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Adsorption of Hg(II) from aqueous solution onto *Borassus Flabeliffer*: equilibrium and kinetic studies

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ABSTRACT

Palm shell (*Borassus Flabellifer*), a ligno-cellulosic agrowaste is used as adsorbent for the removal of Hg(II). A series of experiments were conducted in a batch system to evaluate the effect of system variables. The optimal pH value of mercury(II) adsorption onto palm shell powder was found to be pH 3.0–7.0. The maximum uptake capacity was found 0.04425 mg/g. The pseudo second order rate equation was found to be the best fit for the kinetic data obtained. The equilibrium data were found to follow both Freundlich and Langmuir isotherm models with high coefficients of determination.

Keywords: Natural adsorbent; Mercury; Palm shell powder; Adsorbent kinetics; Thermodynamic parameters

1. Introduction

Worldwide rapid industrialization and technological advances result in toxic heavy metals in water [1]. According to World Health Organization, the metals of most immediate concern are aluminium, chromium, manganese, iron, cobalt, nickel, copper, zinc, cadmium, mercury and lead. In particular, mercury and its compounds are very dangerous and toxic even at a very low concentration and hence must be removed by means of treatments to meet the legislative standards [2]. Maximum permissible limit of mercury in water is 1 μg/L [3]. The conventional methods used for effluent treatment include chemical precipitation, chemical oxidation and reduction, ion-exchange and activated-carbon adsorption [4]. However, these processes have significant disadvantages including incomplete metal removal, particularly at low concentrations, high operational costs and they are not eco-friendly. As metal ions are known to bind to carboxylate, hydroxyl, sulphate, phosphate, amide and amino functional groups, commonly found in ligno-cellulosic materials, bio-sorption has emerged as a new economic and eco-friendly alternative [5,6]. A literature search reveals that no work has been reported on the removal of mercury from aqueous systems using palm shell as adsorbent. So an attempt was made to study the feasibility of palm shell powder (*Borassus Flablifer*), shell of palm fruit an agricultural waste, cheap, easily available biosorbent for the efficient removal of mercury at low concentrations. A sorption process is found to proceed through varied mechanisms such as external mass transfer of solute, intraparticle diffusion and adsorption at sites. For a deeper understanding like prediction of the rate determining step of sorption, an advanced approach is the development of mechanistic models [7,8]. Therefore the most appropriate way to design and assess the performance of the sorption system is to have the sorption isotherms. The isotherm equations — Langmuir I, II, III, IV, and Freundlich isotherms — were studied to model the equilibrium sorption data. The effect of pH, tempera-

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ture, duration of contact, concentration of metal ion and concentration of sorbent on the adsorption kinetics was studied. The batch sorption kinetics was studied for the first order reversible reaction, pseudo first order; all the 4 types for second order reaction and intraparticle diffusion reaction.

2. Experimental

2.1. Bio-sorbent preparation and characterization

Palm shells obtained from the coastal areas of Andhra Pradesh were washed extensively under running tap water for approximately 30 min to remove the unwanted matter, further washed a number of times with deionised water, and then dried in sunlight for 2–3 days. These dried shells were cut into pieces and dried in an air oven at 70°C, and the dried biomass was ground using a jaw crusher and separated using a standard sieve of 40#, and stored in a desiccator at room temperature. Palm shell powder of 40# was characterised for its physical properties, i.e. bulk density, moisture content, ion exchange capacity, pH, matter soluble in water and acid. BET analysis for the surface area, pore volume and pore size of the palm shell powder were measured by a surface area analyzer (Micrometrics, ASAP 2010). Functional components of the biomass were obtained using a Fourier transform infrared spectroscopy (Perkin Elmer RX1 model). Spectra were run in the range of 4000–400 cm^{-1} for the biomass. It is a useful tool to identify functional groups in a species, as each specific chemical bond often has a unique energy absorption band; this energy also gives structural and chemical information of a species [9]. X-ray diffraction pattern was also carried out using Shimadzu XRD 6000. The PXRD of the palm shell powder was taken by holding it on a quartz plate for exposure to CuKα radiation of a wavelength 1.5406 Å [10]. The sample was analyzed at room temperature over a range of 5–70° 2 θ with sampling intervals of 0.02° 2 θ and a scanning rate of 6°/min. The surface morphology and texture of the biomass were observed using a scanning electron microscope (JSM-5610LV).

