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Adsorption of lead from aqueous solution onto coir-pith activated carbon

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ABSTRACT

Adsorption of lead (Pb(II)) from aqueous solution using H_2SO_4-activated carbon developed from coir pith (CPAC) was studied. Batch experiments were performed to explain the effect of initial pH (pH_0), contact time, adsorbent dose, and temperature on adsorption. A pH_0 of 5.0, a dose of $1\,g\,L^{-1}$ for adsorption at a concentration of Pb of $20\,mg\,L^{-1}$ were the optimal conditions. The adsorption of lead onto CPAC was found to be a gradual process and the quasi-equilibrium condition reached in 4 h. The adsorption followed pseudo-second-order kinetics and the adsorption equilibrium followed the Temkin isotherm. Thermodynamic parameters such as ΔG^0, ΔH^0, and ΔS^0 were evaluated by applying the Arrhenius and Van't Hoff equations, and it was found that adsorption of Pb(II) on CPAC was spontaneous and endothermic.

Keywords: Adsorption; Lead; Pith activated carbon; Kinetics; Isotherms; Thermodynamics

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1. Introduction

Excessive release of toxic metals into the environment due to industrialization has created a great global concern. Monitoring and subsequent removal of toxic metals from the industrial effluents have, therefore, been made mandatory in India and throughout the Globe before their discharge into the environment [1]. Lead (Pb) has been widely used in industries and it is regarded as highly toxic. It gets introduced into the water bodies from a variety of sources, such as, storage batteries, lead smelting, tetra ethyl lead manufacturing, mining, plating, ammunition, and from the ceramic glass industries [2]. Anaemia, encephalopathy, and hepatitis are the common diseases caused by Pb contaminated water [3]. Pb(II) ions have affinity for ligands containing thiol and phosphatic groups, and

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they inhibit the biosynthesis of haeme, causing damage both to the kidney and the liver [3]. Removal of Pb(II) ions from wastewater is, therefore, necessary to meet the discharge limit. The World Health Organization has set the permissible limit of Pb in drinking water as $0.05\,\mathrm{mg\,L^{-1}}$ [4]. The Indian discharge limits for Pb is $0.1\,\mathrm{mg\,L^{-1}}$ [5].

The various methods used for the removal of metals from wastewaters include chemical precipitation, membrane filtration, ion exchange and adsorption [1]. Adsorption using activated carbon as an adsorbent is now regarded as a major process for wastewater treatment. However, due to high cost and about 10–15% loss during regeneration, non-conventional and cheap adsorbents have been used as alternative adsorbents [1]. In the last few years, a vast number of research have been done for the removal of Pb(II) from wastewater using the adsorption technique with different low cost materials. Coconut shell [3], grape stalk waste [6], olive cake [7], maize bran [8], olive stone waste [9], crop milling waste [10] are just a few examples of low cost materials used in the removal of Pb (II).

India is the third largest producer of coconuts in the world, producing about 12.8 billion coconuts a year [11]. Coir pith is generated in the process of separation of fiber from the coconut husk. It is estimated that the production of coir pith in India is about 0.5 million tons per year [12]. Accumulation of coir pith around coir industries is a big menace, since it is not easily biodegradable. Preparation of activated carbons from this cheap and abundant biomass eliminates the problem of coir-pith disposal as well as adds economic benefits to it. The objective of the present study is to evaluate the potential of coir-pith activated carbon (CPAC) activated by H_2SO_4 for the removal of Pb (II) from aqueous solution. The authors have already worked on the adsorption of cadmium ions from aqueous solution using granular activated carbon and activated clay [13], zinc onto tea factory waste [14–18], selenium onto granular activated carbon, powdered activated carbon, and bagasse fly ash [19,20], and benzaldehyde and tin onto granular activated carbon [21,22]. There are also other works on adsorption by various researchers [23–34].

