

Mercury adsorption on a carbon sorbent derived from fruit shell of *Terminalia catappa*

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Abstract

A carbonaceous sorbent derived from the fruit shell of Indian almond (*Terminalia catappa*) by sulfuric acid treatment was used for the removal of mercury(II) from aqueous solution. Sorption of mercury depends on the pH of the aqueous solution with maximum uptake occurring in the pH range of 5–6. The kinetics of sorption conformed well to modified second order model among the other kinetic models (pseudo first order and pseudo second order) tested. The Langmuir and Redlich–Peterson isotherm models defined the equilibrium data precisely compared to Freundlich model and the monolayer sorption capacity obtained was 94.43 mg/g. Sorption capacity increased with increase in temperature and the thermodynamic parameters, ΔH° , ΔS° and ΔG° , indicated the Hg(II) sorption to be endothermic and spontaneous with increased randomness at the solid–solution interface. An optimum carbon dose of 4 g/l was required for the maximum uptake of Hg(II) from 30 mg/l and the mathematical relationship developed showed a correlation of 0.94 between experimental and calculated percentage removals for any carbon dose studied. About 60% of Hg(II) adsorbed was recovered from the spent carbon at pH 1.0, while 94% of it was desorbed using 1.0% KI solution.

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1. Introduction

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 dyspnoea [1,2]. According to the Indian Standard Institution (ISI), the tolerance limit for Hg(II) for discharge into inland surface waters is 10 µg/l [3] and for drinking water is 1 µg/l [4]. 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.

Among the many methods available for the removal of trace metals from water, namely chemical precipitation, ion exchange, coagulation, solvent extraction and membrane pro-

cesses, adsorption has been shown to be an economically feasible alternative [5–7]. Activated carbon is one of the most popular adsorbents for the removal of mercury from aqueous solution [5–7] although other materials have been used including onion skin [8], waste rubber [9], coconut husk [10], fertilizer waste slurry [11] and photofilm waste sludge [12]. In view to combine efficiency and cost effectiveness several non-conventional cheap materials like peanut hull [13], jackfruit peel [14], coir pith [15] flax shive [16] and sago waste [17] have been tried as carbonaceous precursors and applied for the removal of mercury. Normally, the precursor material is heated to high temperatures (700–1200 °C) to remove the volatile matter and the resulting carbon is then activated either physically or chemically [18]. Carbonisation can also be performed by dehydration with sulfuric acid or phosphoric acid at low temperature resulting in a colloidal and porous active charcoal [19]. Such carbons have been reported [13–17,20,21] to have the capability of decolorising dyes and ion exchange metal species.

Terminalia catappa Linn. is a large, deciduous tree with smooth grey bark, porous and fibrous pericarp, hard endocarp enclosing the edible seed and whorled branches. In India, this tree is commonly planted in house gardens for ornament and also

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for fruit. As the timber is moderately easy to saw and work and it polishes well, it has wide applications and classed as a good constructional timber. The fruit of this tree, commonly called as ‘Indian almond’, comprises of kernel (10.32%), fibrous covering (8.97%), husk (34.08%) and hard endocarp (46.63%) [22]. Apart from the natural biological cycle, significant amount of fruit shell is discarded as a waste agricultural biomass, as this tree has many timber applications. In the present study, portion of the fruit excluding the kernel, which amounts to about 89.68% of the total fruit [22], is chosen as the precursor for preparation of a carbon adsorbent and applied for the removal of Hg(II) from aqueous solution.

2. Materials and methods

Fruit shell of *T. catappa*, collected from Tiruchirappalli, India, was dried and chopped into small pieces. The chopped pieces were treated with concentrated sulfuric acid (Sp.gr. 1.84) in a weight ratio of 1:0.92 (shell:acid) and the resulting black product was kept in an air oven maintained at $160 \pm 5^\circ\text{C}$ for 10 h followed by washing with distilled water until free of excess of acid and dried at $105 \pm 5^\circ\text{C}$. The carbon adsorbent obtained was designated as TCC. The carbon was ground and the portion retained between 180 and 210 μm sieves were used. All the chemical and reagents used were of analytical reagent grade obtained from E-Merck or Sigma–Aldrich.