2.2. Batch adsorption measurements

Adsorption studies using palm shell powder were carried out by a batch technique to obtain optimum pH, adsorption capacity with respect to variation in the initial concentration of the metal ion and adsorbent and temperature. In each test 25 mL of metal solutions of known concentration along with the sorbent (0.02–0.1 g) were placed in a thermostat provided with shaking at 180 rpm speed. The study of the effect of different parameters was carried by drawing the samples after desired contact time, and the filtrate was analyzed for the remaining metal ion concentration. All the experiments were conducted in triplicate and the average results are reported in the paper.

The mercury uptake by the palm shell powder was calculated as follows:

$$q_e = C_i - C_e / m \tag{1}$$

where C_i is the initial concentration of metal ions, mg/L; C_e — equilibrium concentration of metal ions, mg/L; m — mass of the adsorbent, g/L; q_e — amount of metal ions adsorbed per gram of the adsorbent.

The experiments done without the adsorbent were treated as blanks and they showed no precipitation of metal ions under the conditions selected.

2.3. Column mode adsorption studies

The column used for the removal of mercury under continuous flow conditions was made up of Borosil glass. The outer diameter of the column was 1 cm and the inner diameter of the column was 0.9 cm. 1 g biomass was packed into a column having a column height of 0.5 m. An aqueous solution containing 0.1 mg/L Hg(II) was passed through the column. The effluent samples were collected every 5 min and were analyzed for Hg(II) concentration by using MA 5840 mercury analyzer. The effluent sample was passed continuously until it reached its saturation point. The breakthrough capacity of the palm shell powder for Hg(II) ions was found to be 0.04425 mg/g from the breakthrough curve.

2.4. Adsorption isotherms

The adsorption isotherms for the adsorbent were obtained using 0.1–0.8 ppm of Hg(II) concentration solutions. Experimental data were fitted to Freundlich and Langmuir models.

2.5. Adsorption dynamics

Kinetic experiments were performed using a number of mercury concentrations at different pH (1–8) and temperatures (25–70°C). The initial pH was adjusted by adding 0.1 N NaOH or 0.1 N HNO_3. Sorbent and mercury solutions were mixed and allowed to react at different contact times. Later, the metal solution was separated from the sorbent by filtration, and its concentration was determined by cold vapor atomic absorption spectrophotometer (MA5840 ECIL model). Kinetic data were analysed with six kinetic models, i.e., the pseudo-first-order model, the pseudo-second-order (I, II, III and IV) models, and the intraparticle diffusion model for low initial mercury concentrations.

3. Result and discussion

3.1. Characterisation

Borassus Flabeliffer was characterised by different physical chemical and analytical techniques. Pore volume, surface area and physical properties are given in Table 1. BET surface area was found to be 2.5174 m^2/g. Pore volume studies and scanning electron micrographs showed that it was microporous, and the pore size was found to be 50.031 A. The possible functional groups observed from FTIR spectra were C=O (1870.5, 1739.4 for 5-membered ring, saturated acyclic ester or lactones, saturated acyclic aldehydes), NO_2 (1510, 1460 for aromatic C-nitro compound), C–H deformation (1460.5, 1379.6), C=C (1622.6 for aromatic stretch), C–O stretching and O–H deformation (1379.1 and 1252.3 for 3° alcohols, phenols), S=O stretch (1379.06, 1044.9 for sulphonates and sulphonamides), C=S stretch (1250 thioketones and dithioesters), N–H stretch (3422.37, 1622.64, 1510.19 for 1° and 2° amines) showing the presence of a number of functional groups which help in binding the mercury metal ions. The diffraction pattern suggested the amorphous nature of biomass.

3.2. Effect of pH and contact time

Fig. 1 represents the effect of the initial pH of the solution on the adsorption of Hg(II) onto the palm shell powder using 0.1 mg/L initial mercury concentration and 4 g/L adsorbent. The optimum pH was found to be 3.0–7.0, so a working pH value was chosen as 5.0 for further adsorption studies. At pH values higher than 7.0 precipitation of mercury started taking place, which makes adsorption studies difficult. Efficient removal of mercury at lower pH values is attributed to a higher degree of ionization of metal ions due to the reduced competition of H$^+$ ions with the mercury ions for adsorption sites. The physicochemical characteristics of the adsorbent may also play an important role. It was reported that free metal ions are adsorbed better than hydroxides of the metal ions [11].