2. Experimental procedure

2.1. Adsorbent

Coir pith, collected from nearby coir industry, was washed with distilled water and dried in sunlight. The dried material was washed with 98% sulfuric acid, with the weight ratio being 1 part of coir pith to 1.8 parts of acid, and kept at room temperature for about 5 h with occasional stirring. The acid-carbonized coir pith was washed several times with distilled water and with 5% sodium bicarbonate solution, followed by distilled water for the removal of remaining amount of acid. The dried coir-pith was carbonized in muffle furnace at 600°C for 1 h in the absence of air flow and in the presence of inert gas [3].

The physico-chemical characterization of CPAC was performed. Proximate analysis carried out using the standard procedure showed 18.83% moisture, 20.9% volatile matter, 3.2% ash and 57.1% fixed carbon. The bulk density was determined using the MAC bulk density meter. A high amount of carbon indicates that CPAC is basically organic in nature. For morphological characteristics, SEM analysis was carried out.

2.2. Adsorbate

All reagents used in this study were of analytical reagent grade. A $1\,\mathrm{g\,L^{-1}}$ Pb(II) stock solution was prepared dissolving the accurate quantity of $Pb(NO_3)_2$. All experimental solutions of Pb(II) were prepared by proper dilution of this stock solution with distilled water. The initial pH (pH_0) of the solution was adjusted to the desired value using a 0.1 M NaOH or 0.1 M H_2SO_4 solution prior to the addition of the adsorbent.

2.3. Batch adsorption studies

Adsorption experiments were carried out by agitating 100 mL of Pb(II) solution of a known initial concentration (C_0), pH_0 and a known amount of the CPAC in a 250 mL stoppered conical flask in a temperature-controlled shaking water bath at 30 ± 1°C. Samples were withdrawn at appropriate time intervals. The concentration of Pb(II) in the supernatant was estimated with a UV-VIS spectro-photometer by the PAR reagent (2,4-pyridyl azoresorcinol) method, monitoring the absorbance at 580 nm [35]. The effect of pH_0 on Pb(II) removal was studied in the range of pH_0 from 2 to 11. For the optimal amount of adsorbent dose, 100 mL Pb(II) solutions were contacted with different amounts of CPAC till equilibrium was attained. The kinetics of adsorption was determined by analyzing the adsorptive uptake of the Pb(II) from the aqueous solution at different time intervals. For adsorption isotherms, Pb(II) solutions of different concentrations were agitated with the known amount of CPAC till equilibrium

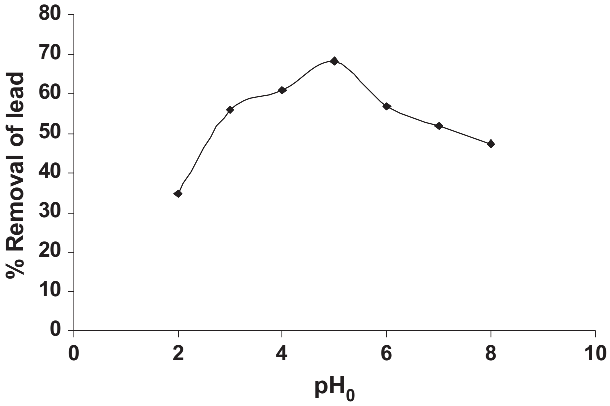

Fig. 1. Effect of pH_0 on the adsorption of Pb(II) using CPAC as the adsorbent. $T = 303\,\mathrm{K}$, $t = 4\,\mathrm{h}$, $C_0 = 20\,\mathrm{mg\,L^{-1}}$, CPAC dose = $1\,\mathrm{g\,L^{-1}}$.

was achieved (4 h). To see the effect of temperature on the adsorption of Pb(II) from solution by CPAC, experiments were also conducted at 30, 50 and 70°C.

