



Equilibrium and kinetic studies on adsorption of Pb(II) by activated palm kernel husk carbon

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ABSTRACT

Batch experiments were conducted to study the ability of activated palm kernel husk carbon (APKHC) to adsorb Pb(II) ions from aqueous solutions. The effects of various operational parameters, such as temperature (25–55°C), pH (1.0–5.0), contact time (0–120 min), and adsorbent dose (0.5–3.5 g/L) were examined. The results suggested that the adsorption of Pb(II) is exothermic and strongly dependent on pH. The optimum pH for Pb(II) removal is 5.0. The Pb(II) uptake by APKHC was fast and reached an equilibrium in 80 min. The Pb(II) removal efficiency increased with increasing APKHC dosage up to 2.0 g/L, and then reached a plateau with approximately 97% of Pb(II) removed. Experimental data were fitted using Langmuir and Freundlich isotherms. The Pb(II) adsorption can be described well by the Langmuir model with a maximum monolayer uptake capacity of 98.04 mg/g. Kinetic study results suggested that the adsorption process proceeded according to the pseudo-second-order kinetic model. In short, APKHC can be used as an effective economical adsorbent for Pb(II) removal.

Keywords: Pb(II); Adsorption; Activated palm kernel husk carbon (APKHC); Isotherm; Kinetics

1. Introduction

Lead is widely used as an industrial raw material in the manufacture of storage batteries, pigments, leaded glass, fuel, photographic materials, matches, and explosives [1,2]. Once released into air, soil, and water bodies, lead can cause environmental pollution. It is poisonous and harmful if swallowed, inhaled, or absorbed through the skin. Moreover, it can

accumulate slowly in the body, which leads to renal impairment and hypertension, cognitive dysfunction, infertility, and abnormalities in pregnant women [3–5]. Given that lead is a highly toxic metal and a very strong poison, the US Environmental Protection Agency has set a guideline limit of 15 µg/L of lead in drinking water [6]. Therefore, there is a great need to treat lead-contaminated wastewater before its discharge into the environment.

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Common approaches for treating lead-bearing effluents include ion exchange, chemical precipitation, coagulation, membrane filtration, reverse osmosis, and solidification/stabilization [7,8]. These methods have several drawbacks, such as high operating costs, low selectivity, incomplete removal, high energy requirement, and production of toxic sludge, thereby limiting their practical use [9,10]. Thus, the development of efficient and cheap separation processes for Pb(II) removal poses a serious problem to environmental researchers.

In recent years, great interest is increasingly expressed to particular agricultural and forestry residues, such as rice husk [11], rice straw [12], corncob [13], fruit peels [14–16], plant leaves [17,18], and nutshells [19–21] because of their less cost, less secondary pollution, and easy recycling. Palm kernel husk is an agricultural biomass residue from the palm oil industry. It can partially take the place of fossil fuel in steam power plant or lightweight aggregate in concrete because it contains large amounts of cellulose, hemicellulose, and some other extractive compounds [22,23]. Palm kernel husk can also be made into activated carbon via industrial standard methods, including chemical catalytic activation using conventional catalysts (ZnCl_2 , NaOH or KOH, and H_3PO_4) and physical activation using steam or carbon dioxide. Activated palm kernel husk carbon (APKHC) often has a micro-mesoporous and large internal surface area structure [24], thereby making it possible to use APKHC as a superior adsorption material.

Thus far, many experiments have been carried out on the adsorption of Pb(II) using some activated carbon materials. However, only a few investigations have focused on the application of APKHC in the removal of Pb(II). In this work, APKHC was employed to remove Pb(II) in solution. The influence of several environmental factors, such as contact time, temperature, pH, and adsorbent dosage, on the Pb(II) adsorption process was examined in detail. The adsorption isotherms and kinetics were also studied. The purpose of this research was to assess the feasibility of the use of APKHC as an adsorbent to remove Pb(II).

