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Investigation of Pb(II) adsorption on a novel activated carbon prepared from hazelnut husk by K_2CO_3 activation

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ABSTRACT

Hazelnut husk, an agricultural waste, was used to prepare an activated carbon (HHPCAC) by chemical activation using K_2CO_3. HHPCAC was characterized by FT-IR spectroscopy, BET surface area determination, SEM, Boehm titration, proximate, and elemental analysis. HHPCAC has a high surface area (980.9 $m^2 g^{-1}$) and contains 2.60 mmol g^{-1} of total acidic functional groups. HHPCAC was used for the removal of Pb(II) from aqueous solutions by investigating a number of effective factors such as initial pH, contact time, dosage, and initial concentration. Adsorption of Pb(II) ions is highly dependent on pH of the solution, and its optimal value was found to be 5.0. Time to equilibrium was found as 20 h. Kinetics of Pb(II) adsorption on HHPCAC followed pseudo-second-order model. Adsorption equilibrium data were analyzed by Langmuir and Freundlich models. Maximum adsorption capacity of HHPCAC for Pb(II) was calculated as 109.9 mg g^{-1} using Langmuir equation. From the findings, it could be concluded that HHPCAC is a feasible adsorbent for the removal of Pb(II) ions from aqueous solutions.

Keywords: Adsorption; Removal; Activated carbon; Hazelnut husk; Lead; Agricultural wastes

1. Introduction

Unregulated and rapid industrialization, which is widely encountered in many developing countries, results in increasing environmental pollution via discharge of industrial waste water without any treatment into receiving environmental water body, such as rivers, streams, or lakes. Environmental pollution with metal ions is mainly caused by metal process industries such as metal plating, mining and metallurgical manufacturing [1–3]. It is well known that metal ions are not biodegradable like organics and can also enter into food chain and reach human metabolism resulting in the accumulation of metal ions in human body [2–4]. High concentration of all metal ions in human body tissues exhibits toxicological properties. Especially, lead (Pb) is known as highly toxic even if at trace

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level. Pb(II) ions damage nervous system, increase blood pressure, and decrease kidney function, which are mainly detrimental effects to human beings [5].

Several methods including ion-exchange, reverse osmosis, nanofiltration, precipitation, evaporation, electrolysis, and adsorption were proposed for the treatment of industrial wastewater containing heavy metal ions [1,6]. Adsorption procedure is widely preferred due to its low cost, high efficiency, and ease of usage [3,7]. Activated carbon (AC) is a widely employed sorbent in the adsorption method. Although commercial AC has large surface area and high adsorption capacity, it is not very useful because of its high cost. Hence, the preparation of low-cost AC is a very important task [8,9]. For this purpose, recently numerous researchers have attempted the preparation of AC's using various agricultural wastes including tea industry waste [10], durian shell [11], pomegranate shell [12], sugarcane molasses [13], coconut shell [14], Prunus domestica [15], pine cone [16], bamboo [17], rice husk [18], olive stone [19], cacao (cocoa) shell [20], cotton seed [21], cow bone [22], dates stone [23], harmal seed residue [24], coconut husk [25], and cashew nut shell [9]. However, despite good adsorption capacity of many AC's reported in the literature, some of them are not advantageous in economic terms because of agricultural wastes which are economically valuable such as wheat bran [26] and sugar beet molasses [27]. In this context, hazelnut husk (HH) could be considered a very useful precursor because of its zero value or very low purchasing cost.

Hazelnut is cultivated in Italy, Greece, Spain, Georgia, Azerbaijan, Iran, USA, and primarily Turkey. Hazelnut exists in the form of clusters (from single to penta) inside a shell surrounded by a green husk. As hazelnut grows, its green husks start to turn yellow and then to brown. After hazelnuts are collected and brought to threshing field, the shelled hazelnuts and husks are separated using thresher. After drying of shelled hazelnuts under the sun, the product is forwarded to food industries. But HH remaining in threshing field is not employed for any purpose by the producers and is generally eliminated by the burning process which takes place very slowly and lasts for several days. Destroying of HH in this manner causes air pollution by CO_2 emission which can be prevented using HH as a precursor for the preparation of AC. Another advantage of HH is that it is a renewable material generated in large amounts every year. Annual production of HH in Turkey is estimated to be 140×10^3 tones. Hence, HH is a superior precursor for the preparation of AC because of its abundance, low cost, renewability, and local accessibility [28–34]. It is also necessary to note that HH and hazelnut shell should not be confused with each other. Although there are numerous studies on hazelnut shell reported in the literature [35,36], the number of studies on use of HH as a precursor for the preparation of AC is limited. Adsorption properties of activated carbons prepared from HH using $ZnCl_2$ [28], H_3PO_4 [29], and H_2SO_4 [32] were reported in the literature. However, since adsorption properties and physical performance of activated carbons depend on the precursor as well as activation method used, the objective of this study is to observe physical and chemical properties and adsorption performance of AC prepared from HH using K_2CO_3 which is one of the widely used chemicals for the preparation of AC by chemical activation method.