The carbon was characterized for physico-chemical properties like pH (6.14), moisture content (9.90%), bulk density (0.75 g/ml), ash content (1.72%), decolourizing power (10.50 mg/g), ion exchange capacity [23] (0.93 meq./g), water soluble matter (1.55%) and acid soluble matter (4.00%), as per the standard procedures [24]. The surface area was measured by nitrogen adsorption and is calculated to be $4.60\text{ m}^2/\text{g}$ from the B.E.T. equation [25]. Similar low values were observed for carbons prepared from coconut shell ($13.00\text{ m}^2/\text{g}$) [26] and flax shive ($19.00\text{ m}^2/\text{g}$) [27] by sulfuric acid treatment. These low values may be due to the presence of oxygen as complexes in the form of C_xO_y [5] that cannot be displaced by nitrogen used in the B.E.T.- N_2 method or may be due to the occurrence of micropores in the structure being either very low or non-existent [27].

Even though the carbonized materials are rather complex, Fourier transform infrared spectrophotometry still can give some information about the probable functional groups on the surface. The carbon was dried in an oven overnight to remove any moisture retained and encapsulated into dry KBr discs. The discs were scanned in transmission mode using a Bruker IRS66V FTIR spectrophotometer from 400 to 4000 cm^{-1} with background subtraction.

Adsorption experiments were conducted in batch mode using polythene bottles of 200 ml capacity provided with screw caps. About 100 ml of mercury(II) solution of predetermined concentration (prepared using HgCl_2) was taken in these bottles and equilibrated with a known amount of carbon for predetermined periods of time in an orbital shaker (250 rpm) equipped with incubation hood for temperature control. After the lapse of this period, the solutions were centrifuged and the supernatant solution was analysed for residual mercury(II) spectrophotomet-

rically [28] using rhodamine-6G as reagent in a Perkin-Elmer EZ 301 UV–vis spectrophotometer at 575 nm. For pH study, 30 mg/l solutions were adjusted to different pH (1.0–10.0) and shaken with 0.2 g/l of carbon dose for 12 h. The kinetic data was obtained by agitating 30 mg/l of Hg(II) solutions with 0.2 g/l of carbon dose for predetermined periods of time. The equilibrium study was carried out by equilibrating a range of Hg(II) concentration solution (5–60 mg/l) with 0.2 g/l of carbon dose for 12 h. For temperature study, 40 mg/l of Hg(II) solutions were shaken with 0.2 g/l of carbon dose at temperatures, 305, 313, 323 and 333 K for 12 h. The effect of carbon dose on adsorption capacity was studied by equilibrating 30 mg/l of Hg(II) solutions with a range of carbon dose (0.05–5.0 g/l) for 12 h. Each batch experiment was carried out in triplicate and mean values were taken for calculation. Analytical error and standard deviation was determined in each case and the maximum error was found to be $\pm 5\%$.

The amount of metal sorbed at time t , q_t and at equilibrium, q_e was calculated from the mass balance equation:

$$q_t = (C_0 - C_t) \frac{V}{m} \quad (1)$$

where C_0 and C_t are the initial and final metal concentrations (mg/l), respectively, V is the volume of metal solution (l) and m is the mass of the sorbent (g). When t is equal to the equilibrium contact time, $C_t = C_e$, $q_t = q_e$, then the amount of metal sorbed at equilibrium, q_e , is calculated using Eq. (1). Calculations and graphical representations were performed in the Microsoft ExcelTM software package.

3. Results and discussion

3.1. Infrared spectrum of TCC

FTIR spectrum for TCC is shown in Fig. 1. The intense and broad absorption band around 3436 cm^{-1} is assignable to O–H stretching of hydroxyl group. As the frequency of free OH groups is sharp and is located above 3500 cm^{-1} [29], these vibrations are associated with hydrogen bonds and thus the surface OH groups probably interact with water molecules adsorbed by the carbon sample, as suggested by Zawadzki [30]. The band at 2925 cm^{-1} denotes the presence of stretching C–H vibrations in CH_2 [31] or C=C–H group. The intense band at 1713 cm^{-1} suggests the presence of stretching C=O vibrations arising from groups such as lactone, quinone [29] and carboxylic acids [31]. The intense band at 1619 cm^{-1} may be due to the asymmetric and symmetric stretching COO^- vibrations or to skeletal C=C aromatic vibrations [29,32]. The band appearing around 1153 cm^{-1} is due to C=S stretching vibration. The bands appearing at 1065, 877, 660 and 599 cm^{-1} are compatible with absorptions due to S=O and S–O stretching in species such as HSO_4^- and SO_4^{2-} [33,34].