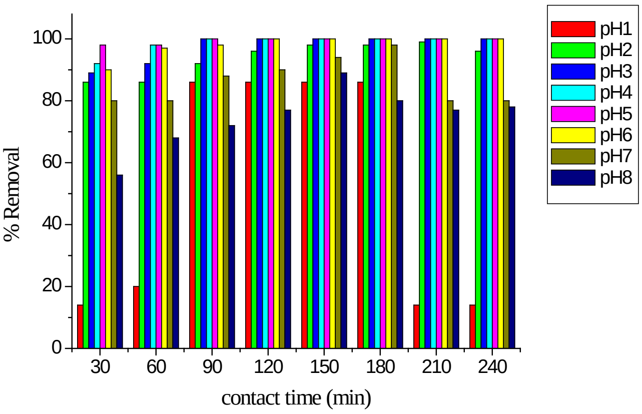

Fig. 1. Effect of pH and contact time on Hg(II) adsorption by palm shell powder (initial Hg concentration — 0.1 mg/L, amount of adsorbent — 4.0 g/L, temperature — 30°C).

The effect of contact time on the removal of mercury(II) by the palm shell powder shows that there is rapid adsorption for the first 60 min, gradually approaching equilibrium between 60–90 min. The maximum removal was found to be ≈98%. The initial faster rate may be due to the availability of the uncovered surface area of the adsorbents, since the adsorption kinetics depends on the surface area of the adsorbents. The Hg^{2+} adsorption takes place at more reactive sites. As these sites are progressively filled, the sorption becomes more difficult, and the sorption process tends to be more unfavourable. These

Table 1
Physical properties of palm shell powder

Surface area	
Single point surface area at P/P_o 0.20033838, m^2/g	2.0989
BET surface area, m^2/g	2.5174
BJH adsorption cumulative surface area of pores between 17.000000 and 3000.000000 A diameter, m^2/g	2.2295
BJH desorption cumulative surface area of pores between 17.000000 and 3000.000000 A diameter, m^2/g	1.1850
Pore volume	
Single point adsorption total pore volume of pores less than 803.8897 A diameter at P/P_o 0.97531489, cm^3/g	0.003149
Pore size	
Adsorption average pore diameter (4V/A by BET), A	50.0309
BJH adsorption average pore diameter (4V/A), A	104.5516
BJH desorption average pore diameter (4V/A), A	173.6304
Bulk density, g/L	2.82×10^{-4}
Moisture content, %	0.4186
Ion exchange capacity, meq/g	0.0041
Water soluble content, g	0.0344
Acid soluble content, g	0.095
pH of adsorbent	6

changes and differences in the metal ion uptake rate could be attributed to 2 different adsorption processes, namely a fast ion exchange followed by chemisorption [12].

3.3. *Effect of initial metal ion concentration*

The adsorbent dose, pH and standing time for the batch experiment were fixed at 0.1 g, 5 and 2 h, respectively. The adsorption process was found to be highly dependent on the concentration of the solution. This is because at low concentrations of adsorbate the ratio of adsorbate to adsorbent is low but at higher concentrations the available sites for adsorption become fewer as the adsorbent has approached its saturation uptake capacity.

3.4. *Effect of sorbent concentration*

The effect of variation in sorbent concentration was studied by varying the initial concentration of the adsorbent from 0.04 g to 0.24 g for fixed initial metal ion concentration of 0.8 mg/L, and it was found that with the increase in the sorbent dose the percentage removal increases but mg of metal ion/g of the sorbent decreases as a greater number of active sites is present on the sorbent in comparison to the metal ions (Fig. 2). The removal of Hg(II) ions was found to increase from 29.39% to 100%, with an adsorbent dose varying from 1.6 g/L to 9.6 g/L. However, with the further increase in the adsorbent dose, there was no appreciable increase in mercury removal.

3.5. *Desorption of mercury from adsorbents*

Hg(II) ions were found to get desorbed using 0.1 N NaCl as eluent. The adsorbent was easily recovered by filtration and could be reused without much change in the efficiency or selectivity of the adsorption process.