2. Materials and methods

2.1. Adsorbent, chemical reagents, and Pb(II) stock solution

APKHC powder was purchased from Guoqing Water Purification Material Co. Ltd. in China, and used as an adsorbent for the subsequent metal adsorption experiments. The quality testing indexes of APKHC were determined as follows according to the National Standard of China for Activated Nutshell

Carbon Testing: coarseness, 40–80 mesh; iodine value, $\geq 1,100$ mg/g; specific surface area, $1,350$ m²/g; CCl_4 adsorption, $\geq 90\%$; strength, $\geq 95\%$; packing density, 430–438 g/L; total pore volume, 0.7–1 cm³/g; methylene blue value, 100–150 mg/g; pH, 8–10; moisture, $\leq 8\%$; and ash, $\leq 2.5\%$. The functional groups on the surface of the APKHC sample were characterized using a NICOLET iS10 FT-IR spectrometer (Thermo Electron Co. NJ, USA). The microstructure of APKHC was characterized via scanning electron microscopy (SEM, Model S-4800 Hitachi, Tokyo, Japan). The sample was gold coated prior to SEM observation.

A stock solution of Pb(II) (1,000 mg/L) was prepared by dissolving lead nitrate in distilled water. This stock solution was then diluted to the required concentrations and their pH values were adjusted to desired values using 1.0 mol/L NaOH or HCl. All chemicals and reagents were of analytical grade and were used as received without any further purification.

2.2. Batch adsorption experiments

Batch adsorption experiments were conducted in a series of 250 mL conical flasks to examine the effect of the aforementioned process variables on Pb(II) removal. Pretrial experiments were also carried out to fix the minimum and maximum levels of each variable. About 100 mL of Pb(II) solution was mixed with a known amount of APKHC powder. Thereafter, the flasks were agitated at 140 rpm on a thermo-controlled rotary shaker. Finally, the equilibrated solutions were withdrawn and the adsorbent was separated from them via centrifugation. The concentration of the Pb(II) in the residual solution was determined via a standard method given by Li et al. [25]. All the experiments were repeated thrice to confirm the results, and the average values are presented.

The Pb(II) removal efficiency and adsorption capacity of APKHC powder were calculated using the following equations:

$$\text{Ad}\% = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

$$Q_t = \frac{(C_0 - C_t) \times V}{W} \quad (2)$$

$$Q_e = \frac{(C_0 - C_e) \times V}{W} \quad (3)$$

where Ad% is the Pb(II) removal efficiency; Q_e and Q_t are the adsorption capacity (mg/g) at equilibrium and at time t (min), respectively; C_0 , C_t , and C_e are the initial Pb(II) concentration, liquid-phase Pb(II)

concentration at time t , and equilibrium Pb(II) concentration (mg/L), respectively; V is the volume of the aqueous solution (L); W is the mass of the adsorbent (g).

2.3. Equilibrium modeling

The Freundlich and Langmuir equations are commonly adopted to describe the adsorption isotherms. The Langmuir model is based on the assumptions of adsorption homogeneity, such as uniformly energetic adsorption sites, monolayer surface coverage, and no interactions between adsorbate molecules on adjacent sites [26,27]. The Freundlich isotherm can be applied to non-ideal sorption onto heterogeneous surfaces involving multilayer adsorption [28]. In this study, the Langmuir and Freundlich adsorption equations were both used to correlate the obtained isotherm data.

The linearized Langmuir equation can be expressed as follows:

$$\frac{C_e}{Q_e} = \frac{1}{Q_{\max}} C_e + \frac{1}{bQ_{\max}} \quad (4)$$

where Q_{\max} represents the maximum monolayer adsorption capacity (mg/g), and b represents the affinity constant related to the free energy of adsorption (L/mg).

The linearized Freundlich equation can be described as follows:

$$\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (5)$$

where K_F is the Freundlich constant indicative of the adsorption capacity of the adsorbent (mg (L/mg)^{1/n}), and n is the Freundlich exponent depicting adsorption intensity (dimensionless).

2.4. Kinetic modeling

Pseudo-first-order and pseudo-second-order kinetic models are often used in kinetic investigations. The pseudo-first-order equation is a simple kinetic model describing the kinetic process of liquid-solid-phase adsorption that was presented by Lagergren [29]. Its non-linearized formula is given as follows:

$$Q_t = Q_e(1 - e^{-k_1 t}) \quad (6)$$

where k_1 is the rate constant of the pseudo-first-order sorption (min⁻¹). Obviously, Q_e and k_1 can be worked

out by plotting Q_t against t and by further non-linear regression analysis.