3.2. Solution pH influence

The pH of the solution plays a vital role in any adsorption process. This study gives information on optimum pH

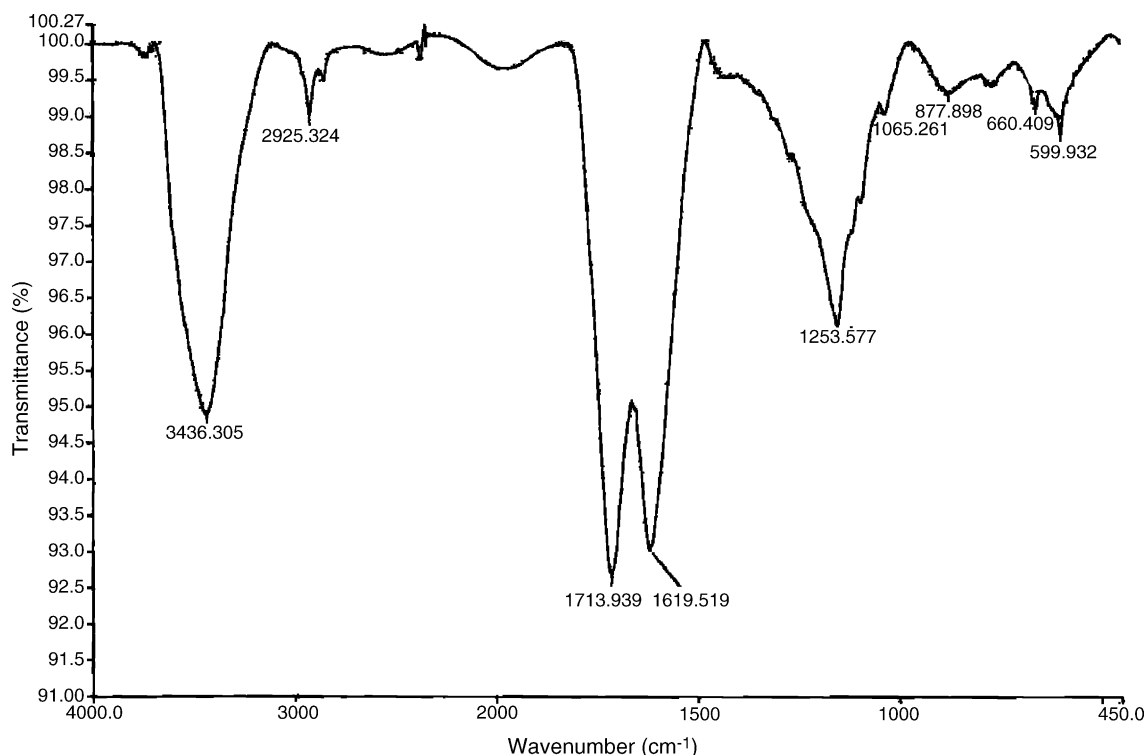


Fig. 1. IR spectrum of the virgin carbon derived from fruit shell of *Terminalia catappa*.

for maximum Hg(II) adsorption and predominant mechanism involved in Hg(II) adsorption on TCC. Fig. 2 illustrates the influence of pH on Hg(II) adsorption by TCC. The removal of Hg(II) increased with increase in pH and reached a