3. Results and discussion

3.1. Effect of initial pH (pH_0)

Adsorption of Pb(II) onto CPAC was studied at various pH_0 (Fig. 1). It can be seen that uptake (~30%) is quite low at a low pH, however with the increase in pH_0 up to 5, significant enhancement (~70%) in adsorption is noticed. The optimum pH_0 was found to be 5 with a maximum removal of about 68% from solution with $C_0 = 20\,\mathrm{mg\,L^{-1}}$. At lower pH values the electrostatic forces of repulsion between the adsorbent and adsorbate (Pb(II)) are prominent which do not allow adsorption onto CPAC. So the efficiency of metal removal is low at lower pH_0. At $pH_0 > 5$, there is a possibility of Pb(II) precipitation as $Pb(OH)_2$ by nucleation which is still in aqueous phase. So the removal efficiency decreases at higher pH_0. For $2 < pH_0 < 5$, the percentage removal increased sharply. This might be due to the fact that the metal ions start replacement of hydrogen ions from the adsorbent surface by the Pb(II). As the pH increases, the H^+ ion concentration decreases and hence the positively charged Pb ions are adsorbed at negatively charged sites on the adsorbent. The Pb(II) ion predominates at pH below 5 and it begins to precipitate out as $Pb(OH)_2$ at pH values just above 5. At pH 6, the species distribution is approximately 90% Pd(II) and 10% $Pb(OH)^-$.

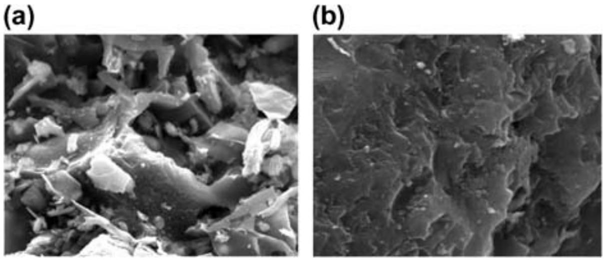

Fig. 2. SEM of CPAC (a) before, and (b) after Pb(II) adsorption from aqueous solution at 1000× magnification.

Scanning electron micrographs (SEM) (Fig. 2) of CPAC were taken before and after adsorption of Pb (II) ions. These photomicrographs show the structure of CPAC. The surface of the CPAC is fibrous in nature before adsorption and becomes smooth after adsorption of Pb(II) due to the coverage of pores.

3.2. Effect of adsorbent dosage

The effect of adsorbent dose on the removal of Pb (II) by CPAC at an initial concentration $C_0 = 20\,\mathrm{mg\,L^{-1}}$ is shown in Fig. 3. It can be seen that the Pb(II) removal increases up to a certain limit and then it remains almost constant. The optimum dose was found to be $1\,\mathrm{g\,L^{-1}}$. The percentage of removal increased with increase in the adsorbent dose due to higher total surface area of the adsorbent particles [36].

3.3. Effect of agitation time (t) and initial Pb(II) concentration (C_0)

The effects of t and C_0 on the removal of Pb(II) by CPAC are shown in Fig. 4. It is evident that the amount of Pb(II) adsorbed per unit mass of adsorbent (q_t) increased with C_0 and remained nearly constant after the equilibrium time of 4 h (optimum contact

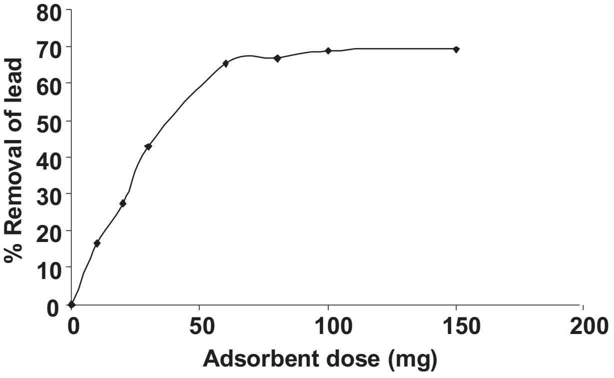

Fig. 3. Effect of adsorbent mass on the adsorption of Pb(II) using CPAC as the adsorbent. $T = 303\,\mathrm{K}$, $t = 4\,\mathrm{h}$, $C_0 = 20\,\mathrm{mg\,L^{-1}}$, $pH_0 = 5$, volume of solution = 100 mL.