The pseudo-second-order model based on the adsorption equilibrium capacity may be expressed as the following linear form [30]:

$$\frac{t}{Q_t} = \frac{1}{Q_e} t + \frac{1}{k_2 Q_e^2} \quad (7)$$

where k_2 is the rate constant of pseudo-second-order adsorption [g/(mg min)]. Obviously, Q_e and k_2 can be determined experimentally by plotting t/Q_t against t and by further linear regression analysis.

3. Results and discussion

3.1. Characterization of APKHC

The surface morphology of APKHC is shown in Fig. 1. Obviously, the adsorbent surface not only displays a rough and irregular texture, but is also fractured and fragmented. Moreover, it has a macroporous feature with the pore diameter of about 10 μm, there is no uniform and well-developed pore structure on the material surface. This pore size can accommodate considerable Pb(II) because the diameter of Pb(II) cation is just about 350 pm. Moreover, this activated carbon material features a high BET specific surface area of up to 1,350 m²/g and a high total pore volume of 0.7–1 cm³/g. Generally speaking, high surface area and porous structure are often the basic parameters for a high-performance adsorbent. Hence, APKHC could be a good adsorbent for treating heavy metals.

Infrared spectroscopy provides important information on the chemical structure of the adsorbent material. The FTIR spectrum of APKHC is shown in Fig. 2. The absorption peak at 1,654.28 cm⁻¹ is the characteristic peak of C=O group. On the other hand, the band that appeared at 1,079.05 cm⁻¹ could be ascribed to the C–O stretch. The absorptions at 1,560.57 cm⁻¹ and 1,577.18 cm⁻¹ could be assigned to carbon-carbon stretching vibrations in the aromatic ring backbone. The bands at about 3438.20 cm⁻¹, as well as the peaks at 3569.31 cm⁻¹ and 3589.28 cm⁻¹ were attributed to O–H stretch vibrations in hydroxyl groups. The location of hydrogen-bonded hydroxyl groups is usually in the range of 3,200–3,650 cm⁻¹ for alcohols and phenols. These results demonstrated the existence of abundant carboxyl and hydroxyl groups on the APKHC surface.

3.2. Effect of contact time

To determine the equilibrium time for maximum adsorption capacity, we examined the effect of contact

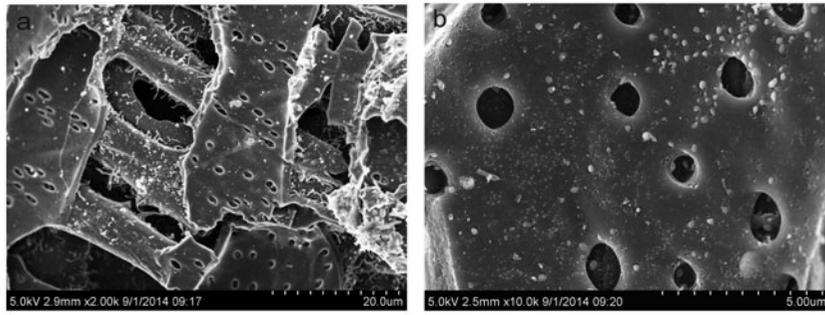


Fig. 1. SEM micrographs of APKHC (magnification: (a) 2,000×; and (b) 10,000×).