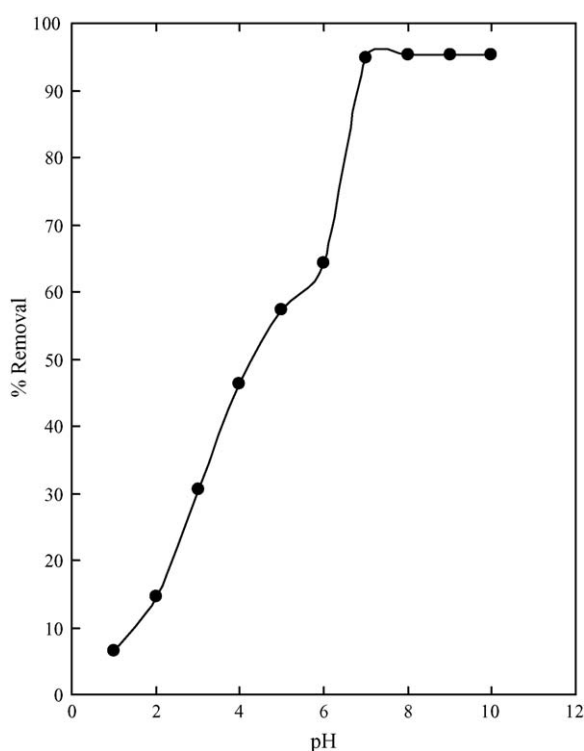


Fig. 2. Solution pH influence on Hg(II) adsorption by TCC.

shoulder-like maximum at pH 6.0 followed by a sharp increase in removal reaching close to 100% over the pH range of 7.0–10.0.

The increase in Hg(II) removal from 6.6 to 64.3% on increasing the pH from 1.0 to 6.0 can be explained as follows: According to stability constant calculations [9], in the presence of Cl^- , the predominant species at $\text{pH} < 4.0$ is HgCl_2 . The formation of HgCl_2 has been found to decrease the Hg(II) adsorption onto a commercial FS-400 GAC [7]. Accordingly, Hg(II) adsorption on TCC decreased when the pH was lowered using HCl from 6.0 to 1.0. Another plausible explanation can be: at low pH values, excess H^+ ions present in solution competes with Hg(II) ions for active sites leading to less Hg(II) removal. However, when the pH was increased the concentration of H^+ ions decreases, but the concentration of Hg(II) ions remains the same leading to increased uptake. Similar trend was observed for the adsorption of Hg(II) onto coir pith carbon [15].

The sharp increase in Hg(II) removal beyond pH 6.0 is due to the precipitation of Hg(II) which leads to a quantitative removal close to 100% over the pH range of 7.0–10.0. This was confirmed by performing a control experiment with Hg(II) solution adjusted to different pH and shaken in the absence of added adsorbent. The Hg(II) started to precipitate between pH 6 and 6.5, as evidenced by the quantitative decrease in Hg(II) concentration in solution above pH 6.0. Cox et al. [16] have chosen an optimum pH of 6.5 for Hg(II) sorption by flax shive carbon as precipitation of Hg(II) was observed above pH 7.0. Therefore, to ensure the removal of Hg(II) by TCC is only due to adsorption, in all further studies the solution pH was maintained at 5.0.

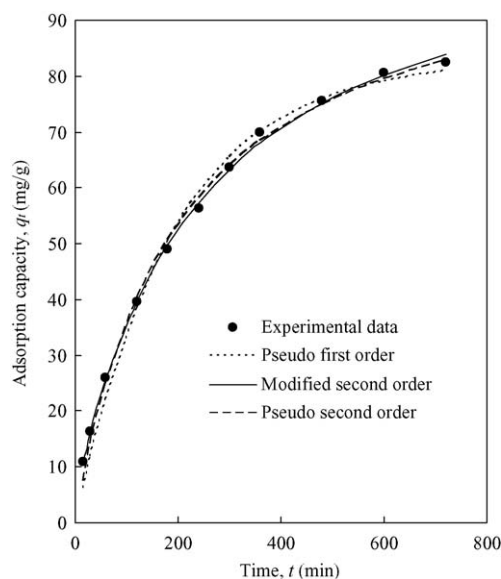


Fig. 3. Fitted curves of the kinetic models with the experimental data.