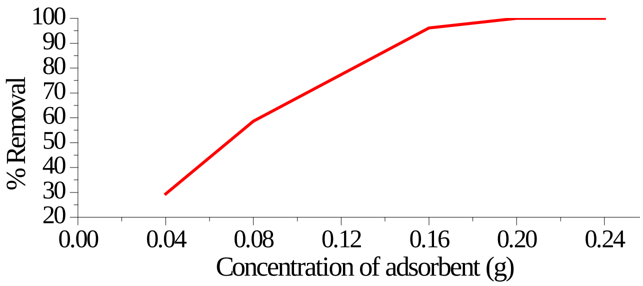

Fig. 2. Effect of adsorbent weight on removal of mercury(II) by palm shell powder (initial Hg concentration —0.8 mg/L, contact time — 180 min, pH — 5, temperature — 30°C).

3.6. *Adsorption dynamics*

The controlling mechanism of the adsorption process such as mass transfer and chemical reaction could be determined using several kinetic models for testing experimental data. For determining the kinetics of adsorption of mercury six kinetic models were used: the pseudo-first-order model, the pseudo-second-order (I, II, III and IV) models, and the intraparticle diffusion model for low initial mercury concentrations (Table 2) discussed and presented in Table 3 and Fig. 3.

In the case of pseudo-first order model data were fitted with poor correlation coefficient showing that the rate of removal of mercury onto palm shell powder does not follow pseudo-first order equation with respect to variation of the adsorbent amount. The pseudo-second order rate equation described the sorption kinetics of mercury(II) with a high correlation coefficient and better than the other equations. The rate constants of the pseudo-second order rate equation was found to be 13.09, 2.303, 1.3, 15.5, 49.8 and 299.3 at adsorbent dosages of 0.04, 0.08, 0.12, 0.16, 0.20 and 0.24 g respectively. q_e increases with the increase in mass of the adsorbent in all the models up to 0.12 g

Table 2
Kinetics models and their linear forms

Kinetics		Linear form	Plot	References
Lagergren	$\mathrm{d}q/\mathrm{d}t = K_1'(q_c - q)$	$\log(q_c - q) = \log(q_c) - K_1't/2.303$	$\log(q_c - q)$ vs. t	[13–15]
First order reversible reaction	$\mathrm{d}X_A/\mathrm{d}t = (K_1 + K_2)(X_{Ac} - X_A)$	$\ln[1 - U(t)] = K_r't$	$\ln[1 - U(t)]$ vs. t	[16–18]
Intraparticle diffusion	$q_t = K_i t^{0.5}$	—	q_t vs. $t^{0.5}$	[18,19]
Pseudo second order	$q_t = q_c^2 K_2' t/(1 + q_c K t)$	$t/q_t = 1/K_2' q_c^2 + t/q_c$	t vs. t/q_t	[20–22]
		$1/q_t = (1/K_2' q_c^2) 1/t + 1/q_c$	$1/q_t$ vs. $1/t$	
		$q_t = q_c - (1/K_2' q_c^2) q_t/t$	q_t vs. q_t/t	
		$q_t/t = K_2' q_c^2 - K_2' q_c q_t$	q_t/t vs. q_t	

