



# Removal of mercury from water by carbonaceous sorbents derived from walnut shell

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## ABSTRACT

The adsorption ability of a powdered activated carbon (PAC) derived from walnut shell was investigated in an attempt to produce more economic and effective sorbent for the control of Hg(II) ion from industrial liquid streams. Carbonaceous sorbents derived from Iranian walnut shell (WS) were prepared by chemical activation method using ZnCl<sub>2</sub> as an activating reagent. To the best of our knowledge, this adsorbent was not used before for removing mercury from water. Adsorption of Hg(II) from aqueous solutions was carried out under different experimental conditions by varying treatment time, metal ion concentration, adsorbent amount, pH and solution temperature. It was determined that Hg(II) adsorption follows both Langmuir and Freundlich isotherms as well as pseudo-second-order kinetics. It was also shown that Hg(II) uptake decreases with increasing pH of the solution. The proper choice of preparation conditions resulted in a microporous activated carbon with 0.45 g/cm<sup>3</sup> density, 737 mg/g iodine number and 780 m<sup>2</sup>/g BET surface area. The monolayer sorption capacity of this optimum adsorbent was obtained as 151.5 mg/g.

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## 1. Introduction

Different industrial wastewaters containing heavy metals are frequently polluting the aquatic streams. The presence of these heavy metals in the environment is of important concern due to their toxicity and health effects on the humans and living creatures. Therefore, elimination of toxic metals is attended by many researchers. There are various methods for removing heavy metals from aqueous streams such as chemical precipitation [1], reverse osmosis [2], ion exchange [3], coagulation [4], and adsorption [5–10]. Mercury is one of the priority pollutant listed by USEPA as it can easily pass the blood-brain barrier and affect the fetal brain. High concentration of Hg(II) causes impairment of pulmonary function and kidney, chest pain and dyspnea [11,12]. According to the standard, the tolerance limit of Hg(II) for discharge into inland surface waters is 10 µg/L [13] and for drinking water is 1 µg/L [14].

Although the flux of mercury into the aquatic systems has declined in recent years, there is still a lack of effective, cheap means for the treatment of mercury containing wastewaters. Activated carbon is one of the most popular adsorbents for the removal of mercury from aqueous solutions [15,16], although several inexpensive materials such as onion skin [17], waste rubber [18], coconut husk [19], fertilizer waste slurry [20], peanut hull [21], jackfruit peel

[22], coir pith [23] flax shive [24] and sago waste [25] have been tried as efficient and cost-effective precursors.

There are two basic methods for the preparation of popular adsorbents i.e. activated carbons: physical and chemical activation. The physical activation method involves carbonization of raw material followed by activation at high temperature in carbon dioxide, steam or water vapor atmosphere [26–28]. Chemical activation which is a well-known method for the preparation of activated carbon, involves one step heat treatment at lower temperature than physical activation in the presence of some chemical agents. This method has been the objective of numerous studies within the last few years as it presents several advantages compared to the so-called physical activation. Walnut is one of the most common agricultural products with large quantity in Iran. The object of the present work was to investigate the possibility of mercury ion removal from water by activated carbon prepared from the local walnut shell by chemical activation. 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 (WS), collected from a local source, was dried in an oven for about 24 h at 120 °C. Dried sample was powdered, sieved (below mesh No. 170), and impregnated with ZnCl<sub>2</sub> concentrated solution (136.28 g/mol, 98%) in a weight ratio of 1:0.5

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### Nomenclature

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

(shell:ZnCl<sub>2</sub>). 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, and then heated to the carbonization temperature under N<sub>2</sub> flow at the rate of 5 °C/min. After carbonization, the sample was cooled down in N<sub>2</sub> atmosphere. The carbonized sample was washed several times sequentially with hot and finally cold distilled water to remove any residual chemicals. Activated carbon product was then dried in the 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.

#### 2.1. Adsorption studies

The adsorption of Hg(II) from aqueous solutions was studied by using the above activated carbon derived from WS. Adsorbate containing solution was prepared by dissolving necessary amount of HgCl<sub>2</sub> 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 microporous 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 as the experimental data.