3.3. Agitation time influence and adsorption kinetics

Fig. 3 shows the kinetic data for Hg(II) adsorption by TCC. The adsorption capacity increased with increase in time and reached a value of 82.93 mg/g at an equilibrium time of 720 min. The curve was single, smooth and continuous indicating monolayer adsorption of Hg(II) on carbon surface. The kinetic data obtained was modeled with pseudo first order [35] (Eq. (2)), pseudo second order [36] (Eq. (3)) and modified second order [20,21] (Eq. (4)) kinetic models to find out the kinetic model that best describes the experimental data.

$$q_t = q_e[1 - \exp(-k_1 t)] \quad (2)$$

$$q_t = \frac{t}{(1/k_2 q_e^2) + (t/q_e)} \quad (3)$$

$$q_t = q_e \left\{ 1 - \left[\frac{1}{\beta_2 + k_2 t} \right] \right\} \quad (4)$$

where q_e and q_t are the amounts of Hg(II) adsorbed (mg/g) at equilibrium and at time t (min), respectively, k_1 and k_2 are the first order and second order rate constants (min^{-1}), respectively, and β_2 is a constant that represents initial particle loading. Modelling was done by non-linear optimisation method using GNUPLLOT program (Version 3.7 for MS Windows) and fitted parameters of the kinetic models obtained are presented in Table 1. The fitted curves of the models along with the experimental data for comparison are illustrated in Fig. 3. To have a

measure of degree of fitness the correlation coefficient, r^2 and normalized standard deviation, Δq (%), (using Eq. (5)) were computed and are presented in Table 1.

$$\Delta q (\%) = 100 \times \sqrt{\sum \frac{[(q_{\text{ex}} - q_{\text{pr}})/q_{\text{ex}}]^2}{(n - 1)}} \quad (5)$$

where q_{ex} and q_{pr} are experimental and predicted equilibrium adsorption capacities, respectively, and n is the number of data points. Though r^2 values obtained were close to 1 for all the kinetic models, higher Δq values obtained for modified second order model indicate that this model could more closely describe the experimental kinetic data than pseudo first order and pseudo second order models.

3.4. Initial Hg(II) concentration influence and adsorption isotherms

The adsorption capacity increased from 21.67 to 85.32 mg/g while increasing the initial Hg(II) concentration from 5 to 60 mg/l. This observed trend is due to the increase in driving force offered by the concentration pressure gradient. The equilibrium data obtained were modeled with two-parameter Freundlich [37] (Eq. (6)) and Langmuir [38] (Eq. (7)) isotherm equations and a three-parameter Redlich–Peterson [39] (Eq. (8)) isotherm equation, which combines the features of Freundlich and Langmuir isotherm models.

$$q_e = K_F C_e^{1/n} \quad (6)$$

$$q_e = \frac{Q_0 b C_e}{1 + b C_e} \quad (7)$$

$$q_e = \frac{\alpha C_e}{1 + \beta C_e^\gamma} \quad (8)$$

where, K_F and n are Freundlich constants indicating the adsorption capacity (mg/g) and intensity of adsorption, respectively, Q_0 and b are Langmuir constants denoting the adsorption capacity (mg/g) and energy of adsorption (l/mg), respectively and α (l/g), β (l/mg) and γ are Redlich–Peterson constants. The fitted isotherm curves along with the experimental data for comparison are depicted in Fig. 4 and the fitted parameters obtained specific for the present system along with r^2 and Δq values are reported in Table 2. The Langmuir and Redlich–Peterson models better described the equilibrium data, as evidenced from the higher r^2 and lower Δq values obtained for these models compared to Freundlich model. As the equilibrium data conforms well to the basic two-parameter Langmuir isotherm model, its assumptions, as detailed elsewhere [38], holds good for the present

Table 1
Fitted kinetic models for adsorption of Hg(II) on TCC with r^2 and Δq values

Kinetic model	Fitted parameters	r^2	Δq (%)
Pseudo first order	$q_t = 82.82[1 - \exp(-0.0052t)]$	0.9947	16.62
Pseudo second order	$q_t = \frac{t}{(1/(0.0052)(105.17)^2) + (t/105.17)}$	0.9969	10.67
Modified second order	$q_t = 110.42 \left\{ 1 - \left[\frac{1}{1.04 + 0.0043t} \right] \right\}$	0.9986	1.46

