Journal of Hazardous Materials 174 (2010) 251–256

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

Studies on adsorption of mercury from aqueous solution on activated carbons prepared from walnut shell

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ARTICLE INFO

Article history:
Received 19 February 2009
Received in revised form 20 August 2009
Accepted 9 September 2009
Available online 19 September 2009

Keywords:
Adsorption
Mercury
Sorbent
Walnut shell
Kinetic

ABSTRACT

The adsorption ability of a powdered activated carbons (PAC) derived from walnut shell was investigated in an attempt to produce more economic and effective sorbents for the control of Hg(II) ion from industrial liquid streams. Carbonaceous sorbents derived from local walnut shell, were prepared by chemical activation methods using $ZnCl_2$ as activating reagents. Adsorption of Hg(II) from aqueous solutions was carried out under different experimental conditions by varying treatment time, metal ion concentration, pH and solution temperature. It was shown that Hg(II) uptake decreases with increasing pH of the solution. The proper choice of preparation conditions were resulted in microporous activated carbons with different BET surface areas of 780 (Carbon A, 1:0.5 $ZnCl_2$) and 803 (Carbon B, 1:1 $ZnCl_2$) m^2/g BET surface area. The monolayer adsorption capacity of these particular adsorbents were obtained as 151.5 and 100.9 mg/g for carbons A and B, respectively. It was determined that Hg(II) adsorption follows both Langmuir and Freundlich isotherms as well as pseudo-second-order kinetics.

1. Introduction

Mercury is regarded as one of the most harmful metals found in the environment, hence, its removal from wastewaters, before these being discharged into the environment, is necessary [1,2]. The MCLG (Maximum Contaminant Level Goals) for mercury has been set at 2 parts per billion (ppb) because EPA believes this level of protection would not cause any of the potential health problems described below [3]. Consequently, removal of mercury in water and wastewater assumes importance. Though the flux of mercury into the aquatic system has declined in recent years, there is still a lack of an effective, cheap means for the treatment of mercury containing wastewaters. Activated carbons are widely used as adsorbents in technologies related to pollution abatement, pharmaceutical and food industries due to their highly porous structure and large adsorption capacity. In the last year, special emphasis on the preparation of activated carbons from different waste materials has been given because of the growing interest in low-cost activated carbons, especially for applications concerning treatment of drinking and wastewaters [4–14]. The characteristics of activated carbon depend on the physical and chemical properties of the precursor as well as on the activation method. There are two methods of preparing activated carbons: physical and chemical activation.

The physical activation method involves: carbonization of raw material and activation at high temperature in carbon dioxide or water vapor [15]. Chemical activation is a well-known method for the preparation of activated carbon, which has been the objective of numerous studies within the last few years [16–26] as it presents several advantages compared to the so-called physical activation. In addition to the starting material and the oxidizing agent, activation time and temperature affect the structural properties of the resulting activated carbon. Many researchers observed that BET surface area and pore volume increased with activation time and temperature [27–29]. The advantage of chemical activation over physical activation is that it is performed in one step and at relatively low temperatures. The most important and commonly used activating agents are phosphoric acid, zinc chloride and alkaline metal compounds, such as KOH [25,30]. The main scope of this communication is to investigate the removal of Hg(II) from aqueous solution by activated carbons prepared from Iranian walnut shell which is cheap and available in our country and it is one of the agriculture waste that has been used for useful way [31]. Several important factors such as temperature, initial concentration, pH and also isotherm models were investigated in the present study of mercury adsorption from water.

2. Materials and methods

Walnut shell, collected from a local source, was dried and ground. The powdered samples (below mesh No. 170) were impregnated with $ZnCl_2$ concentrated solution (136.28 g/mol, 98%)

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doi:10.1016/j.jhazmat.2009.09.044