In this study, a novel activated carbon (HHPCAC) was prepared from HH using K_2CO_3 characterized by BET surface area determination, CHN elemental analysis, pH_{PZC} assay, surface functional group determination, FT-IR spectroscopy, SEM image, etc. Performance of the HHPCAC for the removal of Pb(II) ions from aqueous solutions was evaluated. Pb(II) adsorption experiments were performed batchwise by investigating various factors including pH, dosage, contact time, and initial Pb(II) concentration. Pb(II) adsorption kinetic was evaluated by pseudo-first-order and pseudo-second-order models. Pb(II) equilibrium was analyzed by Langmuir and Freundlich equations.

2. Materials and methods

2.1. Instruments and reagents

Flame-atomic absorption spectrometer (AAS, Shimadzu AA6701F) was used to determine Pb(II) concentration. Temperature-controlled batch shaker (IKA, KS 4OOOi) was used to carry out adsorption experiments. pH of the solutions was determined using a Schott CG 840 pH meter. Pyrolysis of HH was performed using tube furnace (Protherm, Turkey) under N_2 atm. FTIR spectra (4,000–600 cm^{-1}) were recorded on a Perkin-Elmer Spectrum100 FTIR spectrometer by ATR (with a diamond-protected attenuated total reflectance crystal unit). The surface morphology of HHPCAC was examined using Field Emission Scanning Electron Microscopy (FESEM-Carl Zeiss, Supra 40 VP) with an accelerating voltage of 10 kV. The samples were sputter coated with platinum (Quorum Q 150 R ES DC Sputter). Elemental analysis of HHPCAC was performed using LECO CHNS elemental analyzer, (LECO Corp., Michigan, USA). BET surface area determination of HHPCAC was carried out using NOVA 2200e BET surface area analyzer (Quantachrome Instruments, Florida, USA) based on the N_2 adsorption at 77 K.

All chemicals used in the study were of AR grade. In all stages of the study, deionized water obtained from Millipore Milli-Q system (chemical resistance 18 MΩ cm^{-1}) was used. Pb(II) stock solution was prepared from $Pb(CH_3COO)_2·3H_2O$ at 1,000 mg L^{-1}. The standard and working solutions of Pb(II) were prepared by dilution of the stock solution. The pH of working solutions was adjusted to the desired value with diluted (0.1 M) HNO_3 or NaOH solutions.

2.2. Preparation and characterization of activated carbon

HH supplied from the province of Sakarya, Turkey were washed and dried in open air, ground, and sieved. Twenty grams of K_2CO_3 was added to 20 g of HH particles of 0.106–0.300 mm. Then, 125 mL of deionized water was added and thoroughly agitated. It was allowed to stand at 60˚C in drying oven for 24 h and then dried at 110˚C for 24 h. The resultant mixture was pyrolyzed under nitrogen atmosphere (nitrogen gas flow rate; 100 mL min^{-1}) at 600˚C [37]. The obtained AC was first washed with hot deionized water and then with 0.1 M HCl solution. It was repeatedly washed with deionized water until no chloride ion remains in washing waters. The presence of chloride ion in washing waters was checked by 0.1 M $AgNO_3$. Resultant AC was dried at 105˚C for 24 h and used in the following experiments.

In order to characterize the HHPCAC, a number of physicochemical properties were determined. Moisture, ash content, and water solubility were determined according to the standard methods [38]. The point of zero surface charge of HHPCAC was determined using a solid addition method as described in the literature [39,40].