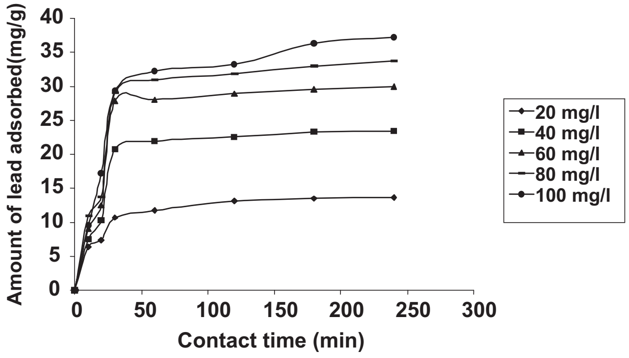

Fig. 4. Effect of agitation time and concentration of Pb(II) on the amount adsorbed using CPAC as the adsorbent. $T = 303$ K, $t = 4$ h, $pH_0 = 5$, CPAC dose $= 1\,\mathrm{g\,L^{-1}}$.

time). It can be seen that the q_t increases with the increase in C_0, this may be due to the resistance to the uptake of Pb(II) from the solution decreases with the increase in C_0. Hence the rate of adsorption increases due to increase in the driving force. The curves are single, smooth and continuous leading to saturation. The adsorption curves of contact time indicate the possible mono-layer coverage of Pb(II) ions on the surface of CPAC which may be represented by the Freundlich isotherm [37].

3.4. Adsorption kinetics

The pseudo-first-order [1] and the pseudo-second-order kinetic models [38] in linear form are given by Eqs. (1) and (2) respectively:

$$\log(q_e - q_t) = \log q_e - \frac{k_f}{2.303}t \tag{1}$$

$$\frac{t}{q_t} = \frac{1}{k_S q_e^2} + \frac{1}{q_e}t \tag{2}$$

where q_t is the amount of adsorbate adsorbed at time t ($\mathrm{mg\,g^{-1}}$), q_e the adsorption capacity at equilibrium ($\mathrm{mg\,g^{-1}}$), k_f the pseudo-first-order rate constant ($\mathrm{min^{-1}}$), k_S the pseudo-second-order rate constant ($\mathrm{g\,mg^{-1}\,min^{-1}}$) and t the contact time (min).

The values of parameters for pseudo-first- and pseudo-second-order kinetic models for Pb(II) adsorption on CPAC at $C_0 = 20$, 40, 60, 80 and $100\,\mathrm{mg\,L^{-1}}$ were determined using linear regression, and are given in Table 1. The calculated correlation coefficients are closer to unity, and also $q_{e,calc}$ is better predicted by the pseudo-second-order kinetic model. Therefore, sorption can be approximated more appropriately by the pseudo-second-order kinetic model.

3.5. Adsorption isotherms

To establish the most appropriate model to represent the equilibrium, Langmuir, Freundlich and Temkin isotherms [39] given by Eqs. (3–5), respectively, were tested:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{4}$$

$$q_e = K_F C_e^{1/n} \tag{5}$$

$$q_e = B_1 \ln(K_T C_e) \tag{6}$$

where q_e is the fraction of the adsorbate under equilibrium condition ($\mathrm{mg\,g^{-1}}$), C_e the equilibrium metal ion concentration ($\mathrm{mg\,L^{-1}}$), K_L the Langmuir adsorption constant ($\mathrm{L\,mg^{-1}}$) related to the energy of adsorption, and q_m the adsorption capacity of the adsorbent ($\mathrm{mg\,g^{-1}}$). K_F is the Freundlich constant ($\mathrm{L\,mg^{-1}}$), and $(1/n)$ the heterogeneity factor, K_T is the equilibrium binding constant ($\mathrm{L\,mg^{-1}}$) corresponding to the maximum binding energy and constant B_1 is related to the heat of adsorption.

