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Adsorption of Pb(II) by Peanut Hull Carbon from Aqueous Solution

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ABSTRACT

Carbon prepared from peanut hulls (PHC) has been used for the adsorption of Pb(II) over a range of initial metal ion concentrations (10–20 mg/L), agitation times (5–140 minutes), adsorbent dosages (5–100 mg/100 mL), and pH values (1.5–10.0). Adsorption of Pb(II) obeyed the Langmuir isotherm. The applicability of the Lagergren kinetic model has also been investigated. Quantitative removal of 20 mg/L Pb(II) by 0.3 g carbon per liter aqueous solution was observed in the pH range of 3.0 to 10.0. A comparative study with a coal-based commercial granular activated carbon (CAC) showed that the adsorption capacity of PHC was 18 times larger than that of CAC.

INTRODUCTION

Lead is introduced into natural waters from a variety of sources, such as storage batteries, lead smelting, tetraethyllead manufacturing, mining, plating, ammunition, and ceramic and glass industries (1). The toxic action of lead includes spasmic diseases of brain, kidneys, liver, stomach, the intestinal system, anemia, and various damages of the nervous system (2). The tolerance limit for Pb(II) for discharge into inland surface waters is 100 μg/L according to the Indian Standards Institution (ISI) (3). Different methods [such as precipitation, reverse osmosis, ion exchange (4), and cementation (5)] have been used for the removal of Pb(II) from aqueous

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solution. Removal of lead by activated carbon has recently gained industrial importance (6–10). Many reports have appeared on the development of activated carbon from cheaper and readily available materials (11). Activated carbon derived from rice husk (12) and coconut shell (13) has been employed for the removal of heavy metals from aqueous solutions. We have recently reported that activated peanut hull carbon (PHC) was able to remove Cr(VI) (14), Hg(II) (15), and Cd(II) (16) efficiently from aqueous solution. Peanut hull, a waste agricultural by-product, is utilized in India as fuel and manure. The investigation reported here deals with a comparative study of PHC and coal-based commercial granular activated carbon (CAC) for the removal of Pb (II) from aqueous solutions.

EXPERIMENTAL

Preparation of PHC

PHC was prepared as reported before (15, 16). One part of peanut hull was stirred with 1.8 parts by weight of concentrated sulfuric acid and kept in an air oven at 150 ± 5°C for 24 hours. The carbonized material was washed with distilled water to remove the free acid, soaked in 1% sodium bicarbonate solution until effervescence ceased, and finally soaked in 1% sodium bicarbonate solution overnight to remove any residual acid. The material was then washed with distilled water, dried at 105 ± 5°C, and sieved to 0.575 mm size (20–50 mesh ASTM). The coal-based CAC was obtained from Burbidges Company, Bombay, India. It was ground and sieved to the same size. Characteristics of the two carbons, such as bulk density, ash content, moisture, solubility in water (inorganic matter) and 0.25 M HCl (inorganic and organic matter), decolorizing power, iron content, and pH, were determined according to ISI methods (17). Phenol number (18), ion-exchange capacity (19), surface area by the *p*-nitro phenol method (20), and porosity (21) were estimated according to published procedures. Ash analysis was carried out as per standard procedures (22). The characteristics of PHC and CAC are summarized in Table 1. All the chemicals used were of analytical reagent grade and were obtained from B.D.H, E. Merck, and Ranbaxy.

Batch Mode Studies

A stock solution of 1000 mg/L Pb(II) was prepared from $Pb(NO_3)_2$ in water containing 1 mL of concentrated nitric acid to prevent hydrolysis. The stock solution was diluted as required to obtain standard solutions containing 10–50 mg/L Pb(II). Thus, the Pb(II) levels used in this study were 100–500 times the ISI tolerance limit. One hundred milliliters of

TABLE 1
Characteristics of the Carbons

Parameter	Peanut hull carbon	Commercial activated carbon
Bulk density, g/mL	0.63	0.60
Porosity, %	61.7	60.4
Moisture, %	14.14	6.79
Ash, %	2.11	3.84
Solubility in water, %	0.74	1.42
Solubility in 0.25 M HCl, %	2.25	1.56
pH	6.68	8.18
Decolorizing power, mg/g	36.00	77.00
Phenol number	68.00	20.00
Ion-exchange capacity, mequiv/g	0.49	Nil
Surface area, m^2/g	208.00	354.00
Iron, %	0.27	1.43
Ash analysis, %		
SiO_2	19.6	79.6
K_2O	0.58	2.3
CaO	0.56	0.28
MgO	9.8	6.5
P_2O_5	0.1	1.2
Na_2O	78.7	12.1
Fe_2O_3	0.76	4.1