#### 2.2. Iodine number

The surface activity of WS activated carbons towards iodine was determined by using the DIN 53582 standard method. The iodine number of the sample was obtained as 737 mg/g.

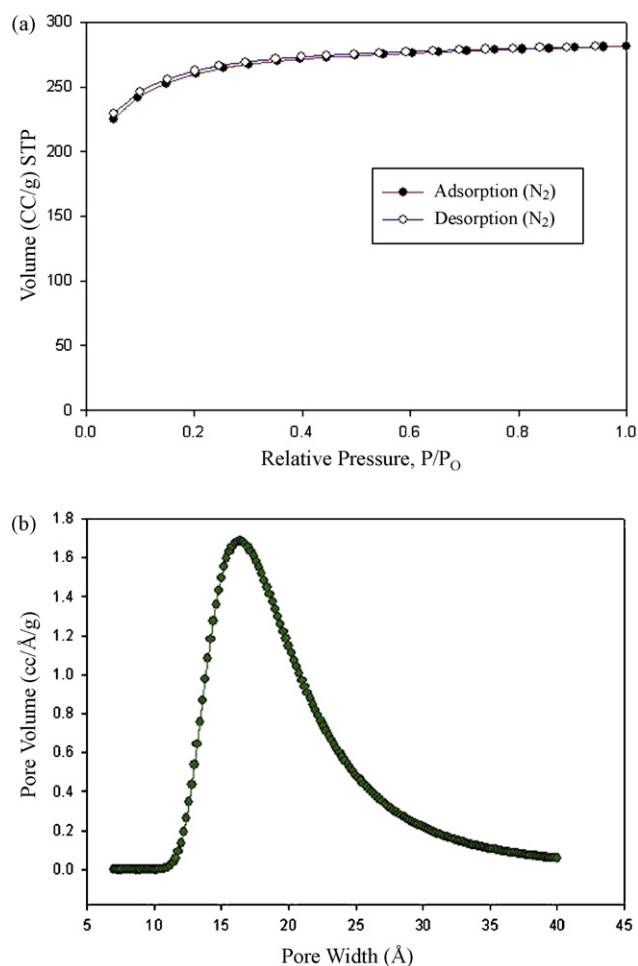


Fig. 1. (a) N<sub>2</sub> adsorption Isotherm at 77 K for WS activated carbon (b) Pore volume distribution for WS activated carbon.

#### 2.3. Sorbent characteristics

The nitrogen adsorption isotherm at 77 K and pore volume distribution of the AC product are presented in Fig. 1. Sharp increase of the adsorbed volume at low pressures is due to the enhanced potential of micropores, and gradual increase at higher pressures indicates the presence of mesopores in the sample. The structural characteristics of WS activated carbon were measured as: BET surface area of 780 m<sup>2</sup>/g, pore volume of 0.426 cm<sup>3</sup>/g, average pore diameter of 16.4 Å, and density of 0.45 g/cm<sup>3</sup>.

#### 2.4. TGA experiment

Thermogravimetric analysis of the WS carbon was performed using a TGA-25 apparatus (Shimadzu, Japan) by heating the sample up to 1000 °C under the air-flow of 55 cm<sup>3</sup>/min (STP) and a heating rate of 10 °C/min. The result is shown in Fig. 2. It is observed from the figure that moisture and ash contents of the sample are 7 and 2.8%, respectively. Weight loss is observed to decrease almost linearly from 400 to 900 °C.