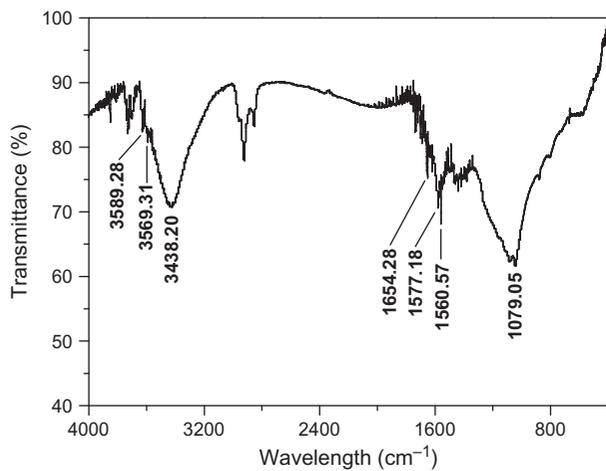


Fig. 2. FTIR spectrum of APKHC.

time on the adsorption of Pb(II) onto APKHC at two initial Pb(II) concentrations (Fig. 3). Obviously, for 100 or 200 mg/L of Pb(II), the adsorption capacity of Pb(II) onto APKHC both increased very fast within the first 30 min. The adsorption process then proceeded relatively slowly and finally reached an equilibrium state after 80 min. The initial fast rate of adsorption can be due to the existence of a great number of vacant surface sites that can be readily accessible for adsorption. However, as the coverage increased, the number of available surface sites for adsorption reduces and the remaining vacant surface sites are difficult to be occupied until equilibrium is achieved when the adsorption is dominated by the rate, at which the adsorbate is transported from the outside to the inside sites of the adsorbent. In this work, a contact time of 80 min was chosen in subsequent sorption experiments to guarantee an optimum Pb(II) uptake.

3.3. Effect of temperature

The removal efficiency of Pb(II) from solution was determined after 80 min of agitation at four different

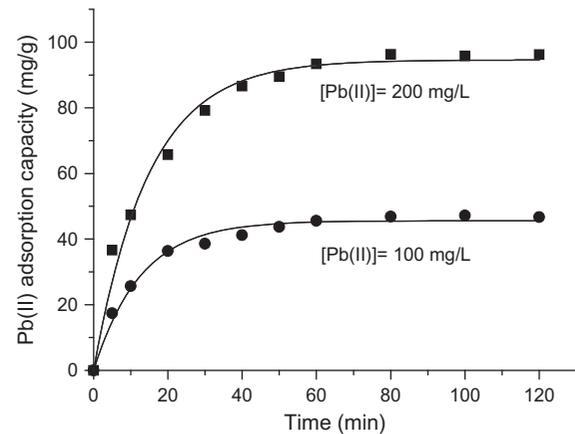


Fig. 3. Effect of time on the Pb(II) adsorption by APKHC (temperature: 25°C; pH: 5.0; APKHC dosage: 2.0 g/L; solution volume: 100 mL).

temperatures: 25, 35, 45 and 55°C (Fig. 4). The Pb(II) removal efficiency slightly changed when the temperature was increased from 25 to 35°C, and then

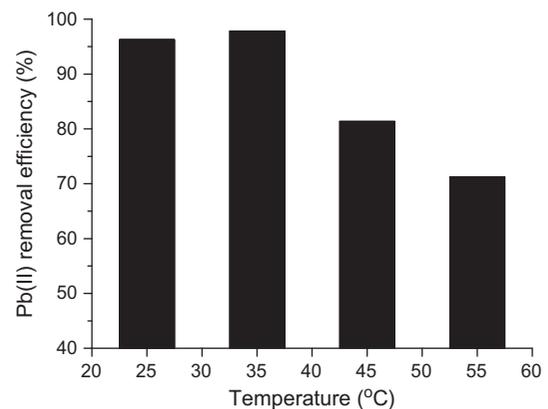


Fig. 4. Effect of temperature on Pb(II) adsorption by APKHC (pH: 5.0; time: 80 min; Pb(II) concentration: 200 mg/L; APKHC dosage: 2.0 g/L; solution volume: 100 mL).

decreased from 97.8 to 71.2% when the temperature was further increased from 35 to 55°C. This decrease in the uptake of Pb(II) with increasing temperature implied that the adsorption process was exothermic in nature.