Nomenclature

q	amount adsorbed (Eq. (1))
C_0	initial metal ion concentration (Eqs. (1) and (4))
C_e	equilibrium concentration (Eqs. (1)–(3))
V	volume of the solution (Eq. (1))
W	weight of sorbent (Eq. (1))
q_e	equilibrium amount adsorbed (Eqs. (2), (3), (5) and (6))
K_f	Freundlich constants related to adsorption capacity (Eq. (2))
n	Freundlich constants related to adsorption intensity (Eq. (2))
q_m	Langmuir constant related to the maximum adsorption capacity (Eq. (3))
b	Langmuir constant related to the energy or net enthalpy of adsorption (Eqs. (3) and (4))
R_L	parameter indicating the type of isotherm (Eq. (4))
q_t	amount adsorbed at time t (Eqs. (5) and (6))
K_1	rate constant of first-order adsorption (Eq. (5))
K_2	rate constant of second-order adsorption (Eq. (6))
t	adsorption time (Eqs. (5) and (6))

in a (shell: $ZnCl_2$) weight ratio of 1:0.5 (Carbon A) and 1:1 (Carbon B). The resulting black product was then dried in an oven at 120 °C for at least 5 h. Impregnated sample was placed on a ceramic boat, inserted in a tubular furnace. The sample was heated to the carbonization temperature under N_2 flow at the rate of 5 °C/min. After carbonization, the sample was cooled down in N_2 atmosphere. The carbonized sample was washed several times sequentially with hot and finally cold distilled water to remove any residual chemicals. The activated carbon product was then dried in an oven at 130 °C. The adsorbent particle size distribution was obtained and the mean diameter was about 0.088 mm. All the chemical and reagents used were of analytical reagent grade obtained from Merck Company. The characteristics of the walnut shell are provided in Table 1.

2.1. Adsorption studies

The adsorption of Hg(II) from aqueous solutions by walnut shell activated carbon was studied as follow. Each adsorbate containing solution was prepared by dissolving necessary amount of $HgCl_2$ in the distilled water. Each solution was then diluted to obtain standard solutions containing 9.7–107 mg/l of Hg(II) prior to adsorption experiments. Batch adsorption studies were carried out with 0.05 g sorbent and 50 ml of Hg solution with a desired concentration at pH 5 in two conical flasks, simultaneously. The flasks containing adsorbent and adsorbate were agitated for predetermined time intervals at 29 °C on a mechanical shaker with 720 rpm. At the end of agitation, the suspensions were filtered by the aid of filter paper. The amount of Hg(II) ion in the final 25 ml volume was determined by atomic absorption spectrophotometer (Varian, spectra-110-220/880 Australia Pty. Ltd.) equipped with a Zeeman atomizer. The obtained results for two similar solutions were averaged and then reported.

Table 1
Characteristics of walnut shell.

Characteristics	Values
pH	7.5
Moisture content	1.97%
Porosity	52%
Color	Brown
Specific gravity	1.1–1.5
C content	42.6%
H content	4.74%

Table 2
Textural characteristics of $ZnCl_2$-impregnated activated carbons.

Sample	BET surface area (m^2/g)	Pore volume (cm^3/g)	Average pore size (nm)
Carbon A	780	0.426	164
Carbon B	803	0.387	17.6

2.2. Iodine number

The surface activity of activated carbons towards iodine was determined by using the DIN 53582 standard method. The iodine No. of the two samples are obtained as Carbon A = 737 mg/g and Carbon B = 760 mg/g.

2.3. Sample characteristics

Textural characteristics of samples were determined by nitrogen (N_2) adsorption at −196 °C with ASAP-1100, micromesetics. The specific surface area was calculated from the isotherms by using the Brunauer–Emmett–Teller (BET) equation.

The pore volume was found from the amount of N_2 adsorbed at a relative pressure of 0.99. Sharp increase of the adsorbed volume at low pressure is due to the enhanced potential of micropores, and gradual increase at higher pressures indicates the presence of mesopores in the sample. The average pore diameter was calculated from four times of the pore volume over the BET surface area. The