### 3. Results and discussion

#### 3.1. Effect of pH

The effect of pH on the extent of adsorption can be seen in Fig. 3. The pH of each solution was adjusted by addition of HCl and/or

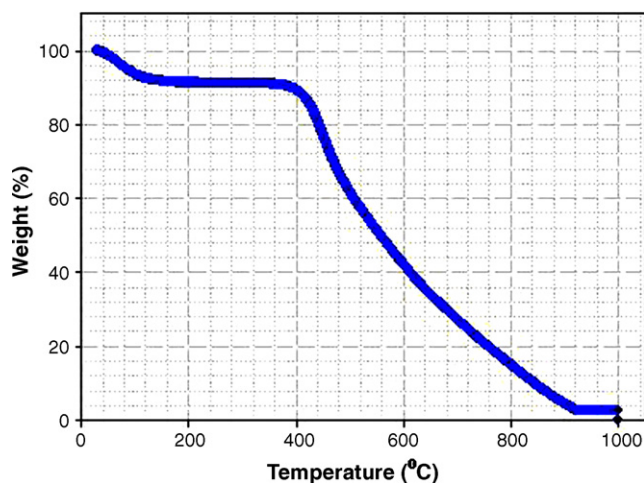


Fig. 2. TGA experiment at 1000 °C, initial weight of adsorbent = 7.08 mg.

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 [18–20] and it is very much dependent on the pH of the solution [15]. By increasing OH<sup>-</sup> functional group, competing between mercury ions and this group starts and OH<sup>-</sup> 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 WS activated carbon, some experiments were conducted using two different initial concentrations of 9.7 and 44.6 mg/L. As it is shown in Fig. 3, the maximum adsorption is observed at pH 2. 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. Generally, adsorption efficiency of Hg(II) has decreased with increasing pH and reached a plateau at the pH range of 8–11. It can be concluded that at any pH, Hg(II) removal by WS activated carbon is very much greater for the concentrated solution than the dilute one. This is the result of stronger adsorption driving force in the concentrated solution. In general, the results indicated that the adsorption is highly pH dependent. Similar results have been also reported in other studies [20,29–31].

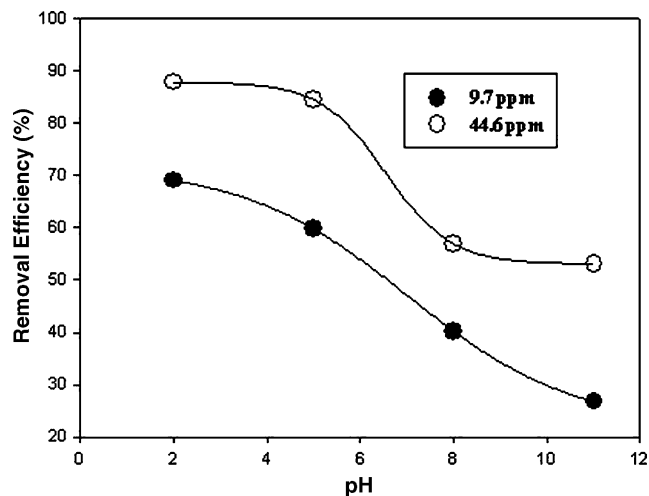


Fig. 3. Effect of pH on the adsorption of Hg(II) (time = 15 min, temperature = 29 °C).

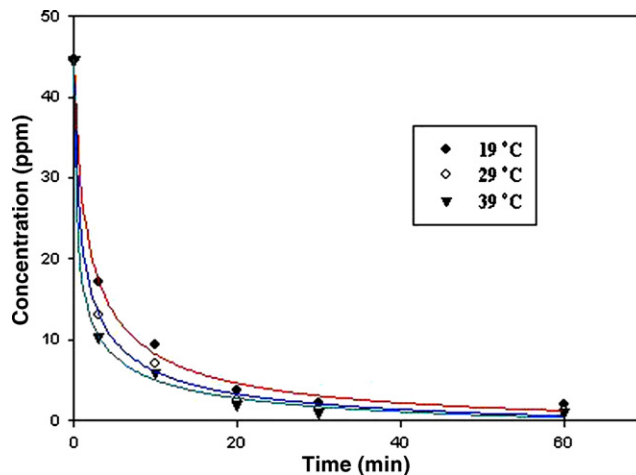


Fig. 4. Effect of temperature of Hg solution.