3.4. Effect of pH

Medium pH is a crucial parameter for the adsorption of heavy metals from aqueous solution because it influences the solubility of metals and the dissociation state of the functional groups on the adsorbent surface during reaction [31]. The solubility product constant value (K_{sp}) for $Pb(OH)_2$ at 25°C is 1.42×10^{-20} . The initial Pb(II) concentration used in this study was 200 mg/L (0.966 mM). Thus, the pH after which the $Pb(OH)_2$ started to form was calculated to be 5.58 in terms of the solubility product principle. Actually, a white precipitate could be observed when the pH value was increased up to 5.3 in the experiment. Therefore, the effect on the removal of Pb(II) was investigated between the pH range from 1.0 to 5.0. In this case, the adsorption process was limited to an ionic state of Pb(II), thereby excluding the interference of $Pb(OH)_2$ precipitate.

The Pb(II) adsorption is relatively low at lesser pH with a minimum Pb(II) adsorption of 36.6% at pH 1.0. However, a significant enhancement in adsorption is observed with increasing pH from 1.0 to 5.0 (Fig. 5). The optimum pH for Pb(II) adsorption was found to be 5.0 with 96.3% of Pb(II) removal from the solution. The fact that the acid environment is not helpful for the adsorption may be explained as follows. At low pH values, many hydrions compete for the available

active sites (carboxyl and hydroxyl groups) on the adsorbent surface and they protonate these functional groups. Thus, the adsorbent surface often showed a cationic feature, which is not beneficial to Pb(II) uptake because of the mutual electrostatic repulsion between protonated APKHC surface and Pb(II). Along with the increase in pH, those protonated carboxyl and hydroxyl groups dissociated and exhibited an anionic character. The dissociation degree of the carboxyl and hydroxyl groups increased and the negative oxyanions of the carboxyl and hydroxyl groups were formed. Thus, the adsorption could be strengthened as a result of the electrostatic attraction between the APKHC surface and Pb(II). Physisorption and chemisorption should both be involved in the Pb(II) adsorption process because APKHC has a porous structure and is rich in hydroxyl and carboxyl on the surface.

3.5. Effect of APKHC dosage

Adsorbent dosage is an important parameter because this factor determines the adsorbate removal efficiency for a given initial concentration of the adsorbate. The effect of adsorbent dosage on the Pb(II) removal was studied at seven different sorbent dosages ranging from 0.5 to 3.5 g/L (Fig. 6). The adsorption increased from 24.1 to 96.3% when the APKHC dosage was increased from 0.5 to 2.0 g/L, which may be due to the rapid increase in surface area and number of available active sites for the adsorption of Pb(II). When the APKHC amount was further increased from 2.0 to 3.5 g/L, the percentage removal of Pb(II) slightly changed with $(97 \pm 1.1)\%$ of Pb(II) removed. This phenomenon can be interpreted as

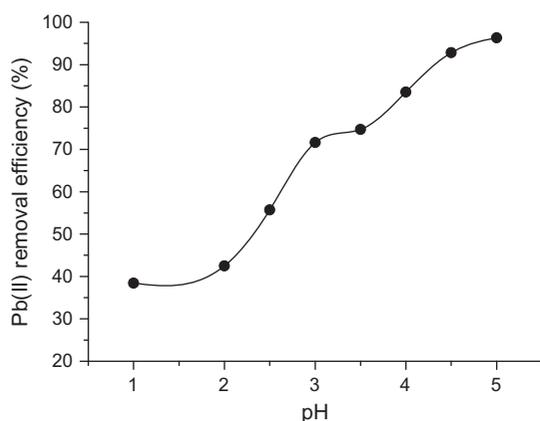


Fig. 5. Effect of pH on Pb(II) adsorption by APKHC (temperature: 25°C; contact time: 80 min; Pb(II) concentration: 200 mg/L; APKHC dosage: 2.0 g/L; solution volume: 100 mL).