2.3. Adsorption procedure

Adsorption of Pb(II) ions on HHPCAC was performed by batch method. For this purpose, 50 mL of Pb(II) solution with various specified concentrations were added to a specified amount (50 mg, unless otherwise stated) of HHPCAC, and the resultant suspensions were agitated at 200 rpm at 25˚C for a specified period (for 20 h in all experiments, except for the experiment on effect of agitation time). The Pb(II) concentrations varied between 50 and 300 mg L^{-1}, the amounts of HHPCAC varied between 50 and 300 mg, and agitation time varied between 5 min and 30 h. Unadsorbed Pb(II) concentration in the solution was determined by FAAS. Amount of Pb(II) adsorbed per gram of HHPCAC (q_e) and percentage of adsorption were calculated by the following equations [10,32], respectively:

$$q_e = \frac{(C_o - C_e) \times V}{m} \tag{1}$$

$$\text{Adsorption } \% = \frac{C_o - C_e}{C_o} \times 100 \tag{2}$$

where q_e is the amount of Pb(II) adsorbed per gram of HHPCAC (mg g^{-1}); C_o is the initial Pb(II) concentration (mg L^{-1}); C_e is the concentration of remained Pb (II) in the solution (mg L^{-1}); V is the volume of Pb(II) solution (mL); and m is the amount of HHPCAC (g).

3. Results and discussion

3.1. Characterization of HHPCAC

Carbon content of HHPCAC was found to be 79.72% indicating that a highly carbonaceous sorbent material was obtained. Surface functional groups were found to be 1.10 mmol g^{-1} for carboxyl group, 0.85 mmol g^{-1} for phenolic group, 0.65 mmol g^{-1} for lactonic group, and 2.60 mmol g^{-1} for total acidic groups which can play an important role for Pb(II) uptake from aqueous solutions. Moisture (wt.%), volatile matter (wt.%), fixed carbon (wt.%), and ash (wt.%) were 3.47, 23.01, 76.31, 0.96, and 0.68, respectively. Also, BET surface area (m^2 g^{-1}) and pH$_{pzc}$ were found to be 980.9 and 4.47, respectively. Production yield of HHPCAC was calculated to be 25.5. High surface area, production yield, carbon content, and low ash content are desired properties of ACs for the removal of pollutants from aqueous solutions. Hence, HH is a very useful precursor for the preparation of AC using K_2CO_3 activation.

According to SEM image presented in Fig. 1, it can be concluded that HHPCAC has a porous structure. The FTIR spectroscopy is widely utilized in order to obtain idea related to the surface functional groups of sorbents. In the spectrum of HHPCAC (Fig. 2), the band located at 3,167 cm^{-1} is assigned to the O–H stretching vibrations sourced from phenols, alcohols, or adsorbed water molecules [41]. The asymmetric position and the deformations at the lower wave numbers indicate the presence of strong hydrogen bonds [42]. The broad band at 2,494 cm^{-1} can be attributed to the aliphatic C–H stretching vibrations. The other two major bands at 2,162 and 2,034 cm^{-1} are ascribed to C≡C and C=C bonds which are in the conjugated form [11]. However, the vibration that appeared at 1,560 cm^{-1} can be associated with the free C=C bonds

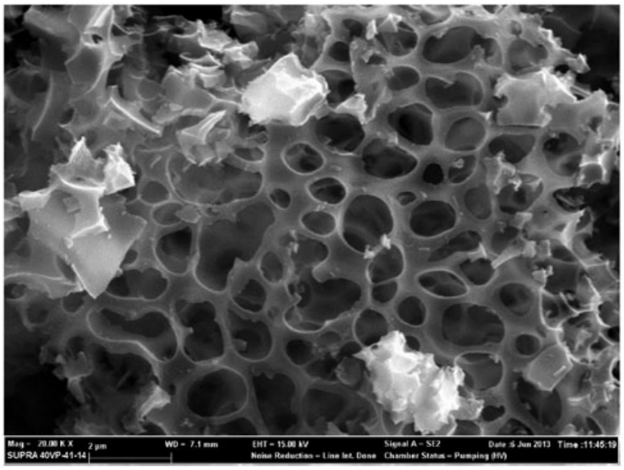

Fig. 1. SEM image of HHPCAC (magnification 20,000×).