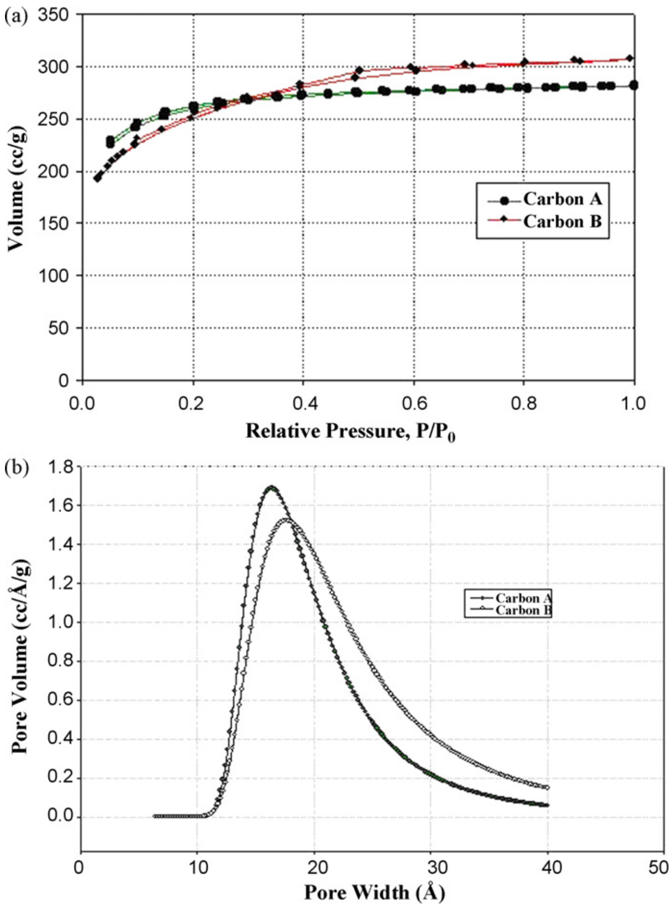

Fig. 1. (a) N_2 adsorption isotherm at 77 K for walnut shell activated carbons. (b) Pore volume distribution for walnut shell activated carbons.

Table 3
The Boelun analysis results of activated carbons and raw materials (mequiv./g).

Material	Basic groups	Carboxylic groups	Lactonic groups	Phenolic groups	Total acidic groups
Walnut shell	0.52	0.45	0.49	0.39	1.33
Carbon A	0.42	0.54	0.48	0.35	1.37
Carbon B	0.29	0.72	0.42	0.30	1.44

textural characteristics of $ZnCl_2$ impregnated activated carbons are seen in Table 2 and Fig. 1.

The types and amounts of surface functional groups that present in the activated carbon samples were determined by the Boehm analysis and the results were given in Table 3. As shown in the table, the amount of acidic and basic group is as follow: walnut shell: basic > lactone > carboxyl > phenol groups, Carbon A: carboxyl > lactone > basic > phenol groups and Carbon B carboxyl > lactone > phenol > basic groups.

3. Results and discussion

3.1. Influence of pH

The pH of the solution plays a vital role in any adsorption process. The effect of pH of the solution on the extent of adsorption can be seen in Fig. 2. The pH was adjusted by the addition of HCl and NaOH. It is seen that the removal efficiency of Hg(II) decreases with increasing pH. Some authors indicated that surface charge is an important factor for the adsorption of metal ions [1,8,9]. The surface charge is very much dependent on the pH of the solution [4]. By increasing OH^- functional group, competing between mercury ions and this group starts and OH^- occupies active sites of the adsorbent. Therefore, accessed surface area and subsequently adsorption of mercury ions will be decreased.

In order to determine the effect of pH on Hg(II) removal by two different activated carbons, experiments were conducted using two initial concentrations of 9.7 and 44.6 mg/L. The effect of pH on mercury adsorption are illustrated in Fig. 2. The maximum adsorption is observed at pH = 2 for these carbons. In general, the results indicated that adsorption is highly pH-dependent. Similar results have been reported in previous studies [9,32,33]. From Fig. 2a (Carbon A) it is seen that, for the dilute solution (9.7 ppm) the removal efficiency was decreased with a mild slope, but for the concentrated one (44.6 ppm), it was constant in the initial stage (pH range of 5–8) and then sharply decreased. It is seen from Fig. 2b (Carbon B) that for two different initial concentrations, the removal efficiency was decreased with a mild slope. Generally, the adsorption efficiency of Hg(II) has decreased with increasing pH. It can be concluded that at any pH, Hg(II) removal by different modifications of walnut adsorbent is very much greater for the concentrated solution than the diluted one. This is the result of stronger adsorption driving force in the concentrated solution. The other result concluded from Fig. 2, is that the removal efficiency of mercury by Carbon A is much greater than Carbon B.

