. However, this was not carried out in the present investigation. The desorption with 1.0 M HCl, and recycling studies were carried out as for single aqueous solutions. Complete desorption of metal ions from the carbon was possible, but separation of the desorbed Cu(II), Cd(II), and Zn(II) was not attempted. However, the mixture of these ions can be concentrated and then can be separated by precipitation or by other conventional methods. The carbon was effective for only two cycles of operation as against five cycles with that of aqueous metal solutions. The reduced efficiency of the carbon when applied to effluent is due to the presence of other ions that interfere and compete for the active sites on the carbon during adsorption process.

The adsorption efficiency of metal ions by GCPC follows the order $\mathrm{Cu} > \mathrm{Cd} \gg \mathrm{Zn}$. The higher adsorption capacity of Cu(II) and Cd(II) may be due to their small radii and, thereby, easy penetration into smaller pores in the carbon and, thus, having greater access to the surface of the carbon. Similar results have been reported by Johns et al.[18]

Table 5. Copper, cadmium, and zinc removal from industrial effluents by batch process.

		Cu(II)		Cd(II)		Zn(II)	
S. no.	Carbon type	Final concentration (mg/L)	Removal (%)	Final concentration (mg/L)	Removal (%)	Final concentration (mg/L)	Removal (%)
1	CPC-K	2.4	99.5	6.8	96.6	29.8	77.1
2	GCPC-K	10.6	97.8	13.2	93.5	37.4	71.3
3	CAC[a]	312.2	37.6	149.6	26.7	108.3	17.0
4	GCAC[a]	387	22.7	170.9	16.3	119.8	8.0

[a] Commercial carbons.

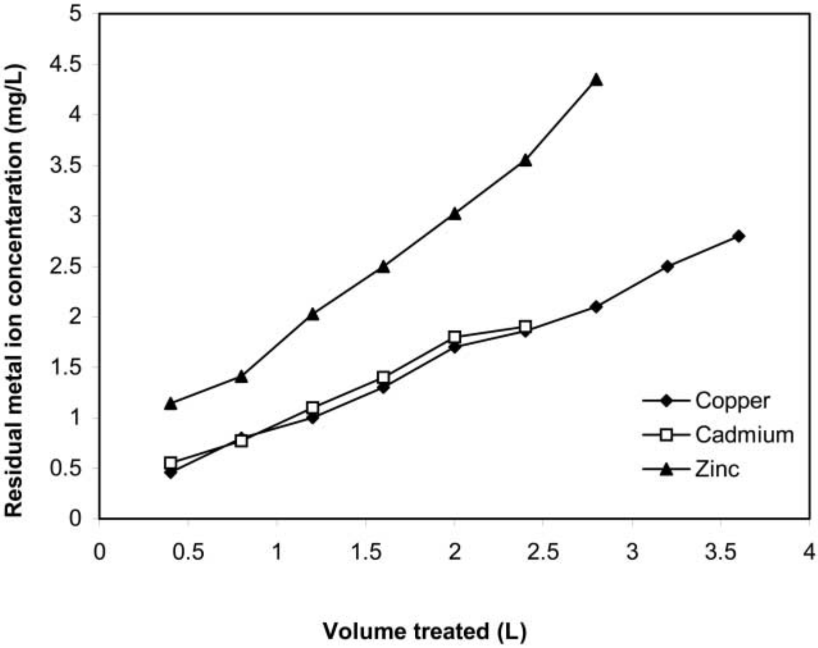

Figure 12. Removal of metal ions from industrial effluent by CPC column operation.

CONCLUSION

The present investigation has revealed that activated carbon prepared by KOH treatment of coconut coir pith possessed desirable characteristics as an adsorbent for the effective removal of contaminants of cationic nature such as heavy metals.

In the batch mode operation, efficiency of adsorption of metal ions studied viz. Cu(II), Cd(II), and Zn(II) on CPC increased with increase of contact time, pH, carbon dose, and decrease of metal–ion concentration. The adsorption was found to follow the Freundlich isotherm. The kinetics of adsorption followed Ist order. The Lagergren rate constants for Cu(II), Cd(II), and Zn(II) were in the range of 2.5×10^{-2}–$3 \times 10^{-2}\,\text{min}^{-1}$. The presence of common anions such as chloride and sulfate did not affect the removal of metal ions up to 3000 mg/L. The increase in concentration of anions above 3000 mg/L, however, retarded the adsorption.

Column studies carried out on the metal ions revealed that the adsorption capacity of the column increased with decrease in flow rate and with increase in bed depth. Column studies with GCPC showed a breakthrough capacity of 264, 192, and 176 for Cu(II), Cd(II), and Zn(II), respectively.

Use of 1.0 M HCl was found to be effective in desorbing quantitatively all the metal ions adsorbed, and the adsorption capacity of the exhausted beds could be effectively restored. The capacity remained unaffected up to five cycles of operation. The BDST analysis revealed linear relationships for all metal ions between bed depth and service time, which could be used successfully for scale-up purposes. The efficiency of CPC and GCPC were evaluated for the treatment of effluent from a metal-polishing industry containing different heavy metals and was found to be superior to that of commercially activated carbon.

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Received April 2003
Accepted April 2004