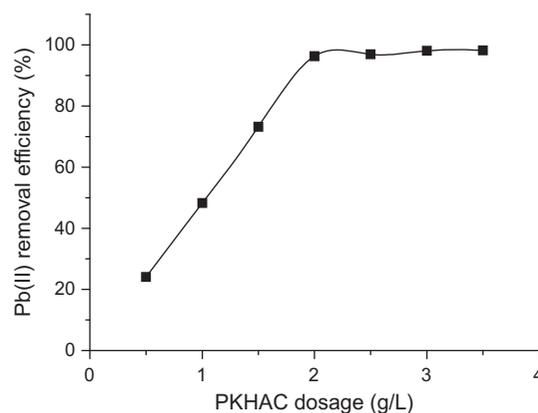


Fig. 6. Effect of APKHC dosage on Pb(II) adsorption by APKHC (temperature: 25°C; contact time: 80 min; pH: 5.0; Pb(II) concentration: 200 mg/L; solution volume: 100 mL).

follows. The adsorbed Pb(II) could either hinder the access into the interior pores of APKHC or may cause particles to conglomerate, thereby reducing the availability of active sites for adsorption. Therefore, the optimum APKHC dosage was selected as 2.0 g/L for Pb(II) removal.

3.6. Interference effect of various ions

To examine the selectivity of APKHC, selective adsorption tests were performed at the optimum conditions. The results are given in Table 1. The presence of Ca(II), Mg(II), Zn(II), Cu(II), SO_4^{2-} , and Cl^- had a particular effect on the uptake of Pb(II) on APKHC. When these aforementioned foreign ions are present in the solution, the Pb(II) removal efficiency decreased ranged from 2.8 to 9.3% (Table 1). Therefore, the Pb(II) removal by APKHC is non-selective in the presence of other interfering ions.

3.7. Sorption isotherms

Adsorbate–adsorbent interactions can be characterized by adsorption isotherms, which have a key function in optimizing the use of adsorbents. Thus, fitting equilibrium data with empirical Langmuir and Freundlich equations is crucial to the practical design and operation of adsorption systems. The constants of Langmuir and Freundlich models are obtained from fitting the adsorption equilibrium data from Fig. 6 and are listed in Table 2. The square of correlation coefficient (R^2) of the latter is 0.518, which indicates that the adsorption on APKHC cannot follow the Freundlich isotherm. By contrast, the Langmuir isotherm can describe this uptake process very well because the corresponding R^2 value (0.999) is very close to 1.0 and the calculated theoretical value of Q_{\max} (98.04 mg/g) is very close to the experimental one

(approx. 98 mg/g). The adsorption of Pb(II) on activated carbon is classified as monolayer adsorption because the Langmuir isotherm is a monolayer adsorption model.

Q_{\max} is an important parameter for evaluating the adsorption ability of adsorbents. The Q_{\max} value for Pb(II) uptake by APKHC in this study was 98.04 mg/g, which suggests that it has a better adsorption performance compared with most of the other adsorbents reported in literature (Table 3).

Based on further analysis of the Langmuir equation, the Langmuir adsorption isotherm can be described using an equilibrium parameter (R_L) calculated by the following equation [42]:

$$R_L = \frac{1}{1 + b \times C_0} \quad (8)$$

where C_0 is the initial Pb(II) concentration (mg/L), b is the Langmuir constant (L/mg) mentioned previously, and R_L parameter is a useful indicator for estimating whether the adsorption is unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$). In this research, the R_L value was calculated out to be 0.0066 and just lied between 0 and 1.0 (Table 2), which indicated that the adsorption of Pb(II) onto APKHC was greatly favorable.

3.8. Kinetic modeling

To study the mechanism of Pb(II) adsorption by APKHC and the potential rate-controlling steps, two most frequently used kinetic models, namely, pseudo-first-order and pseudo-second-order kinetic models, were used to assess the adsorption of Pb(II). The derived model parameters from fittings of the data in Fig. 3, including the kinetic constant (k), square of correlation coefficient (R^2), and equilibrium adsorption capacity (Q_e), are shown in Table 4.