The Marquardt's percent standard deviation (MPSD) error function [40] was used to test the adequacy of the isotherm equations to represent the experimental data. This error function is given as

$$\mathrm{MPSD} = 100\sqrt{\frac{1}{n-p}\sum_{i-1}^{n}\left(\frac{q_{e,meas} - q_{e,calc}}{q_{e,meas}}\right)_i^2} \tag{7}$$

where $q_{e,mean}$ is the measured value and $q_{e,calc}$ is the calculated value The parameters of the isotherms and the correlation coefficient, R^2, as determined by using the solver add-in function of the MS excel, for fitting of experimental data are listed in Table 2. By comparing the results of the values for the MPSD error function and the correlation coefficients, it is found that the Temkin isotherm best represents the equilibrium adsorption of Pb(II) by CPAC.

3.6. Thermodynamic study

The Gibbs free energy change of the adsorption process is related to the equilibrium constant by the classic Van't Hoff equation [1]:

$$\Delta G^0 = -RT \ln K_D \tag{8}$$

According to thermodynamics, the Gibbs free energy change is also related to the entropy change and heat of adsorption at constant temperature by the following equation:

Table 1
Kinetic parameters for the removal of Pb(II) from waste water by CPAC. $pH_0 = 5$, $T = 303\,\mathrm{K}$, CPAC dose = 1 g L^{-1}

Pseudo-first-order

C_0 (mg L^{-1})	$q_{e,\exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	k_f (1 min^{-1})	R^2
20	13.6250	8.8145	0.0258	0.9844
40	23.4500	11.8522	0.0233	0.8759
60	29.9000	12.0642	0.0205	0.7571
80	33.8000	16.1622	0.0179	0.8201
100	37.2500	21.7220	0.0168	0.8864

Pseudo-second-order

C_0 (mg L^{-1})	$q_{e,cal}$ (mg g^{-1})	h (mg g^{-1})	k_s (mg g^{-1})	R^2
20	14.6627	1.0081	0.0047	0.9983
40	26.1780	1.3428	0.0019	0.9843
60	33.5570	1.6377	0.0014	0.9773
80	37.3734	1.8241	0.0013	0.9818
100	40.9836	1.7504	0.0010	0.9860

Table 2
Isotherm parameters for removal of Pb(II) from aqueous solution by CPAC. $pH_0 = 5$, $t = 4\,\mathrm{h}$, CPAC dose = 1 g L^{-1}

Freundlich constants

T (K)	K_F ((mg g^{-1})/(mg L^{-1})$^{1/n}$)	$1/n$	R^2	MPSD
303	6.3949	0.4381	0.9896	4.3414
323	6.7087	0.4418	0.9911	4.1163
343	7.0696	0.4439	0.9823	3.9028

Langmuir constants

T (K)	K_L (L mg^{-1})	Q_m (mg g^{-1})	R_L	R^2	MPSD
303	0.0631	46.0829	0.4421	0.9994	1.4015
323	0.0641	48.5437	0.4382	0.9991	1.8706
343	0.0656	51.0204	0.4395	0.9987	2.352

Temkin constants

T (K)	K_T (L mg^{-1})	B_1	R^2	MPSD
303	0.5911	10.295	0.9998	0.4476
323	0.6065	10.819	0.9998	0.5372
343	1.3093	9.1554	0.9995	0.5326