### 3.2. Effect of temperature

Fig. 4 shows the variation of Hg(II) concentration versus time at different sorption temperatures of 19, 29 and 39 °C. Parameters with constant values in the 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 temperature increase, indicating an endothermic nature of the sorption processes, while the time required for reaching equilibrium remains practically unaffected. Also, the concentration of Hg (II) in solutions decreases with the time and attained equilibrium within 30–60 min.

It is well recognized that the characteristic of the sorbent surface is a critical factor which affects sorption rate parameters and also diffusion resistance plays an important role in the overall transport of the ions. Improving the adsorption capacity with temperature suggests that active centers on the surface available for adsorption increase with the temperature. This could also be attributed to the pore size variation and enhancing rate of intraparticle diffusion of solute since diffusion is an endothermic process.

### 3.3. Effect of initial concentration and contact time

Usually in mercury adsorption studies, it is observed that the Hg(II) removal increased almost linearly with the enhancement of Hg(II) concentration. Many researchers have experienced similar results. For example, Zhang et al. [32] studied mercury adsorption with sewage sludge carbon and showed that the Hg removal increased linearly with the enhancement of Hg(II) concentration. Inbaraj and Sulochana [33] 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. [34]. 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. 5. It is clear that the sorption amount of Hg(II) increased with increasing the initial ion concentration. Also, the amount of metal ion adsorbed is increased sharply with time in the initial stage (0–20 min range) and then has a gradual enhancement to reach equilibrium in approximately 60 min time. It is seen that further increase in the contact time has a negligible effect on the sorption amount of ion.

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

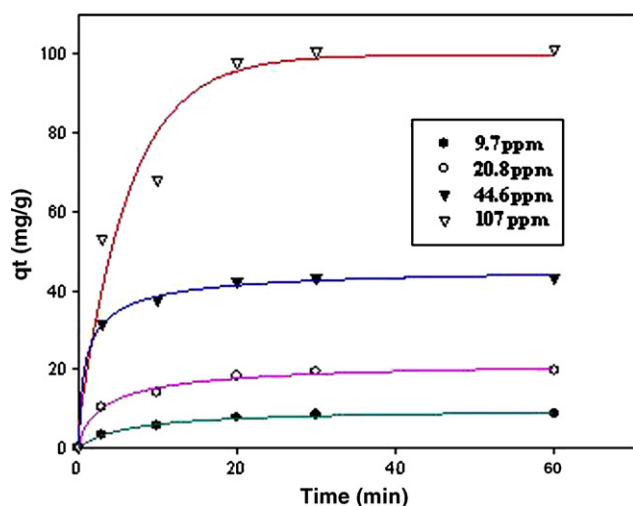


Fig. 5. Effect of initial concentration of Hg solution.

the equilibrium was reached. Increase in the uptake capacity of WS material with increasing initial ion concentration may be due to the higher collision probability between ions and WS particles. Variation in the extent of adsorption may also be due to the fact that initially all active sites on the surface of WS were vacant and metal ion concentration 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 surface of WS. Rapid uptake of metal ions by WS 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:

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

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 and 60 min time are: 8.7, 19.6, 43.39 and 101 mg/g, respectively.

### 3.4. Adsorption Isotherms

Fig. 4 shows adsorption isotherm of mercury ions on WS activated carbon. A study on the variation of initial mercury concentration at a fixed amount of adsorbent (0.02 g) per 50 mL solution was carried out at room temperature (29°C). The adsorption isotherms for Hg(II) sorption on WS carbon were obtained for concentrations ranging from 9.7 to 107 mg/L, while keeping all other parameters constant. These parameters were: shaking time = 60 min, stirring speed = 720 rpm and pH 5. Contact time was also kept constant at 60 min for all equilibrium experiments. The adsorption isotherm of mercury ions is shown in Fig. 6. It is apparent that there is a very mild rising in the adsorption by increasing mercury concentration, particularly in the lower concentration range, indicating high affinity performance. Fig. 6 clearly indicates that the isotherms exhibit Langmuirian behavior.