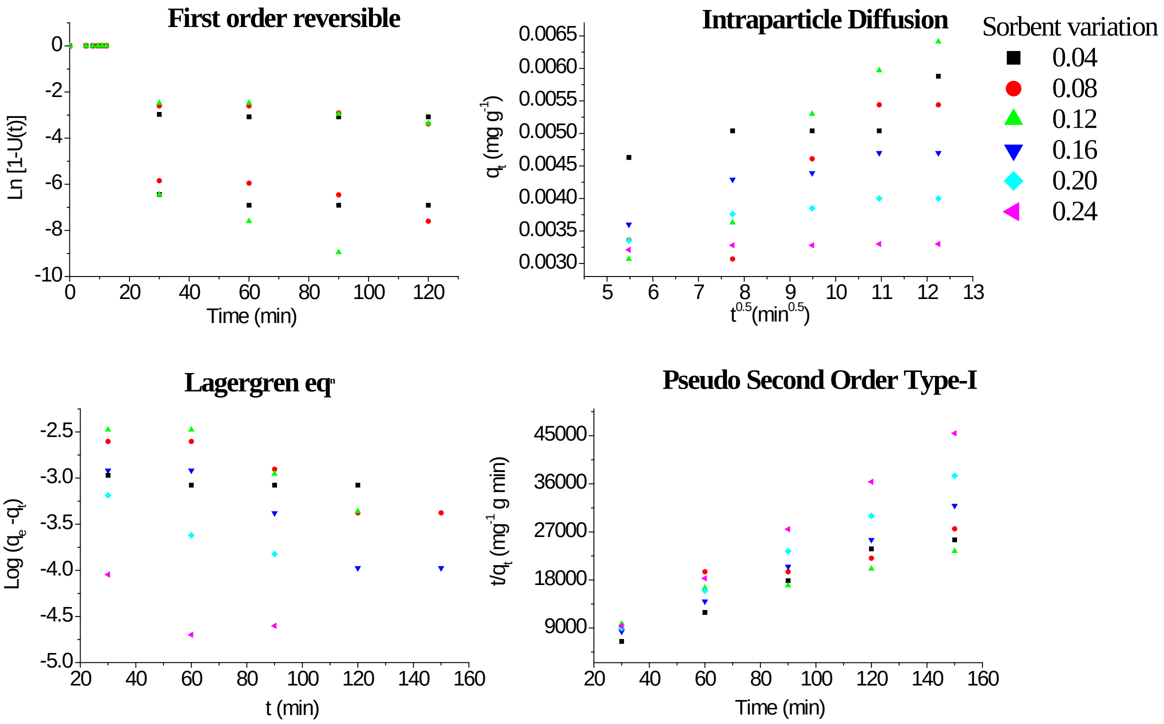

Fig. 3. Kinetics of sorbent variation on removal of mercury(II) by palm shell powder (initial Hg concentration — 0.1 mg/L, pH — 5, contact time — 180 min, temperature — 30°C).

and then there is a decrease in the adsorption capacity because the mg of metal ion/g of sorbent increases until a comparable number of active sites is present but decreases due to a greater number of active sites as compared to the metal ions present in the solution. A decrease in q_e with the increase in the adsorbent mass was also observed by Abdelwahab for copper removal [22].

3.7. Adsorption isotherms

The equilibrium adsorption isotherms data are important to develop an equation which accurately represents the results and will be useful for designing the bio sorption reactors. Equations for the isotherm models studied are given in Table 4. The specific sorption of mercury (C_e/q_e) against the equilibrium concentration (C_e) and $1/C_e$ against $1/q_e$ were plotted to determine the equilibrium constants q_m and K_a from the slope and intercept (Table 5). Linearity of the plots indicates the validity of Langmuir adsorption isotherm, consequently suggesting the formation of monolayer coverage of the adsorbate on the surface of the adsorbent in the concentration range studied. The q_m values of the Langmuir plots were found to increase with the increase in pH till pH 7.0 followed by a decrease from pH 7.0 probably due to precipitation. Isotherm data reveals high K_a values indicating high adsorption affinity. The Freundlich model as observed from the high correlation coefficients (r^2) was also found to be linear. Values of Freundlich constants are depicted in Table 5. Since the value of $1/n$ is less than 1, it indicates favourable adsorption. A smaller value of $1/n$ indicates better adsorption mechanism and formation of relatively stronger bond between adsorbate and adsorbent [24]. The results show that r^2 value is greater than 0.95. The linear analysis using different forms of the Langmuir equations showed that all of them provided a good fit to the experimental data thus suggesting that adsorption of mercury ions onto palm shell powder follows both Freundlich and Langmuir isotherms.

3.8. Gibbs free energy

The increase or decrease in adsorption with the change in thermodynamic property ΔG^0 can be calculated from the following relationship:

$$\Delta G^0 = -RT \ln K \qquad (2)$$

The equilibrium constant K from the Langmuir plots defines the free energy changes and is described in Table 5. The negative values of free energy changes indicate the spontaneous nature of adsorption.