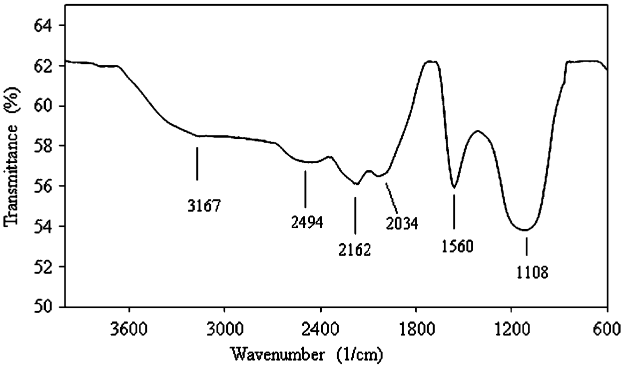

Fig. 2. FTIR spectrum of HHPCAC.

of aromatics or unsaturated aliphatic carbon chains. The broad peak at 1,108 cm^{-1} is assigned to the C–O vibrations of phenols, alcohols, or anhydrides which occurred during the carbonization, as well [43].

3.2. Effect of initial pH

It was demonstrated by numerous studies carried out on adsorption of metal ions using various adsorbents that solution pH significantly affects adsorbed amount of metal ions [36,44,45]. Therefore, variation of adsorption of Pb(II) ions with pH was studied using two Pb(II) solutions with different concentrations (100 and 150 mg L^{-1}) by changing their initial pH between 1.0 and 6.0. The results are depicted in Fig. 3. At pH values of 1.0 and 2.0, adsorption of Pb(II) ions on HHPCAC did not occur. By increasing the pH from 2.0 to 5.0, a gradual increase in Pb(II) adsorption was observed. At pH 5.0 and 6.0, almost identical adsorption was found. Therefore, pH 5.0 was selected as the optimal value for the removal of Pb(II) ions using HHPCAC.

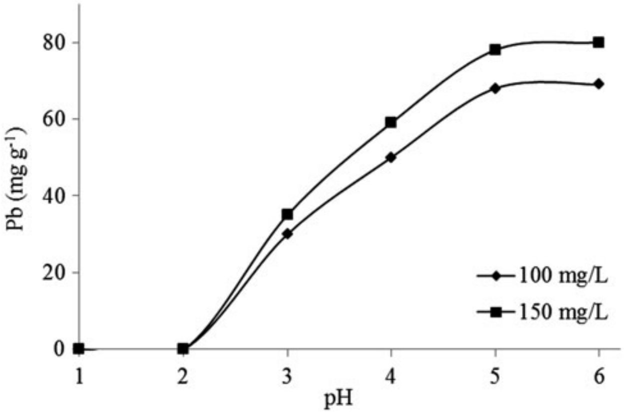

Fig. 3. Effect of initial pH on the adsorption of Pb(II) by HHPCA.

Poor adsorption of Pb(II) at low pH values is due to competition between H_3O^+ and Pb^{2+} for adsorption on HHPCAC surface and protonation of the functional groups on HHPCAC surface, which hinders the interaction between Pb(II) and HHPCAC surfaces. Also, at a lower pH than pH_{PZC}, the surface of HHPCAC is positively charged and electrostatic attraction between the same charged two species is not favored. On the other hand, the level of H_3O^+ ions is decreased by increasing pH values. So, HHPCAC surface is charged negatively at pH values higher than 4.47. At the same time, due to decreasing H_3O^+ concentration, the competition between H_3O^+ and Pb^{2+} results in increase in the adsorption of Pb(II). Although increasing pH increases Pb(II) adsorption, reaching basic levels can cause precipitation of Pb(II) ions and thus, removal of Pb(II) ions takes place along with both precipitation and adsorption mechanisms, which negatively affect the determination adsorption capacities of adsorbents [9,46]. Therefore, adsorption of Pb(II) ions on HHPCAC was studied up to a maximum pH value of 6.0.

3.3. Effect of time and Pb(II) adsorption kinetics

Influence of agitation time on adsorption of Pb(II) ions on HHPCAC was investigated using two initial concentrations of Pb(II), namely 100 and 150 mg L^{-1} over a time range of 5 min–30 h. As seen in Fig. 4, the adsorption of Pb(II) increased with increasing time and the equilibrium was reached at 20 h for two initial concentrations and the following experiments were all conducted at a contact time of 20 h. Although it takes a long time to reach equilibrium, minimum 66% of Pb