Pb(II) solution of a desired concentration, adjusted to pH 5.0, was taken in reagent bottles of 300 mL capacity, and 100 mg PHC or CAC was added. The pH was adjusted using dilute nitric acid or sodium hydroxide solutions. The solutions were agitated for a predetermined period at 30 ± 1°C in a reciprocating shaker. The carbons were separated by centrifugation at 10,000 rpm, 8600*g*, and the Pb(II) in the centrifugate was analyzed spectrophotometrically (23). The effects of adsorbent dose were studied using 100 mL solution of 20 mg/L Pb(II) and varying amounts of carbon from 5 to 100 mg for PHC and from 50 to 1000 mg for CAC. Adsorption isotherm studies were carried out with different initial concentrations of Pb(II) while maintaining the carbon dose at a constant level. The pH effects were studied using 30 mg PHC or 350 mg CAC and a 100-mL solution of Pb(II) with concentrations ranging from 20 to 50 mg/L for PHC and 10 to 25 mg/L for CAC. Sodium concentrations in the solutions were estimated using a flame photometer. To correct for any adsorption of Pb^{2+} and Na^{+} on the containers, control experiments were carried out without adsorbent, and there was negligible adsorption of either by the container walls.

Desorption studies were carried out as follows. After adsorption experiments with 20 mg/L Pb(II) and 100 mg PHC or CAC, the lead-laden carbons were separated and gently washed with distilled water to remove any unadsorbed Pb(II). Several such spent carbon samples were prepared for both PHC and CAC. They were then agitated with 100 mL hydrochloric acid of various strengths for 3 hours in the case of PHC or for 5 hours in the case of CAC, and the amount of desorbed lead was estimated by analyzing the acid solutions.

All experiments were performed in duplicate, and the mean values are presented. The maximum error was 2%.

RESULTS AND DISCUSSION

Effect of Agitation Time and Initial Concentration

Figure 1 shows the effect of agitation time on the removal of Pb(II) by PHC and CAC. The removal (mg Pb/g carbon) increases with time. The equilibrium time for initial Pb(II) concentrations of 10, 15, and 20 mg/L was approximately 80 minutes for PHC and approximately 240 minutes for CAC. This indicates that the contact time required for maximum Pb(II) removal by PHC is one-third that required by CAC.

Effect of Carbon Dose

Figure 2 shows that the removal of Pb(II) increases with carbon dose for both PHC and CAC. It is clear that for the quantitative removal of Pb(II) from a 100-mL solution of 20 mg/L requires a minimum carbon dosage of 30 mg PHC or 350 mg CAC.

Langmuir Adsorption Isotherm

The Langmuir isotherm was applied for the adsorption equilibrium of both PHC and CAC (24):

$$C_e/q_e = 1/(Q_0 b) + (C_e/Q_0) \tag{1}$$

where C_e is the equilibrium concentration (mg/L), q_e is the amount adsorbed at equilibrium (mg/g), and Q_0 and b are Langmuir constants related to adsorption capacity and energy of adsorption, respectively. Linear plots of C_e/q_e vs C_e show that the adsorption obeys the Langmuir isotherm model for both PHC and CAC (Fig. 3). The correlation coefficients were 0.9969 and 0.9975 for PHC and CAC, respectively. Q_0 and b were determined from the Langmuir plots and found to be 210.53 mg/g and 0.475 L/mg, respectively, for PHC, and 11.55 mg/g and 0.072 L/mg, respectively, for CAC. The ratio of Q_0 values of PHC and CAC is 18.23. The Langmuir