The isotherm data are correlated with Freundlich and Langmuir model for further investigation. The amount of mercury adsorption is calculated from equation (1) replacing  $q$  by  $q_e$ , where  $q_e$  (mg/g) is the amount of ion adsorbed by the adsorbent phase at equilibrium.

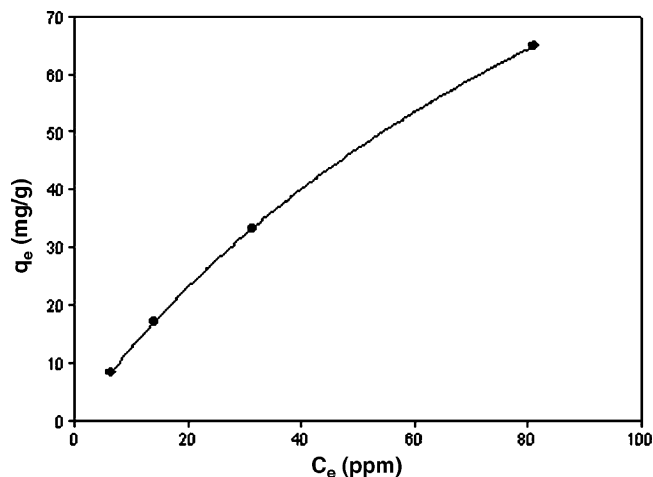


Fig. 6. Adsorption isotherm of mercury on WS.

#### 3.4.1. Freundlich model

Langmuir and Freundlich equations are commonly used for describing adsorption equilibrium for water and wastewater treatment applications. Freundlich model often gives a better fit particularly for adsorption from liquids and can be expressed as [35]:

$$q_e = K_f C_e^{1/n} \quad (2)$$

where  $K_f$  and  $n$  are Freundlich constants related to adsorption capacity and adsorption intensity. The plot for the adsorption of Hg(II) on WS is shown in Fig. 7. It is seen that the model gives a good fit to the experimental data with the correlation coefficient of  $R^2 = 0.99651$ . 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.

#### 3.4.2. Langmuir model

Langmuir adsorption isotherm is given by the following equation [36]:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e \quad (3)$$

In the model,  $q_m$  (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. When  $C_e/q_e$  is plotted against  $C_e$ , a straight line with the slope of  $1/bq_m$

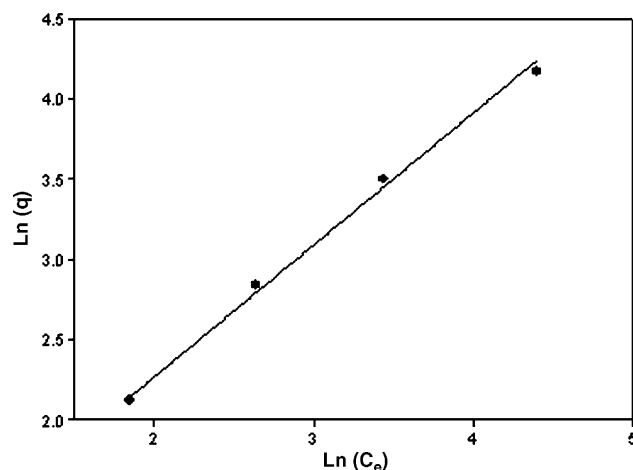


Fig. 7. Freundlich plot of Hg(II) at 29°C.

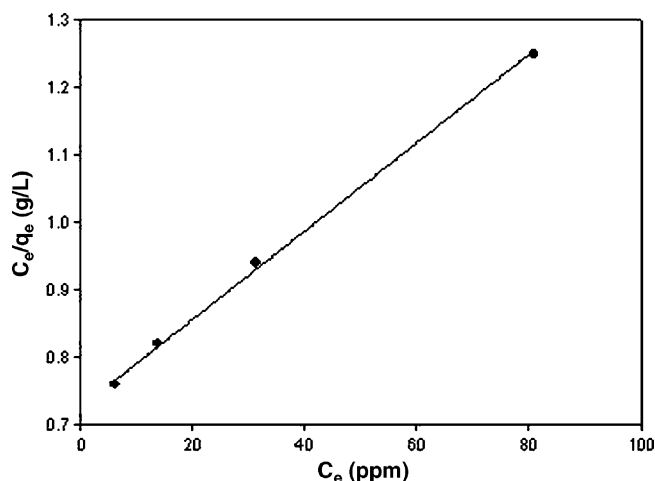


Fig. 8. Langmuir plot of Hg(II) at 29 °C.