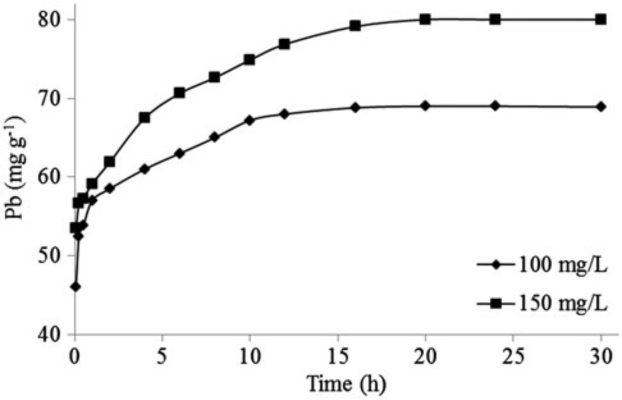

Fig. 4. Effect of agitation time on the adsorption of Pb(II) by HHPCAC.

(II) removal for two initial concentrations was achieved within the first 5 min.

Adsorption kinetics of Pb(II) ions onto HHPCAC was investigated using Lagergren first-order model [47] and pseudo-second-order model proposed by Ho and McKay [48].

Pseudo-first-order model is represented by:

$$\ln\,(q_e - q_t) = \ln q_e - k_1 t \tag{3}$$

where q_e (mg g^{-1}) and q_t (mg g^{-1}) are the amounts of Pb(II) adsorbed on HHPCAC at equilibrium and at any time t, respectively; and k_1 (h^{-1}) is the rate constant of the first-order model. The values of k_1 and q_e can be computed from the slope and intercept of a linear plot of ln ($q_e - q_t$) against t, respectively.

Pseudo-second-order model is given by the following equation:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

where k_2 (g mg^{-1} h^{-1}) is the rate constant of the second-order equation; q_e (mg g^{-1}) is the maximum sorption capacity; and q_t (mg g^{-1}) is the amount of adsorption at time t (h). The values of q_e and k_2 can be calculated from the slope and intercept of a plot of t/q_t vs. t, respectively [10,32].

The constants and correlation coefficients calculated from both models are given in Table 1. Although high r^2 values were obtained from both models, experimental q_e values correlate with predicted q_e values of pseudo-second-order model better than those calculated for the pseudo-first-order model. Therefore, it was concluded that Pb(II) adsorption on HHPCAC follows the pseudo-second-order model [10,32,49].

3.4. Effect of HHPCAC dosage

In order to study the effect of HHPCAC dosage, the sorbent amount ranging from 50 to 250 mg was added to 50 mL of Pb(II) solution with a concentration of 200 mg L^{-1} and agitated for 20 h. The results are shown in Fig. 5. Increasing HHPCAC dosage decreases the amount of Pb(II) adsorbed as well as increases adsorption percentage. Decrease in amount of Pb(II) adsorbed was expected because reverse proportionality between amount adsorbed and amount of adsorbent was reported in previous studies in the literature [50]. Moreover, increase in adsorption percentage is also caused by an increase in the number of binding sites where Pb(II) ions can be adsorbed [51].

3.5. Effect of Pb(II) initial concentration

Influence of Pb(II) initial concentration on adsorption of Pb(II) was examined at two dosages namely 50 and 75 mg per 50 mL of the solution by varying initial Pb(II) concentration between 50 and 300 mg L^{-1}. According to findings presented in Fig. 6, adsorbed amount of Pb(II) increased with increasing initial Pb (II) concentration because high Pb(II) concentration supports Pb(II) adsorption. On the other hand, increasing initial concentration decreases adsorption percentage in a stepwise fashion and this is a result of inadequate number of adsorption sites on HHPCAC surface where Pb(II) ions can be adsorbed.

3.6. Pb(II) adsorption isotherms by HHPCAC

Pb(II) adsorption equilibrium data were analyzed by Langmuir [52] and Freundlich [53] equations which are given by following equations, respectively.

$$q_e = \frac{K_L q_{\max} C_e}{1 + K_L C_e} \tag{5}$$

$$q_e = K_F {C_e}^{1/n} \tag{6}$$

where q_e (mg g^{-1}) is the amount of the metal adsorbed per unit mass of HHPCAC, C_e (mg L^{-1}) is the equilibrium metal concentration in the solution, $q_{\max}$ (mg g^{-1}) and K_L are Langmuir constants corresponding to maximum monolayer adsorption capacity (L mg^{-1}) and the equilibrium constant or bonding energy, respectively, K_F (mg g^{-1}) and n are Freundlich constants related to adsorption capacity and intensity (or surface heterogeneity), respectively [10,32,41].