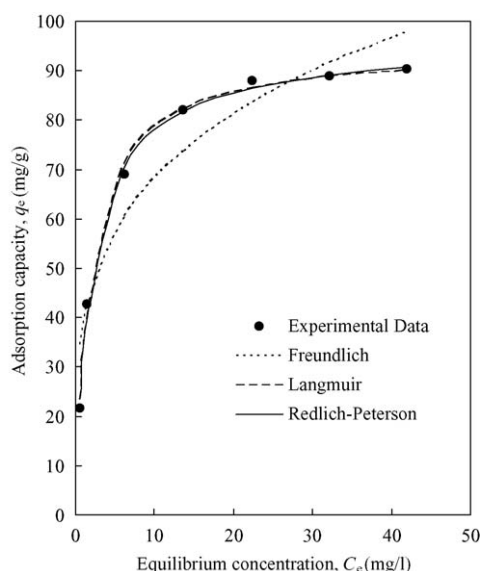


Fig. 4. Fitted curves of the isotherm models with the experimental data.

system. The monolayer adsorption capacity of Hg(II) on TCC is 94.43 mg/g at pH 5.0 and at room temperature ($32 \pm 0.5^\circ\text{C}$). A lower Hg(II) adsorption have been reported for several adsorbents such as sago waste carbon (55.60 mg/g) [17], commercial activated carbon (12.38 mg/g) [13], waste rubber (4.00 mg/g) [9] and granular activated carbon (0.8 mg/g) [7]. Using the best-fit Langmuir isotherm equation with fitted parameters (Table 2), the volume of wastewater that could be treated was determined. If a wastewater contains Hg(II) concentration of 5 mg/l, 1 g of TCC can treat about 13 l of wastewater.

The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter [40], R_L , defined as $1/(1 + bC_0)$, 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 3) indicate favorable isotherm shape ($0 < R_L < 1$) for adsorption of Hg(II) on TCC in the concentration range studied.

3.5. Temperature influence and thermodynamic parameters

The effect of temperature on Hg(II) adsorption by TCC revealed the adsorption to be endothermic, as the adsorption capacity increased with increase in the temperature of the system (Table 4). The enhancement of adsorption capacity on increasing the temperature may be attributed to the increase in mobility of Hg(II) ions and/or the ‘activated diffusion’, which

Table 2
Fitted isotherm models for adsorption of Hg(II) on TCC with r^2 and Δq values

Isotherm model	Fitted parameters	r^2	Δq (%)
Freundlich	$q_e = 38.25C_e^{1/0.25}$	0.9128	7.89
Langmuir	$q_e = \frac{(94.43)(0.49)C_e}{1+0.49C_e}$	0.9956	1.94
Redlich–Peterson	$q_e = \frac{49.04C_e}{1+0.55C_e^{0.98}}$	0.9960	1.83

Table 3

The separation factor, R_L , values

Initial Hg(II) concentration, C_0 (mg/l)	R_L
5	0.2882
10	0.1684
20	0.0919
30	0.0632
40	0.0482
50	0.0389
60	0.0326

can cause small pores to widen and provide more surfaces for adsorption [41].

From this study results thermodynamic parameters like change in enthalpy, ΔH° , change in entropy, ΔS° and change in free energy, ΔG° , were determined using the following relations:

$$K_c = \frac{C_{Ae}}{C_e} \quad (9)$$

$$\Delta G = -RT \ln K_c \quad (10)$$

$$\log K_c = \frac{\Delta S^\circ}{2.303R} - \frac{\Delta H^\circ}{2.303RT} \quad (11)$$

where K_c is the equilibrium constant, C_{Ae} is the solid phase concentration at equilibrium (mg/l), T is the temperature in Kelvin and R is the gas constant. The ΔH° and ΔS° were obtained from the slope and intercept of the van’t Hoff plot of $\log K_c$ versus $1/T$ (Fig. 5) and are presented in Table 4. The positive value of ΔH° confirms the endothermic adsorption of Hg(II) onto TCC. The positive value of ΔS° shows increased randomness at the solid–solution interface during Hg(II) adsorption and reflects the affinity [42] of TCC for Hg(II) ions. The adsorbed water molecules, which are displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate molecules, thus allowing prevalence of randomness in the system. The negative values of ΔG° and decrease in ΔG° with increase in temperature (Table 4) indicate the spontaneous nature of Hg(II) adsorption on TCC at higher temperatures.