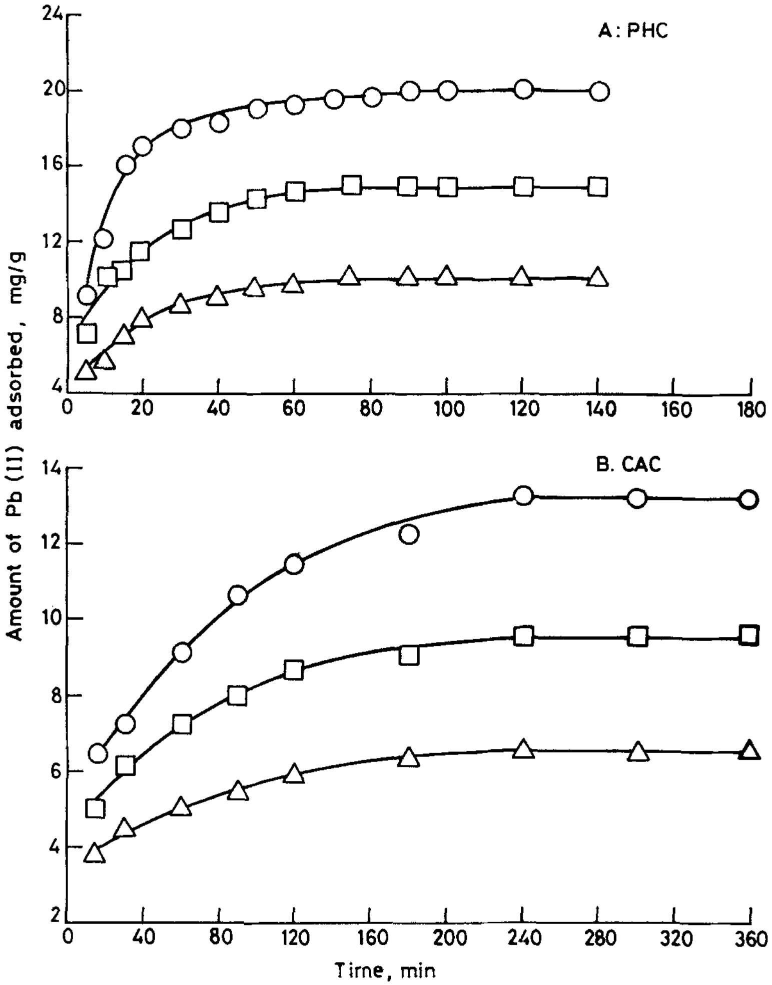

FIG. 1 Effect of agitation time on the adsorption of Pb(II). Pb(II) concentration = (○) 20 mg/L, (□) 15 mg/L, (△) 10 mg/L; Carbon dose for both PHC and CAC = 100 mg/100mL; pH 5.0; A = PHC; B = CAC.

isotherm can be represented in terms of a dimensionless constant separation factor or an equilibrium parameter, R_L, which is defined by $R_L = 1/(1 + bC_0)$, where b is the Langmuir constant and C_0 is the initial concentration of Pb(II) (25). The R_L value indicates the type of isotherm. According to McKay et al. (25), R_L values between 0 and 1 indicate favorable adsorp-

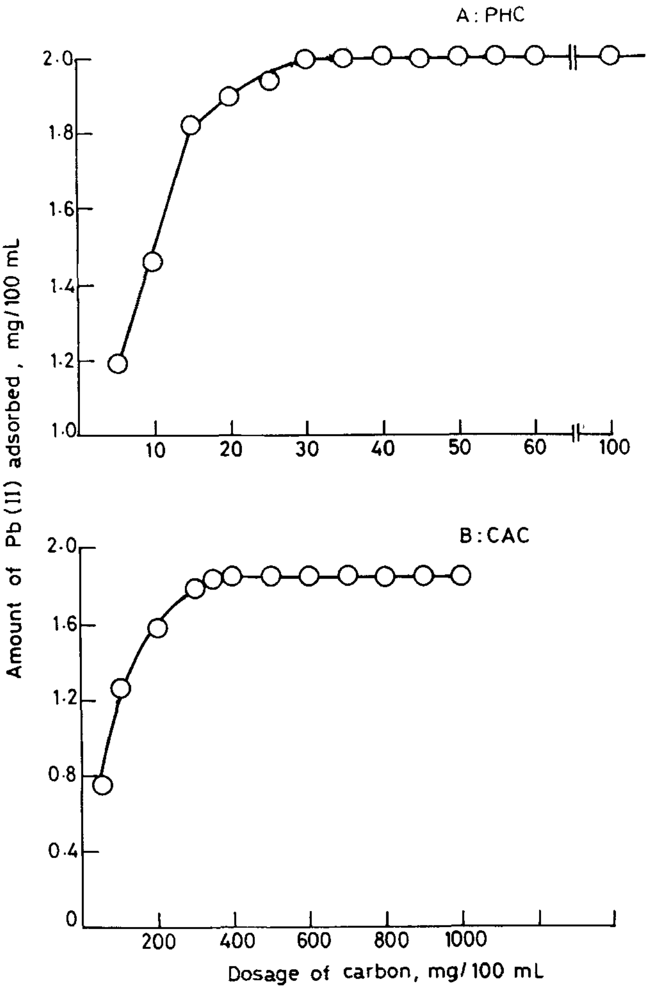

FIG. 2 Effect of carbon dosage on the adsorption of Pb(II). Pb(II) concentration = 20 mg/L; pH 5.0; volume of solution = 100 mL; A = PHC, agitation time = 3 hours; B = CAC, agitation time = 5 hours.

tion. For both PHC and CAC, R_L values were found to be between 0 and 1 at all concentrations of Pb(II) studied.