Table 1
Parameters of pseudo-first-order and pseudo-second-order kinetic models for the adsorption of Pb(II) onto HHPCAC

		Pseudo-first-order			Pseudo-second-order		
C_o (mg L^{-1})	$q_{e\ exp}$ (mg g^{-1})	$k_1 \times 10$ (h^{-1})	$q_{e\ cal}$ (mg g^{-1})	r^2	$k_2 \times 10^2$ (g mg^{-1} h^{-1})	$q_{e\ cal}$ (mg g^{-1})	r^2
100	69.0	2.6	20.5	0.9638	4.8	69.4	0.9995
150	80.0	1.3	27.2	0.9933	2.5	81.3	0.9996

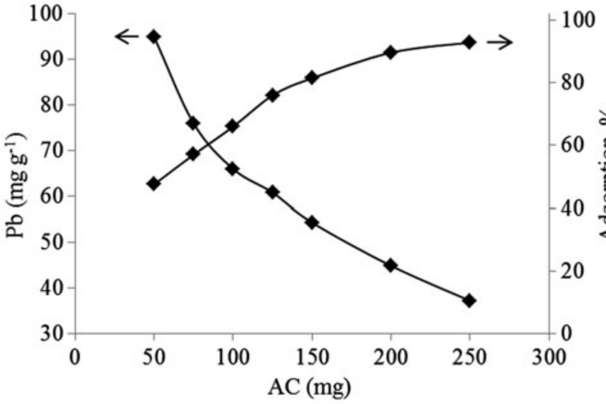

Fig. 5. Effect of HHPCAC dosage on the adsorption of Pb (II).

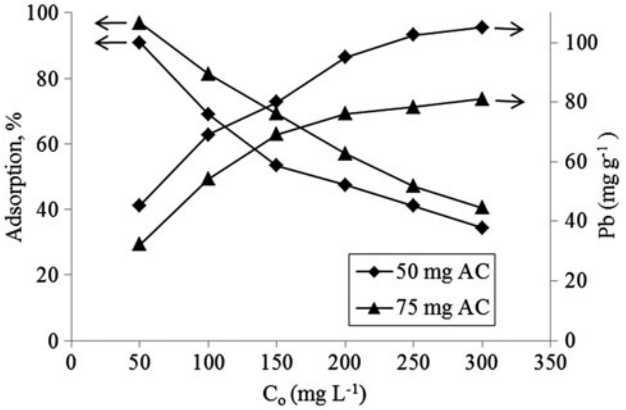

Fig. 6. Effect of Pb(II) initial concentration on the adsorption of Pb(II) onto HHPCAC at various dosages.

Experimental, Langmuir, and Freundlich plots for Pb(II) adsorption are shown in Fig. 7. The constants and correlation coefficients obtained from both isotherms are summarized in Table 2. From the figures, it can be seen that points at low Pb(II) concentration are better estimated from Freundlich equation, while q_e values corresponding to high concentrations fit Langmuir isotherm better. Nevertheless, r^2 values for Langmuir isotherm are higher than those for Freundlich isotherm, so adsorption of Pb(II) ions on HHPCAC fits Langmuir model better [54,55]. Maximum adsorption capacity of HHPCAC for Pb(II) ions was predicted to be 109.9 mg g^{-1} by Langmuir model.