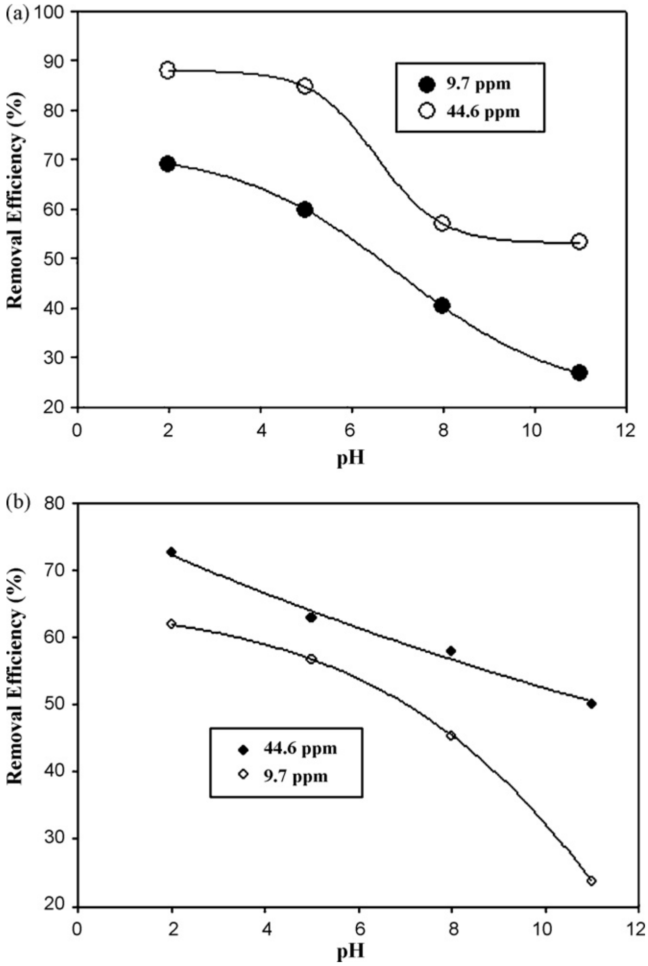

Fig. 2. (a) Effect of pH on the adsorption of Hg (II), Carbon A (time = 15 min, temperature = 29 °C). (b) Effect of pH on the adsorption of Hg (II), Carbon B (time = 15 min, temperature = 29 °C).

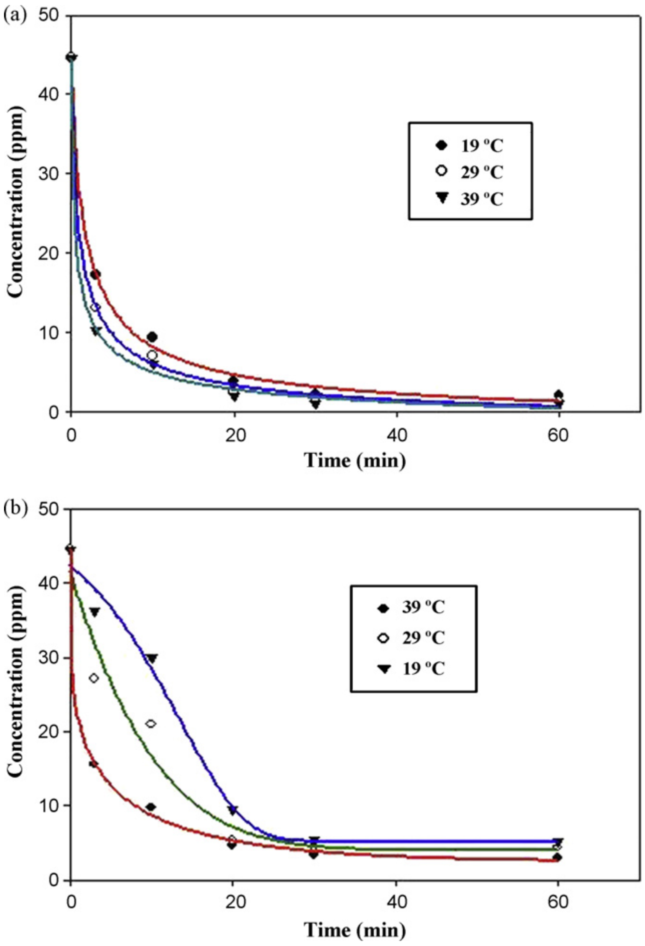

Fig. 3. (a) Effect of temperature of Hg solution, Carbon A. (b) Effect of temperature of Hg solution, Carbon B.