3.6. Adsorbent dose influence

Mercury(II) adsorption increased from 34.2 to 99.5% with increase in TCC dose from 0.05 to 5.0 g/l. This increasing trend is ascribed to the introduction of more binding sites for adsorption on increasing the carbon dose. A minimum amount of 4.0 g/l of TCC was required for maximum Hg(II) removal (98.6%) from 30 mg/l solution.

Table 4
Temperature influence on adsorption capacity and thermodynamic parameters

Temperature (K)	q_e (mg/g)	K_c	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol/K)
305	82.75	16.55	0.88	83.03	268.78
313	119.55	23.91	−1.03		
323	155.85	31.17	−3.39		
333	184.00	36.80	−6.76		

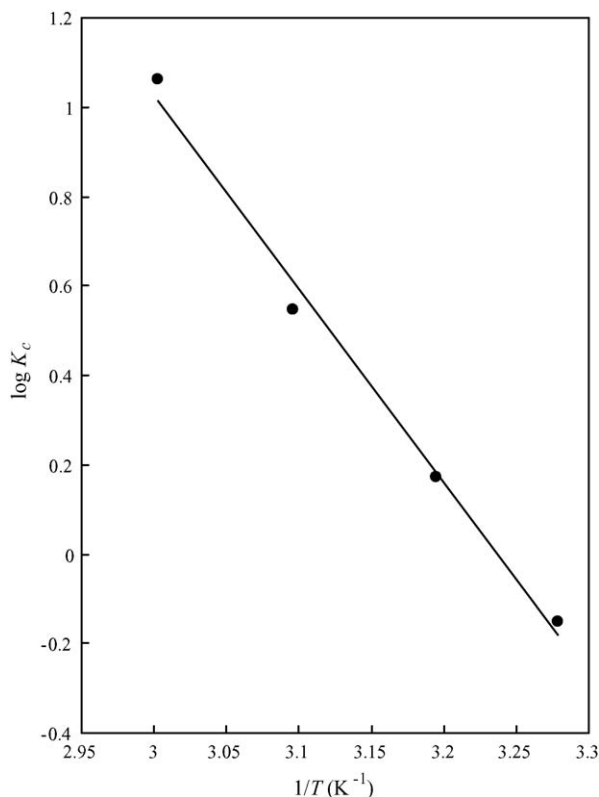


Fig. 5. van't Hoff plot for determination of ΔH° and ΔS° .

The results of this experiment were used to develop a mathematical relationship between percentage removal (R) and adsorbent dose (m , g/l) by non-linear optimisation method. The relationship developed is as follows:

$$R = \frac{m}{1.45 \times 10^{-3} + 1.05 \times 10^{-2}m} \quad (12)$$

This equation can be used to predict the percentage Hg(II) removal for any TCC dose within the experimental conditions studied. The correlation coefficient, r^2 , obtained between the experimental and calculated percentage removal values was 0.94. However, a closer discerning shows a correlation of 0.99 between experimental and predicted percentage removal values for the carbon dose range of 0.05–1.5 g/l and 1.5–5.0 g/l separately (Fig. 6).

3.7. Desorption study

Desorption study was conducted to recover the metal, to regenerate the spent carbon for repeated use and to recycle the metal-recovered wastewater. After performing adsorption experiments with 20 mg/l of Hg(II) solution and 0.2 g/l of carbon dose at pH 5.0, the solutions were adjusted to different pH values from 1.0 to 6.0 and shaken for 1 h to find the effect of varying pH on desorption. The results are illustrated in Fig. 7 which shows that a maximum Hg(II) desorption of 60% was achieved at pH 1.0. In order to regenerate the carbon and achieve complete desorption of Hg(II), three differ-