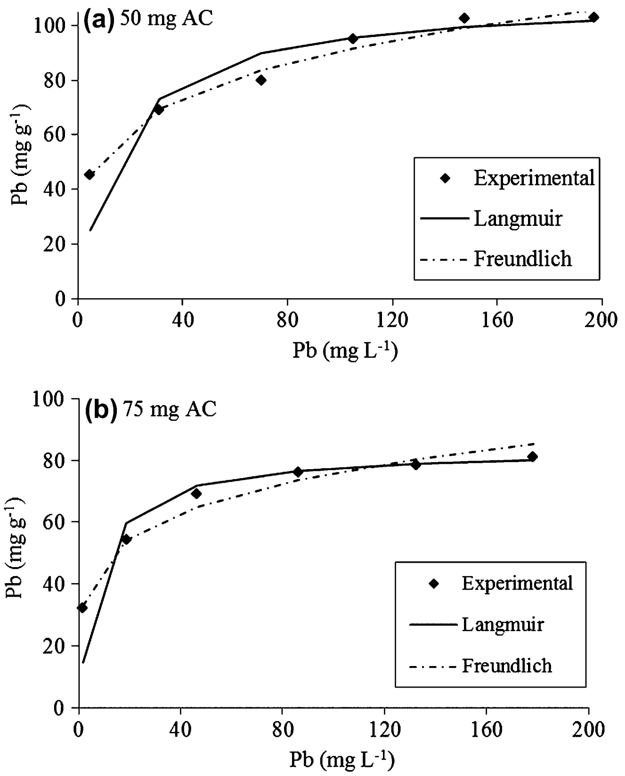

Fig. 7. Experimental, Langmuir, and Freundlich isotherms for the adsorption of Pb(II) onto HHPCAC at various dosages, (a) 50 mg and (b) 75 mg.

In order to investigate whether the adsorption is favorable or not, R_L values are used. R_L, separation factor or equilibrium parameter, is a non-dimensional constant and can be calculated by the following equation:

$$R_L = \frac{1}{1 + K_L C_o} \tag{7}$$

Table 2
Parameters of Langmuir and Freundlich isotherms for the adsorption of Pb(II) onto HHPCAC at various dosages

	Langmuir constants			Freundlich constants		
Amount of HHPCAC (mg)	$q_{\max}$ (mg g^{-1})	K_L (L mg^{-1})	r^2	K_F (mg g^{-1})	n	r^2
50	109.9	0.06	0.9911	31.9	4.4	0.9895
75	83.3	0.13	0.9981	30.0	5.0	0.9867

Table 3
Pb(II) uptake capacities of various adsorbents given in the literature

Adsorbent	Pb(II) adsorption capacity (mg g^{-1})	Reference
Cashew nut shell treated with H_2SO_4	408.6	[9]
Poly(m-phenylenediamine) spheroids	240.0	[56]
Modified orange peel	209.8	[57]
Modified spent Lentinus edodes	59.17	[3]
Melocanna baccifera roxburgh AC	53.76	[58]
Tamarind wood AC	43.85	[59]
Apricot stone AC	21.38	[60]
Hazelnut husk AC by $ZnCl_2$	13.05	[28]
Mentha arvensis waste biomass	111.97	[61]
Red rose waste biomass	112.0	[62]
Citrus reticulata waste biomass	238.09	[63]
HHPCAC	109.9	Present study

where C_o (mg L^{-1}) is the initial Pb(II) concentration in the solution and K_L is Langmuir constants corresponding to maximum monolayer adsorption capacity (L mg^{-1}). The adsorption is accepted as favorable in the case of $0 < R_L < 1$ and unfavorable in the case of $R_L > 1$. If the R_L is calculated to be zero, the adsorption is irreversible. The values of 1 for R_L show linearity of adsorption [10]. In this study, R_L values for adsorption of Pb(II) onto HHPCAC was found to be in the range of 0.04 and 0.83 showing a favorability of the adsorption.

Pb(II) adsorption of an AC prepared from HH using $ZnCl_2$ was reported as 13.05 mg g^{-1} [28], while Pb(II) adsorption capacity of AC prepared using K_2CO_3 was found as 109.9 mg g^{-1}, which suggest that preparation procedure and activating agent significantly affect the adsorption properties of the activated carbons. If it is intended to prepare an AC from HH for the removal of Pb(II) ions, K_2CO_3 should be used as chemical activation agent. Pb(II) adsorption capacity of HHPCAC was compared with capacities of some other sorbents reported in the literature [3,9, 56–63]. As the data in Table 3 show there are sorbents with either higher or lower adsorption capacities than HHPCAC, however, HHPCAC can also be considered an AC with high Pb(II) adsorption capacity.

4. Conclusion

HH could be used for the preparation of AC bearing high functional group content. Adsorption Pb(II) onto HHPCAC is highly dependent on the pH of aqueous solution and contact time. Optimum pH was determined as 5 and time to equilibrium was found as 20 h. Experimental data follow Langmuir model better than Freundlich model and Pb(II) adsorption kinetics follows pseudo-second-order model.

In conclusion, HH, an agricultural waste, can be converted to an efficient adsorbent using K_2CO_3 activation method for the removal of Pb(II) ions from aqueous solution.

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