Adsorption Kinetics

The kinetics of Pb(II) adsorption on both PHC and CAC follows the first-order rate expression given by Lagergren (24):

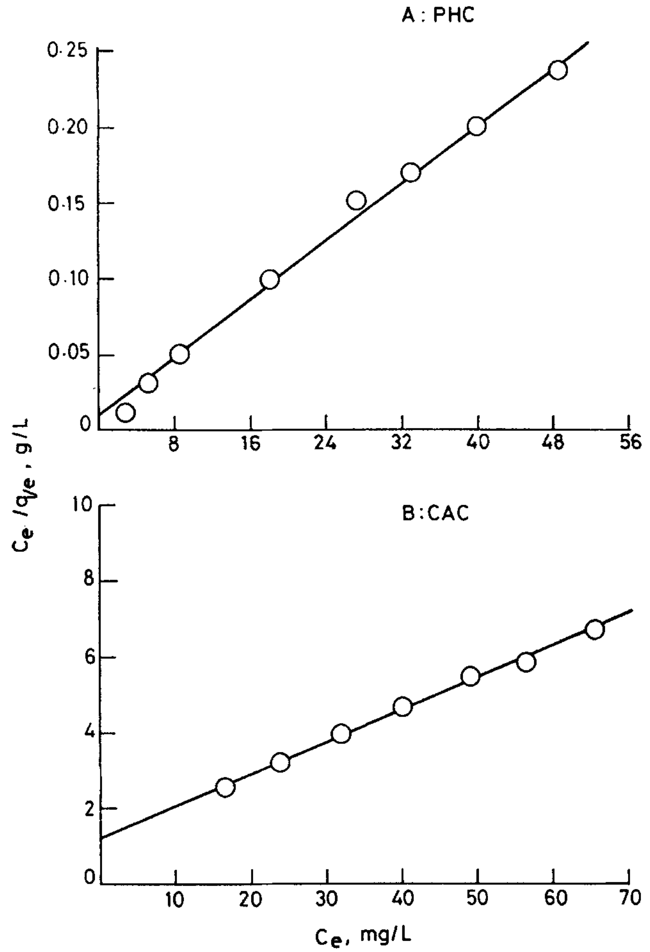

FIG. 3 Langmuir plots for the adsorption of Pb(II). Pb(II) concentration = 40–100 mg/L; pH 5.0; agitation time = 24 hours; A = PHC, Carbon dose = 30 mg/100 mL; B = CAC, Carbon dose = 350 mg/100 mL.

$$\log(q_e - q) = \log q_e - k_{ad}t/2.303 \qquad (2)$$

where q and q_e are the amounts of Pb(II) adsorbed (mg/g) at time t (minutes) and at equilibrium time, respectively, and k_{ad} is the rate constant of adsorption (1/min). Linear plots of $\log(q_e - q)$ vs t show the applicability of the above equation for both PHC and CAC (Fig. 4). The k_{ad} values calculated from the slopes of the plots for PHC are 5.03×10^{-2}, 5.63×10^{-2}, and 6.02×10^{-2} 1/min for the initial Pb(II) concentrations of 10,