The R^2 values of the pseudo-second-order model (0.999 and 0.998) are closer to 1.0 and greater than those of the pseudo-first-order model (0.964 and 0.986). On the other hand, the Q_e values calculated from the pseudo-second-order model are nearer to the observed experimental ones (Q_{exp}). These results indicated that the adsorption of Pb(II) on APKHC was well fitted to the pseudo-second-order kinetic model compared with the first-order model. It can be concluded that the pseudo-second-order kinetic model gives a good correlation for the adsorption of Pb(II) on APKHC, and the chemisorption might be the rate-limiting step in the adsorption process. Similar results were also previously reported for the adsorption of Pb(II) by other adsorbents, such as carbonate

Table 1
Effect of foreign ions on the removal of Pb(II) by APKHC^a

Foreign ions	% decrease in removal of Pb(II)
Ca(II)	6.2
Mg(II)	5.8
Zn(II)	8.7
Cu(II)	9.3
SO_4^{2-}	3.4
Cl^-	2.8

^aAdsorption condition: temperature, 25°C; pH, 5.0; contact time, 80 min; APKHC dosage, 2.0 g/L; initial Pb(II) concentration, 200 mg/L; metal/lead(II) molar ratios, 1:1; solution volume: 100 mL.

Table 2
Adsorption equilibrium constants obtained from the Langmuir and Freundlich isotherms

Langmuir			Freundlich		
Q_{\max} (mg/g)	b (L/mg)	R^2	K_F (mg (L/mg) ^{1/n})	n	R^2
98.04	0.751	0.999	60.09	9.259	0.518

Table 3
Comparison of the Pb(II) adsorption capacity with other agricultural residues and activated carbon

Adsorbent	Q_{\max} (mg/g)	References
Maple sawdust	3.19	[32]
Hazelnut husk-activated carbon	13.05	[33]
<i>Eichhornia</i> -activated carbon	16.61	[34]
Activated carbon developed from apricot stone	21.38	[35]
Coconut shell carbon	26.5	[36]
HNO ₃ -modified activated carbon from peanut shell	35.5	[37]
Green coconut shell	54.62	[38]
Tea waste	65	[39]
Activated carbon prepared from bio-plant stems activation with H ₃ PO ₄	119	[40]
Na ₂ S-HNO ₃ -modified activated carbon	129.5	[41]
Activated palm kernel husk carbon	98.04	This study

Table 4
Pseudo-first-order and pseudo-second-order adsorption rate constants for lead sorption by APKHC

Pseudo-first-order			Pseudo-second-order			Experimental value Q_{exp} (mg/g)
k_1 (min ⁻¹)	Q_{e1} (mg/g)	R^2	k_2 (g/(mg min))	Q_{e2} (mg/g)	R^2	
0.067	94.57	0.964	9.26×10^{-4}	96.49	0.998	ca. 96
0.079	45.58	0.986	2.56×10^{-3}	47.15	0.999	ca. 47

hydroxyapatite extracted from an eggshell waste [43], sesame leaf [44], and carbon-coated monolith [45].

3.9. APKHC regeneration

To make the adsorption process more effective and economically feasible, the adsorbent regeneration and Pb(II) recovery should be examined. The regeneration of APKHC was carried out using 1.0 mol/L HNO₃. Adsorption/desorption cycles were conducted repeatedly. The regeneration efficiency can be calculated in terms of the following equation.

$$\text{Regeneration efficiency (\%)} = \frac{\text{Uptake of Pb(II) in the second cycle}}{\text{Uptake of Pb(II) in the first cycle}} \quad (9)$$

The regeneration efficiency achieved $92.4 \pm 3.2\%$ over three cycles. The relatively high regeneration efficiency indicates that APKHC is very promising for Pb(II) removal from wastewater.

4. Conclusion

The adsorption of Pb(II) from aqueous solution using APKHC was investigated. Various impact factors such as temperature, pH, contact time, and adsorbent dosage were optimized. The adsorption of Pb(II) is exothermic and strongly depended on solution pH. The most favorable pH for Pb(II) removal is 5.0. The Pb(II) removal efficiency increased with increasing APKHC dosage and reached the maximum value (ca. 97%) at an APKHC dosage exceeding 2.0 g/L. The adsorption isotherm can be much better defined by the Langmuir equation than the Freundlich equation. The maximum monolayer Pb(II) adsorption capacity

derived from Langmuir isotherm was 98.04 mg/L. Kinetic study results suggested that the pseudo-second-order is more appropriate to describe the adsorption characteristics of Pb(II). Based on the results, it is concluded that APKHC is an attractive candidate for removing Pb(II) from industrial effluents.

Acknowledgments

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