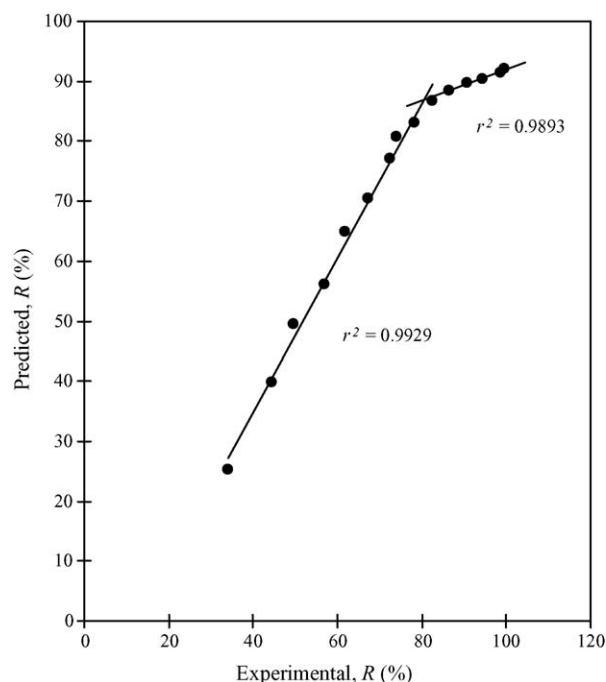


Fig. 6. Correlation between experimental and predicted % removal values for any carbon dose studied.

ent percentage solutions of KI (0.5, 1.0 and 2.0%) were tried as desorbing agents. About 85.2, 94.2 and 94.6% of Hg(II) was recovered by using 0.5, 1.0 and 2.0% of KI solutions, which indicates that maximum Hg(II) recovery was obtained by using a minimum of 1.0% KI solution. Maximum Hg(II) recovery by KI solution is due to the formation of relatively

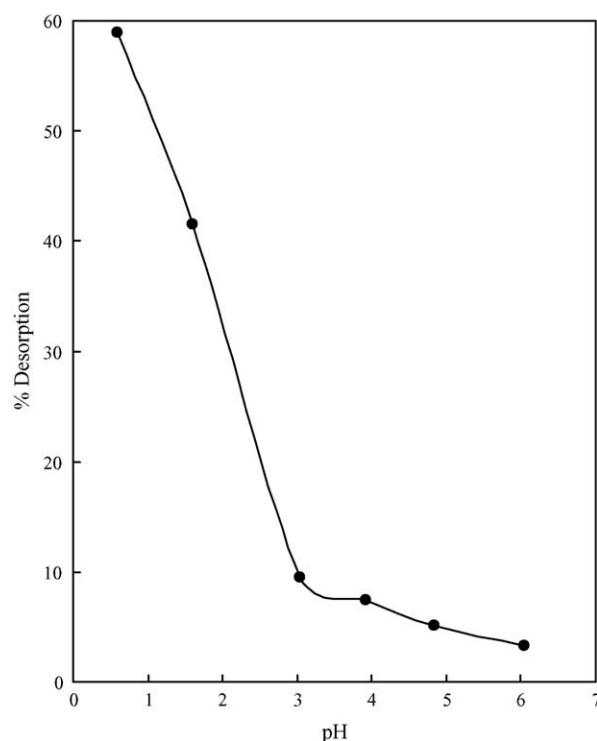


Fig. 7. Effect of pH on desorption of Hg(II) from spent carbon.

stable iodide complexes of Hg(II) compared to the chloride complexes [13–15,17]. The fact that only partial or incomplete Hg(II) desorption was achieved (60% in HCl and 94% in KI) shows that in addition to predominant ion exchange mechanism, other mechanisms like hydrolysis/precipitation, chemisorption or redox reaction might also be responsible for the adsorption of Hg(II) on TCC.

4. Conclusion

The foregoing study has revealed the feasibility of using a carbon sorbent derived from the fruit shell of *T. catappa* for the removal of mercury from aqueous solution. The adsorption was well described by modified second order kinetic and Langmuir isotherm models. The adsorption capacity of the carbon was 94.43 mg/g at pH 5.0 for the particle of 180–210 μm . The carbonaceous precursor chosen being an inexpensive waste material the treatment is expected to be economical.

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