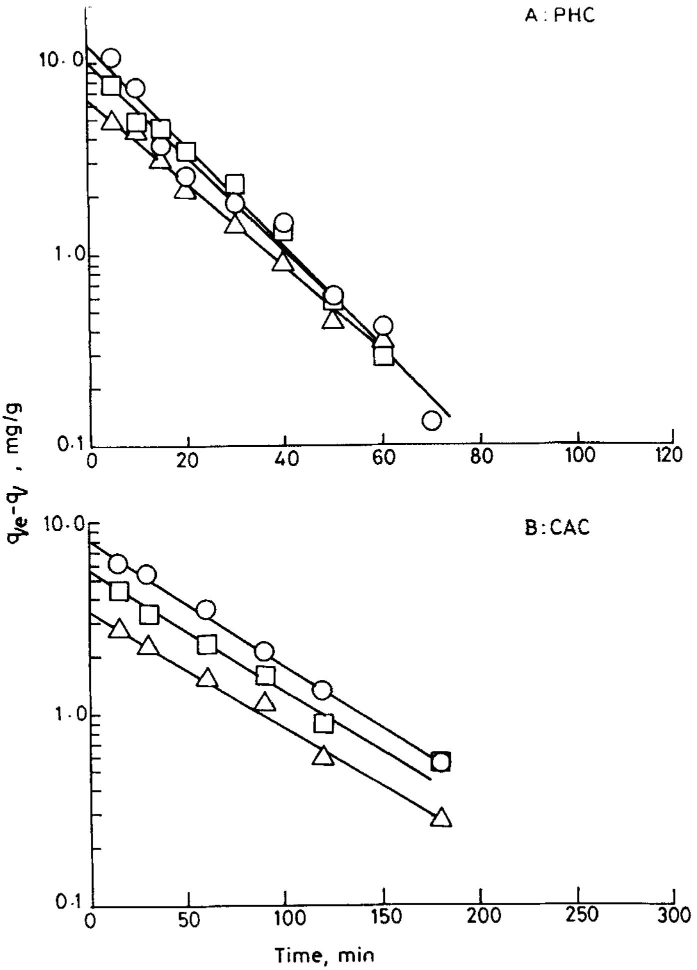

FIG. 4 Lagergren plot for the adsorption of Pb(II). Pb(II) concentration = (○) 20 mg/L, (□) 15 mg/L, (△) 10 mg/L; A = PHC; B = CAC.

15 and 20 mg/L, respectively; the corresponding values for CAC are 1.40 $\times$ 10^{-2}, 1.43 $\times$ 10^{-2}, and 1.50 $\times$ 10^{-2} 1/min, respectively.

Effects of pH

One of the conventional methods of removing metals from water is the precipitation of metals as metal hydroxides by using alkali. This method is limited because metals cannot be completely removed from solution owing to solubility product of metal hydroxides. Hence comparison is made between adsorption and precipitation as a metal hydroxide. Effects of pH on Pb(II) removal in the presence and absence of adsorbent are shown in Figs. 5 and 6 for PHC and CAC, respectively, for different Pb(II) concentrations. Clearly, Pb(II) adsorption by both carbons is much more efficient than metal hydroxide precipitation for the different concentrations of Pb(II) studied. Pb(II) removal by both carbons increases with an increase in pH and attains a maximum in the pH range 3 to 10 for PHC and 4 to 10 for CAC for the different concentrations of Pb(II) studied. In the absence of any adsorbent, precipitation of metal hydroxide starts only at pH 6.2. Even at higher pH values, such as 10, the percent removal by precipitation is only 25 for a Pb(II) concentration of 20 mg/L, whereas for adsorption removes 98% Pb(II) for PHC and 78% Pb(II) for CAC for the same Pb(II) concentration even at such low pH values as 3.0 if sufficient absorbent is provided.

The influence of pH on Pb(II) removal can be explained on the basis of an electrostatic interaction model (26). As the pH increases, the number

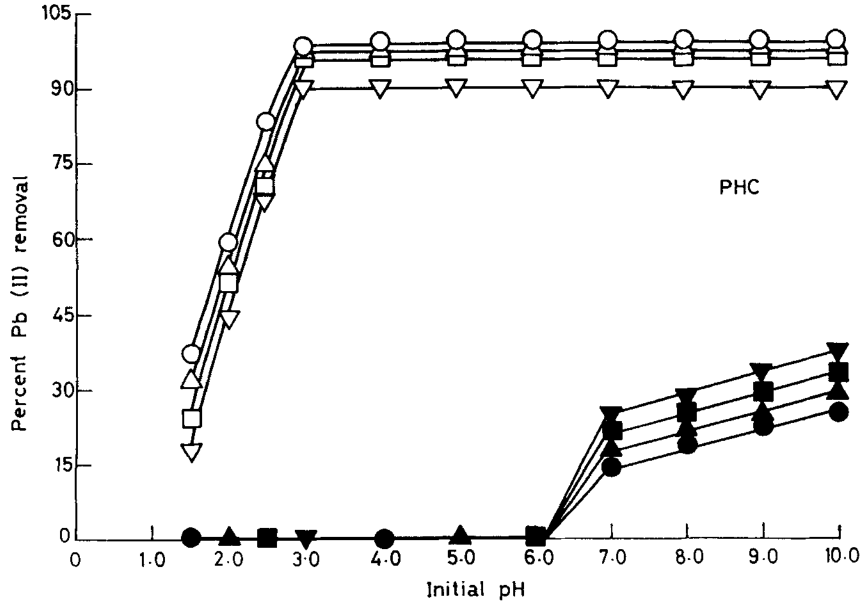

FIG. 5 Effects of pH on the removal of Pb(II) by PHC at different Pb(II) concentrations: (○) 20 mg/L, (△) 30 mg/L, (□) 40 mg/L, (▽) 50 mg/L; Carbon dose = 30 mg/100mL; agitation time = 3 hours. Lead hydroxide precipitation = (●) 20 mg/L, (▲) 30 mg/L, (■) 40 mg/L, (▼) 50 mg/L.