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \tag{9}$$

Combining the above two equations, we get

$$\ln K_D = \frac{-\Delta G^0}{RT} = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \tag{10}$$

where ΔG^0 is the free energy change (kJ mol^{-1}), ΔH^0 the change in enthalpy (kJ mol^{-1}), ΔS^0 the entropy change (J mol^{-1} K^{-1}), T the absolute temperature (K), R the universal gas constant (8.314 J mol^{-1} K^{-1}) and K_D is the thermodynamic equilibrium constant. K_D for the adsorption reaction can be defined:

$$K_D = \frac{a_s}{a_e} = \frac{n_s C_0}{n_e C_e} \tag{11}$$

where a_s is the activity of the adsorbed solute, a_e the activity of the solute in the equilibrium solution, v_s is the activity coefficient of the adsorbed solute, and v_e the activity coefficient of the solute in the equilibrium solution. As the concentration of the solute in the solution approaches zero, the activity coefficient v approaches unity. Eq. (11) may then be written as:

$$\lim_{C_e \to 0} \frac{C_0}{C_e} = \frac{a_s}{a_e} = K_D \tag{12}$$

The values of K_D are obtained by plotting $\ln(C_0/C_e,)$ vs. C_e, and extrapolating to zero C_e [41]. From constant K_D, the ΔG^0 of adsorption Pb(II) ion onto CPAC at 303, 323 and 343 K is found to be -2.82, -3.10 and $-3.53\,\mathrm{kJ\,mol^{-1}\,K^{-1}}$, respectively. $\Delta H^0 = 2.54\,\mathrm{kJ\,mol^{-1}}$ and $\Delta S^0 = 17.63\,\mathrm{J\,mol^{-1}\,K^{-1}}$ are determined from the slope and intercept of the linear plot of $\ln K_D$ vs. $1/T$. The positive ΔH^0 value confirms the endothermic nature of the overall-sorption process. The adsorption process in the solid-liquid system is a combination of two processes: (a) the desorption of the molecules of solvent (water) previously adsorbed and (b) the adsorption of adsorbate species. The value of ΔH^0 is less than $20\,\mathrm{kJ\,mol^{-1}}$ indicating the physical nature of the adsorption process. The positive value of ΔS^0 suggests increased randomness at the solid/solution interface with some structural changes in the adsorbate and adsorbent, and affinity of the Pb(II) towards CPAC. Also, the positive ΔS^0 value corresponds to an increase in the degree of freedom of the adsorbed species [39].

4. Conclusions

The present study shows that the H_2SO_4-treated activated carbon developed from the agricultural waste coir-pith (CPAC) is an effective and low-cost adsorbent for the removal of Pb(II) from aqueous solution. The rate of adsorption increases with the increase in C_0 due to increase in the driving force and the higher percentage of Pb(II) removal by CPAC was possible provided the C_0 in the solution was low. The optimum conditions for Pb(II) removal were found to be $pH_0 \approx 5$, adsorbent dose $\approx 1\,\mathrm{g\,L^{-1}}$ of solution for Pb (II) concentration of $20\,\mathrm{mg\,L^{-1}}$. The equilibrium between the adsorbate in the solution and on the adsorbent surface was practically achieved in 4 h. Adsorption kinetics was found to follow the second-order kinetic model. The Temkin isotherm was found to represent the adsorption equilibrium. The negative values of ΔG^0 indicate that the adsorption of Pb(II) onto CPAC is feasible and spontaneous. ΔH^0 and ΔS^0 were found to be $2.54\,\mathrm{kJ\,mol^{-1}}$ and $17.63\,\mathrm{J\,mol^{-1}\,K^{-1}}$, respectively. The positive ΔH^0 value confirms the endothermic nature of the overall-sorption process. The value of ΔH^0 is less than $20\,\mathrm{kJ\,mol^{-1}}$ indicating the physical nature of the adsorption process. The positive value of ΔS^0 suggests increased randomness at the solid/solution interface with some structural changes in the adsorbate and adsorbent, and affinity of the Pb(II) towards CPAC. Also, the positive ΔS^0 value corresponds to an increase in the degree of freedom of the adsorbed species.

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