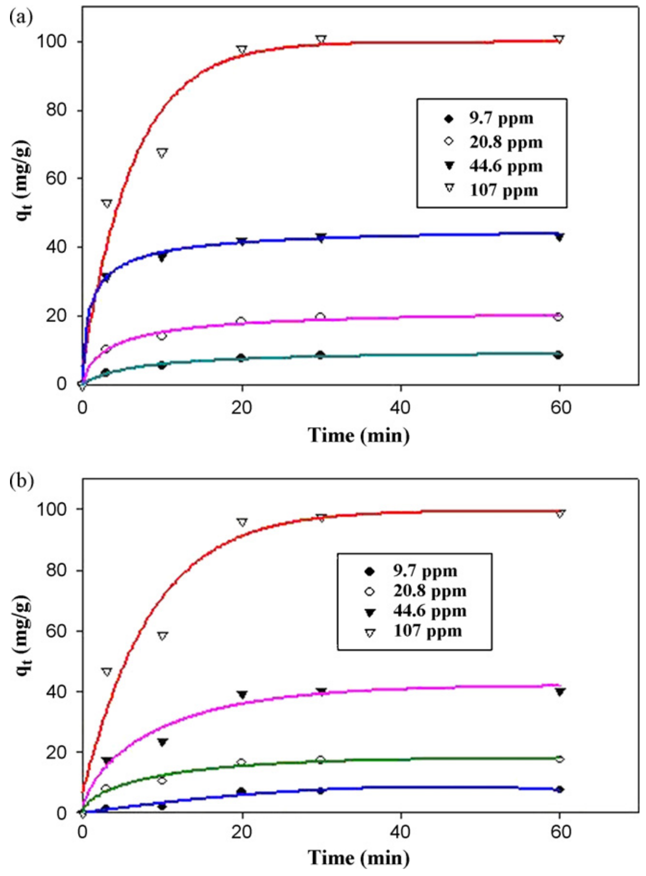

Fig. 4. (a) Effect of initial concentration of Hg solution, Carbon A. (b) Effect of initial concentration of Hg solution, Carbon B.

3.2. Effect of temperature and contact time

Fig. 3 shows the variation of Hg(II) concentration versus time at different sorption temperatures of 19, 29 and 39 °C. Constant parameters of solutions were: ion concentration of 44.6 mg/l, adsorbent dose of 0.05 g and pH = 5. The experimental data show that solution concentration of Hg(II) ions reduces with increase in the temperature indicating an endothermic nature of the sorption processes, while the time required reaching equilibrium remains practically unaffected. Also, the concentration of Hg(II) in solutions decreases with time and attained equilibrium within 30–60 min. It is well recognized that the characteristic of the sorbent surface is a critical factor which affect sorption rate parameters and also diffusion resistance plays an important role in the overall transport of the ions. Increase in the adsorption capacity with temperature suggests that active centers on the surface available for adsorption increase with temperature. This could also be attributed to the change in pore size and enhanced rate of intraparticle diffusion of the solute as diffusion is an endothermic process.

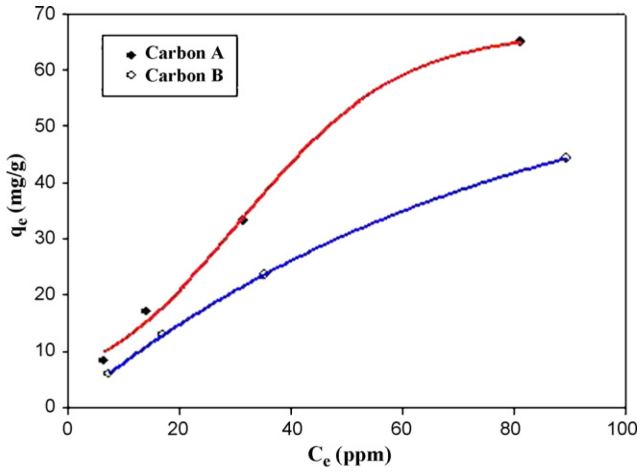

Fig. 5. Adsorption isotherm of mercury on walnut shell activated carbons.