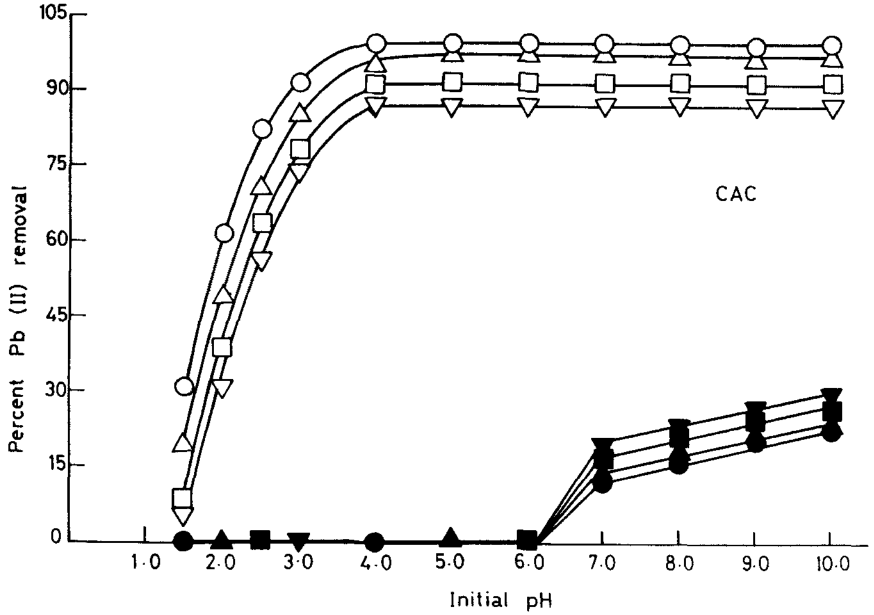

FIG. 6 Effects of pH on the removal of Pb(II) by CAC at different Pb(II) concentrations: (○) 10 mg/L, (△) 15 mg/L, (□) 20 mg/L, (▽) 25 mg/L. Carbon dose = 350 mg/100 mL; agitation time = 5 hours. Lead hydroxide precipitation = (●) 10 mg/L, (▲) 15 mg/L, (■) 20 mg/L, (▼) 25 mg/L.

of positively charged sites on the carbon surface decreases and the electrostatic attraction of positively charged lead species, Pb^{2+} and $PbNO_3^+$ (27), is favored. In alkaline medium, removal of positively charged lead species, $PbOH^+$ and $Pb_4(OH)_4^{4+}$ (28), and neutral species, $Pb(OH)_2$ and $Pb(NO_3)_2$ (29), is favoured by the negatively charged carbon surface.

The major mechanism of adsorption of Pb(II) seems to be ion exchange. A pure carbon surface is considered to be nonpolar, but in actual practice some carbon–oxygen complexes (C_xO, CO_x, and C_xO_2) are usually present, and they render the surface slightly polar. Since there is no satisfactory method for determining the polar character of the surface quantitatively, the above statement is relative (30–32). The surface oxygen complexes hydrolyze water molecules as shown below:

$$C_xO + 2H_2O \rightarrow C_xOH_2^{2+} + 2OH^- \tag{3}$$

$$CO_x + xH_2O \rightarrow C(OH)_x^+ + xOH^- \tag{4}$$

$$C_xO_2 + H_2O \rightarrow C_xO^{2+} + 2OH^- \tag{5}$$

Since the PHC is prepared upon treatment with H_2SO_4 and $NaHCO_3$, groups such as C_xONa^+, $C_xONa_2^{2+}$, C_xSO_3H, and C_xSO_3Na are also assumed to be present. Na^+ in the above groups are also exchanged with H^+ in the medium as follows:

$$C_xONa^+ + H^+ \rightarrow C_xOH^+ + Na^+ \quad (6)$$

$$C_xONa^+ + 2H^+ \rightarrow C_xOH_2^{2+} + Na^+ \quad (7)$$

$$C_xONa_2^{2+} + 2H^+ \rightarrow C_xOH_2^{2+} + 2Na^+ \quad (8)$$

$$C_xSO_3Na + H^+ \rightarrow C_xSO_3H + Na^+ \quad (9)$$

Figure 7 shows the effect of initial pH on final pH at different concentrations of Pb(II) for PHC. The curve referred to as "blank" was obtained under conditions such that [Pb(II)] = 0. Reactions (3) to (9) contribute