Table 1  
Fitted isotherm models for the adsorption of Hg(II) on WS.

Model	Linearized equation	Parameters	$R^2$
Freundlich	$\ln(q_e) = \ln(K_f) + \frac{1}{n} \ln(C_e)$	$K_f = 1.8623$ $n = 1.215$	0.99651
Langmuir	$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e$	$q_m = 151.5$ $b = 0.0091025$	0.99812

is obtained (Fig. 8). The plot indicates that adsorption of mercury follows the Langmuir isotherm.

A host of research workers have applied this model to interpret their sorption data [37,38]. In the present work, we have observed that the Langmuir plot gives a fairly good fit to the experimental data with  $R^2 = 0.99812$ .

The values of Freundlich and Langmuir constants obtained from the plots are given in Table 1 for comparison. It is seen that the Langmuir model fitted the results slightly better than the Freundlich model. It should be mentioned that  $R_L$  value indicates the type of isotherm.  $R_L$  values between 0 and 1 suggest favorable adsorption [33].

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

where  $b$  is the Langmuir constant and  $C_0$  is the initial metal-ion concentration (mg/L).  $R_L$  values of mercury presented in Table 2 are between 0 and 1 for all concentrations at 29 °C, indicating favorable adsorption.

### 3.5. Kinetic studies

The rate constant for surface adsorption of Hg(II) ion on WS is also studied using the pseudo-first-order rate expression of Lagergren model and the pseudo-second-order kinetic rate expression of Ho [39]. The integrated form of the Lagergren equation is given by:

$$\text{Log}(q_e - q_t) = \text{Log}(q_e) - \frac{K_1}{2.303} t \quad (5)$$

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  $K_1$  ( $\text{min}^{-1}$ )

Table 2  
 $R_L$  values for adsorption of mercury onto WS activated carbon at 29 °C.

Hg(II) initial concentration, $\text{mg L}^{-1}$	9.7	20.8	44.6	107
$R_L$	0.918	0.841	0.711	0.5066

Table 3  
Parameters of the kinetic models for adsorption of Hg(II) on WS.

Kinetic model	Fitted parameters $K_1$ ( $\text{min}^{-1}$ ), $K_2$ ( $\text{gm g}^{-1} \text{min}^{-1}$ )	$R^2$	Temp. ( $^{\circ}\text{C}$ )
Pseudo-first-order	$K_1 = 0.1645$	0.95697	19
Pseudo-first-order	$K_1 = 0.1500$	0.98884	29
Pseudo-first-order	$K_1 = 0.1578$	0.90546	39
Pseudo-second-order	$K_2 = 0.0119$	0.99785	19
Pseudo-second-order	$K_2 = 0.0181$	0.99845	29
Pseudo-second-order	$K_2 = 0.0256$	0.99837	39

is the rate constant of first-order adsorption.  $q_e$  is extrapolated from the experimental data at time  $t = \infty$ . A straight line of  $\text{log}(q_e - q_t)$  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:

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

where  $K_1$  and  $K_2$  are the first-order and second-order rate constants ( $\text{min}^{-1}$ ).

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 3. Also, the fitted curves of the models along with the experimental data are illustrated in Figs. 9 and 10 for comparison. 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 WS carbon is calculated as 151.51 mg/g at pH=5.0 and room temperature of 29 °C. For comparison, the monolayer adsorption capacities of mercury using several other adsorbents are presented in Table 4. It is seen from the table that our adsorbent (WS) has a high capacity for Hg(II) removal from the aqueous solutions compared to the other adsorbents.