3.3. Effect of initial concentration and contact time

Usually in studies of mercury adsorption, the Hg(II) removal increased almost linearly with the enhancement of Hg(II) concentration. Many researchers have observed similar results. For example, Zhang et al. [34] studied mercury adsorption with sewage sludge carbon and showed that the Hg removal increased linearly Hg(II) concentration. Inbaraj and Sulochana [35] found that the adsorption capacity increased from 21.67 to 85.32 mg/g, while initial Hg(II) concentration increased from 5 to 60 mg/l. Similar results have been also obtained from the study of Yardim et al. [36]. In the present investigation, the effect of initial Hg(II) concentration was studied using solutions with different initial concentrations of 9.7, 20.8, 44.6 and 107 mg/l at 302 K, while keeping all other parameters constant. The results are shown in Fig. 4 for two activated carbons (A and B). It is clear that the sorption amount of Hg(II) increases with increasing the initial ion concentration. Also, the amount of metal ion adsorbed increases sharply with time in the initial stage (0–20 min range) and then gradually increase, to reach equilibrium in approximately 60 min. It is seen that further increase in contact time has a negligible effect on the amount of ion sorption. The equilibrium time was found to be independent of the initial concentration. Based on these results, the agitation time was fixed at 20 min for the rest of batch experiments to make sure that the equilibrium was reached. Increase in the uptake capacity of two modifications of walnut sorbent (A and B) with increasing initial ion concentration may be due to the higher probability of collisions between ions and sorbents particles. Variation in the extent of adsorption may also be due to the fact that initially all active sites on the surface of activated carbons were vacant and metal ion con-

Table 4
Adsorbed amounts of Hg (II) (mg/g) at different initial concentration after 60 min by two activated carbons derived from walnut shell.

Sample	Initial concentration (ppm)	q_t (mg/g)
Carbon A	9.7	8.7
	28.8	19.6
	44.6	43.39
	107	101
Carbon B	9.7	7.5
	20.8	17.5
	44.6	40.3
	107	98.8

Table 5
Fitted isotherm models for the adsorption of Hg(II) on two activated carbons derived from walnut shell.

Adsorbent type	Model	Parameters	R^-
Carbon A	Freundlich	$K = 1.8623$	0.99651
		$n = 1.215$	
	Langmuir	$q = 151.5$	0.99812
		$b = 0.0091025$	
Carbon B	Freundlich	$K = 1.2099$	0.99542
		$n = 1.219$	
	Langmuir	$q = 100.9$	
		$b = 0.0087166$	0.99982

Table 6
R_L values for the adsorption of mercury on two types of activated carbon derived from walnut shell at 29 °C.

Sample	Initial concentration (ppm)	R_L
Carbon A	9.7	0.918
	20.8	0.814
	44.6	0.711
	107	0.5066
Carbon B	9.7	0.922
	20.8	0.847
	44.6	0.720
	107	0.517

centration gradient was relatively high. Consequently, the extent of ion uptake decreases significantly by increasing contact time, depending on the reduction rate of vacant sites on the adsorbent surface. Rapid uptake of metal ions by these carbons is one of the parameters that can be taken into consideration for the economical wastewater treatment plant applications. The amount of mercury adsorption is calculated by

$$q = \frac{(C_0 - C_e)V}{W} \tag{1}$$

where q (mg/g) is the amount of ion adsorption by the adsorbent phase, C_0 and C_e are the initial and equilibrium concentrations of mercury ion (mg/l) in the solution, V is the volume of solution (l), and W is the weight of adsorbent (g) in the mixture. Adsorbed amounts per unit weight for Hg(II) removal at 9.7, 20.8, 44.6 and 107 mg/l initial concentrations at 60 min time are shown in Table 4.

3.4. Adsorption isotherms

The adsorption capacity increased while increasing the initial Hg(II) concentration from 9.7 to 107 mg/l. This observed trend is due to the increase in driving force offered by the concentration pressure gradient Fig. 5.