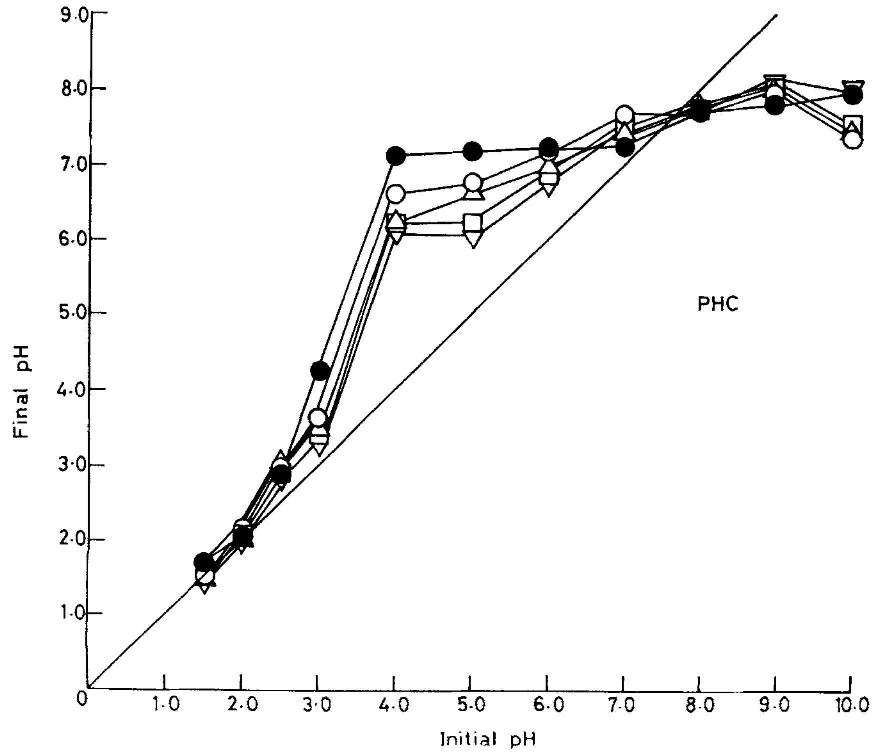

FIG. 7 Effects of pH on final pH on the adsorption of Pb(II) by PHC. Pb(II) concentration = (○) 20 mg/L, (△) 30 mg/L, (□) 40 mg/L, (▽) 50 mg/L, (●) blank [0 mg/L Pb(II)]; carbon dose = 30 mg/100 mL; agitation time = 3 hours.

to an increase in pH in the blank curve. At the same time, Reactions (6) to (9) lead to the release of Na^+. Excess Na^+ was introduced into PHC when it was washed with $NaHCO_3$ to neutralize any free sulfuric acid during the preparation of PHC (see Table 1). When Pb(II) is present in solution, its adsorption will free some H^+ and the pH increase will be lower than in the blank (Eqs. 10, 11, and 14). At the same time, Na^+ will also be released according to Reactions (12), (13), and (15).