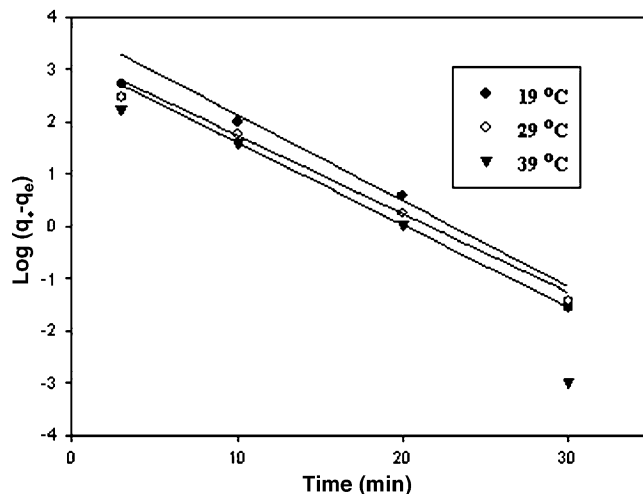


Fig. 9. Lagergren plots for the adsorption of Hg(II) at 19, 29, and 39 °C, first-order model.

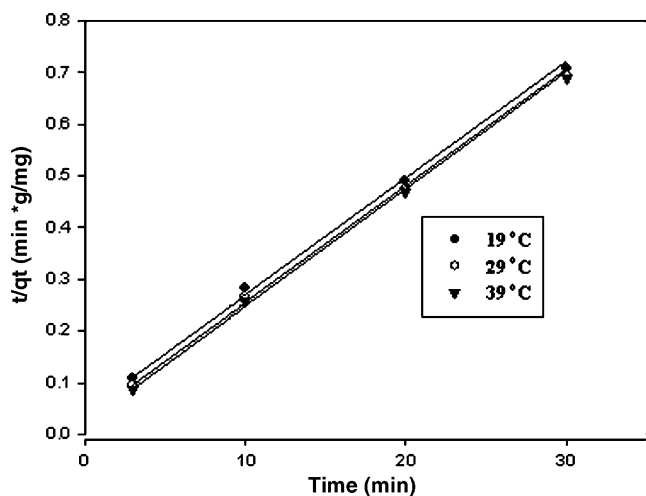


Fig. 10. Lagergren plots for the adsorption of Hg(II) at 19, 29, and 39 °C, second-order model.

Table 4

Monolayer ad solution capacity of mercury in various adsorbents.

Adsorbent type	$q_m$ (mg/g)	Ref.
Acrylic textile fibre	290–710	[40]
Kynol fiber		
Fur fural	174	[34]
Activated carbon (coirpith)	154	[23]
Walnut shell (WS) activated carbon	<b>151.51</b>	<sup>a</sup>
Activated carbon (antibiotic waste)	129	[41]
PHC-peanut hull carbon	110	[21]
Coal adsorbents (Some)	105	[42]
Activated carbon (Indian almond)	94.43	[33]
Coal adsorbents (Mengen)	92	[42]
Activated carbon	69.44	[43]
Coal adsorbents (Seyitorner)	56	[42]
Sago waste carbon	55.6	[25]
Coal adsorbents (Bolluca)	37	[42]
Carbon aerogel	34.96	[44]
Commercial activated carbon	12.38	[21]
Sulfo-calcic ashes	4.9	[45]
Waste rubber	4	[18]
Silico-aluminous ashes	3.2	[45]
Fuller's earth	1.145	[43]
Granular activated carbon	0.8	[17]
Activated carbon (fertilizer waste)	$3.62 \times 10^{-3}$	[31]

Bold values belongs to the adsorbent prepared in the current study. It shows the significance of our sample compared to the previous adsorbent samples by other researchers.

<sup>a</sup> Adsorbent used in the present study.

#### 4. Conclusion

The present study revealed the feasibility of using a new cheap adsorbent (WS) derived from an agriculture waste 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 WS is obtained as 151.51 mg/g at pH 5.0 for adsorbent with 0.088 mm particle sizes.

#### Acknowledgment

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