The equilibrium data obtained were modeled with two-parameter Freundlich [37] (Eq. (2)) and Langmuir [38] (Eq. (3)) isotherm equations.

$$q_e = K_f {C_e}^{1/n} \tag{2}$$

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e \tag{3}$$

where K_f and n are Freundlich constants related to adsorption capacity and adsorption intensity. Usually, for a good adsorbent $1 < n < 10$. A smaller value of n indicates better adsorption and formation of relatively strong bond between adsorbate and adsorbent, respectively, q_m (mg/g) is the amount of adsorption corresponding to complete monolayer coverage and b (l/mg) is the Langmuir constant related to the energy or net enthalpy of adsorption. The fitted parameters obtained specific for the present system along with R^2 are reported in Table 5. It is seen that the Langmuir model fitted the results slightly better than the Freundlich model. The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter [39], R_L, defined as

$$R_L = \frac{1}{(1 + bC_0)} \tag{4}$$

where b is Langmuir constant and C_0 is initial metal concentration (mg/l). The value of R_L indicates the shape of isotherm to be either unfavorable ($R_L > 1$) or linear ($R_L = 1$) or favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The R_L values obtained (Table 6) indicate favorable isotherm shape ($0 < R_L < 1$) for adsorption of Hg(II) on walnut shell activated carbon the concentration range studied.

3.5. Kinetic studies

The rate constant for surface adsorption of Hg(II) ion on these two types of activated carbon is also studied under the light of the pseudo-first-order rate expression of Lagergren model and the pseudo-second-order kinetic rate expression of Ho [40]. The integrated form of the Lagergren equation is given by

$$Log\left(\frac{q_e}{q_e - q_t}\right) = \frac{K_1}{2.303} t \tag{5}$$

where q_t (mg/g) is the amount of adsorbed metal ion on the adsorbent at time t, q_e is the equilibrium sorption uptake, and K_1 (min^{-1}) is the rate constant of first-order adsorption. q_e is extrapolated from the experimental data at time $t = \infty$. A straight line of $ln(q_e - q_t)$ versus t suggests the applicability of this kinetic model. q_e and K_1 can be determined from the intercept and slope of the plot, respectively.

The pseudo-second-order kinetic rate expression in the integrated form is

$$q_t = \frac{t}{(1/K_2 q_e^2) + (t/q_e)} \tag{6}$$

where K_2 is second-order rate constants (g mg^{-1} min^{-1}).

The values of different parameters determined from pseudo-second-order and pseudo-first-order kinetic model for mercury ions with their corresponding correlation coefficients are presented in Table 7. The experiments were performed at three different temperatures and with the initial mercury concentration of 44.6 ppm.

The correlation coefficients of the second-order kinetic model are very close to 1, showing that this model is better in explaining the adsorption kinetics of the present case.

The monolayer adsorption capacity of Hg(II) on these two types of activated carbon derived from walnut shell are calculated as 151.51 mg/g for Carbon A, 100.9 mg/g for Carbon B at pH = 5.0 and room temperature of 29 °C. The value of the adsorption capacity is larger than that of the granular activated carbon (0.8 mg/g) [6], antibiotic activated carbon (129 mg/g) [41], Indian almond activated carbon (94.43 mg/g) [35], Waste rubber activated carbon (4 mg/g) [7], Fuller's earth sorbent (1.145 mg/g) [42] and lower than others: coir pith activated carbon(154 mg/g) [12], furfural activated carbon (174 mg/g) [36]. It is seen that our adsorbents (A and B) has a high capacity for Hg(II) removal from the aqueous solutions compared to the other adsorbents.

Table 7
Rate constant values of pseudo-first and second-order kinetic models in adsorption of Hg(II) on two walnut shell ACs.

Adsorbent type	T (°C)	$K_{1,\mathrm{als}}$ pseudo-first-order (min^{-1})	R^2	$K_{2,\mathrm{als}}$ pseudo-second-order (g mg^{-1} min^{-1})	R^2
Carbon A	19	0.1645	0.95697	0.0119	0.99785
	29	0.1500	0.98884	0.0181	0.99848
	39	0.1578	0.90546	0.0256	0.99842
Carbon B	19	0.2149	0.91709	0.0007	0.99653
	29	0.1962	0.93599	0.0039	0.99644
	39	0.1424	0.98785	0.0167	0.99745

4. Conclusion

The present study revealed the feasibility of using new cheap adsorbents derived from an agriculture waste (walnut shell) for the removal of mercury from aqueous solutions. The adsorption behavior is well described by pseudo-second-order kinetic and Langmuir isotherm models. The adsorption capacity of new adsorbents are obtained as 151.5 (Carbon A), 100.9 (Carbon B) mg/g at pH = 5.0 for adsorbent with 0.088 mm particle sizes.

Acknowledgments

The authors are gratefully acknowledged the assistance of the Water supply organization of Khorasan.

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