$$2C_xOH^+ + Pb^{2+} \rightarrow (C_xO)_2Pb^{2+} + 2H^+ \tag{10}$$

$$C_xOH_2^{2+} + Pb^{2+} \rightarrow C_xOPb^{2+} + 2H^+ \tag{11}$$

$$2C_xONa^+ + Pb^{2+} \rightarrow (C_xO)_2Pb^{2+} + 2Na^+ \tag{12}$$

$$C_xONa_2^{2+} + Pb^{2+} \rightarrow C_xOPb^{2+} + 2Na^+ \tag{13}$$

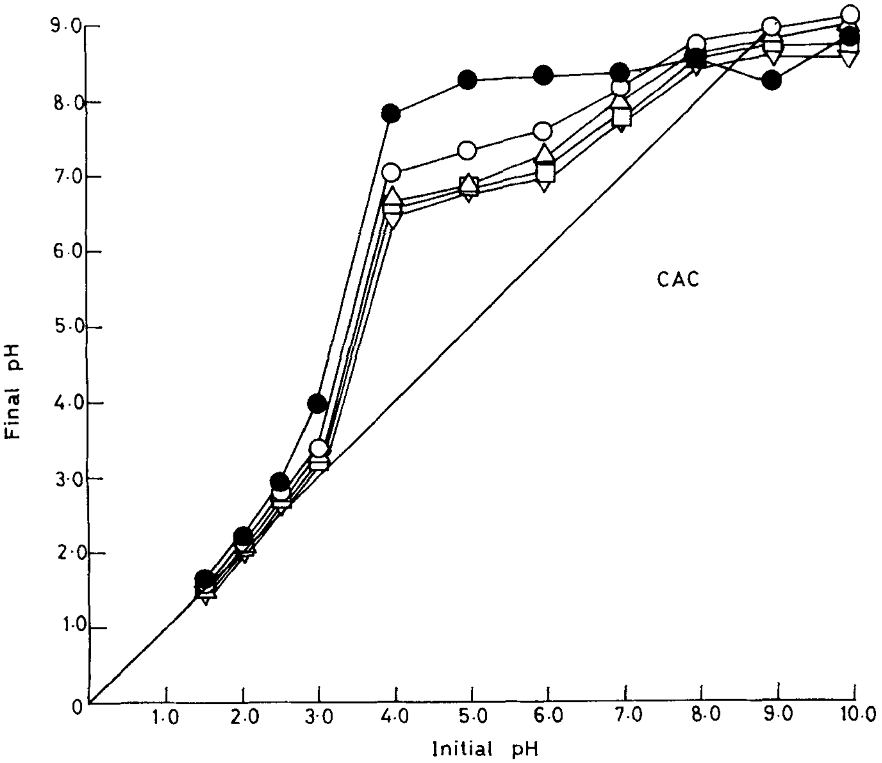

FIG. 8 Effects of pH on final pH on the adsorption of Pb(II) by CAC. Pb(II) concentration = (○) 10 mg/L, (△) 15 mg/L, (□) 20 mg/L, (▽) 25 mg/L, (●) blank [0 mg/L Pb(II)]; carbon dose = 350 mg/100 mL; agitation time = 5 hours.

$$2C_xSO_3H + Pb^{2+} \rightarrow (C_xSO_3)_2Pb + 2H^+ \qquad (14)$$

$$2C_xSO_3Na + Pb^{2+} \rightarrow (C_xSO_3)_2Pb + 2Na^+ \qquad (15)$$

Tests performed by agitating 30 mg PHC for 3 hours at an initial pH of 4.0 led to a sodium concentration of 18 mg/L in the remaining liquid. Also, when the Pb(II) concentration in solution is increased from 0 to 50 mg/L, the difference between the final pH value of the test containing Pb(II) and that of the blank increases, as expected.

The sodium release increased from 18 to 43 mg/L when the concentration of Pb(II) was increased from 0 to 50 mg/L. This indicates that apart from exchange of H^+ ions on the adsorbent with Pb(II) ions, significant Na^+ ions were present in the adsorbent and also exchanged with Pb(II) ions.

Since the details of manufacturing and activation processes for CAC are not known, it is not possible to discuss how Na^+ ion was introduced into CAC. Observations on the effect of initial pH on the final pH for CAC were similar to those for PHC (Fig. 8). Hence, it can be said that an ion-exchange mechanism is important in the adsorption processes for both carbons.

DESORPTION STUDIES

Desorption studies help elucidate the nature of adsorption and the recovery of precious metals from wastewaters and the adsorbent. Attempts were made to desorb Pb(II) from spent carbons using hydrochloric acid of various strengths. The percent recoveries of Pb(II) for PHC were 76.5, 91.7, and 100 by 0.05, 0.10, and 0.15 M hydrochloric acid, respectively. In the case of CAC, the corresponding values were 75.0, 82.8, and 100. This is further evidence that ion exchange is involved in the adsorption mechanism and confirms the effects of pH on adsorption that were shown in Figs. 5 and 6.

CONCLUSION

The present study shows that peanut hull carbon is 18 times more efficient for the removal of Pb(II) from dilute aqueous solutions than the same mass of a commercial activated carbon. An ion-exchange mechanism seems to be operative in the adsorption of Pb(II) by both carbons. As the adsorbent is easily prepared from an agricultural waste product, PHC should be useful for the economic treatment of wastewater containing Pb(II).

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