3.9. Comparison of commercial adsorbents with present study

The permissible limit for mercury concentration in

Table 3
Kinetics parameters for sorbent variation

Type		Weight of adsorbent (g)	0.04	0.08	0.12	0.16	0.20	0.24
		Parameters						
Pseudo 1st order		q_e (exp.), mg/g	0.00107	0.00531	0.00925	0.00331	0.00124	0.00013
		K_1', min^{-1}	2172.64	297.16	220.81	217.67	216.85	248.44
		r^2	0.6	0.891	0.897	0.897	0.959	0.6224
Pseudo 2nd order	Type 1	q_e (exp.), mg/g	0.006	0.00757	0.00973	0.00512	0.00422	0.00332
		H, mg/gmin	0.00047	0.00013	0.00012	0.00041	0.00055	0.0033
		K_2', g/mgmin	13.09	2.303	1.3034	15.5525	49.889	299.39
		r^2	0.975	0.864	0.928	0.999	0.999	0.999
	Type 2	q_e (exp.), mg/g	0.00562	0.00635	0.00806	0.00512	0.00422	0.0033
		H, mg/gmin	0.00083	0.00023	0.00015	0.00041	0.00054	0.00315
		K_2', g/mgmin	26.493	5.84	2.38	15.6	30.52	288.59
		r^2	0.6173	0.972	0.891	0.983	0.993	0.945
	Type 3	q_e (exp.), mg/g	0.00564	0.00638	0.00824	0.00511	0.00422	0.00332
		H, mg/gmin	0.00082	0.00023	0.00015	0.00041	0.00054	0.0032
		K_2', g/mgmin	25.693	5.732	2.25554	15.686	30.35	288.85
		r^2	0.516	0.919	0.724	0.9696	0.987	0.943
	Type 4	q_e (exp.), mg/g	0.00458	0.00013	0.00013	0.00040	0.00054	0.00302
		H, mg/gmin	0.0613	0.00794	0.00952	0.00513	0.00423	0.00333
		K_2', g/mgmin	2924.7	455547	578207	32019.2	14796.3	364.43
		r^2	0.514	0.383	0.725	0.9697	0.987	0.96
Intraparticle		k_i, mg/gmin$^{0.5}$	0.00014	0.00038	0.00054	0.00016	0.0001	0.00002
		r^2	0.7048	0.8204	0.958	0.9076	0.9010	0.80734
1st order reversible reaction		K_c	0.4164	1.416	1.0595	25.117	—	—
		K_r'	0.04762	0.0527	—	0.0934	—	—
		r^2	0.551	0.707	—	0.832	—	—

Initial Hg concentration 0.1 mg/L, pH 5, contact time 180 min, temperature 30°C

Table 4
Adsorption isotherms and their linear forms [23]

Isotherm		Linear form	Plot
Freundlich	$q_e = K_f C_e^{1/n}$	$\log q_e = \log K_f + \frac{1}{n} \log C_e$	$\log q_e$ vs. $\log C_e$
Langmuir I	$q_e = \frac{q_m K_a C_e}{1 + K_a C_e}$	$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_a q_m}$	$\frac{C_e}{q_e}$ vs. $\frac{1}{C_e}$
Langmuir II		$\frac{1}{q_e} = \left[\frac{1}{K_a q_m}\right] \frac{1}{C_e} + \frac{1}{q_m}$	$\frac{1}{q_e}$ vs. $\frac{1}{C_e}$
Langmuir III		$q_e = q_m - \left[\frac{1}{K_a}\right] \frac{q_e}{C_e}$	q_e vs. $\frac{q_e}{C_e}$
Langmuir IV		$\frac{q_e}{C_e} = K_a q_m - K_a q_e$	$\frac{q_e}{C_e}$ vs. q_e

Table 5
Isothermal parameters (initial Hg concentration 0.1 mg/L, contact time 180 min, temperature 30°C, amount of adsorbent 4 g/L)

Isotherm	pH	1	2	3	4	5	6	7	8
Langmuir I	q_m, mg/g	–0.013	–0.041	0.0925	0.1288	0.1365	0.133	0.1169	0.106
	K_a, dm^3/mg	–4.136	–8.162	148.58	45.8	41.68	32.92	182.36	37.32
	R^2	0.954	0.828	0.954	0.956	0.963	0.979	0.973	0.97
	ΔG, kJ/mol	—	—	–12596	–9629.3	–9392.3	–8797.9	–13108	–9113.9
Langmuir II	q_m, mg/g	0.016	0.0466	0.0973	0.116	0.1275	0.143	0.156	0.118
	K_a, dm^3/mg	3.66	1.4706	90.57	45.66	55.59	28.25	92.59	28.57
	R^2	0.967	0.992	0.971	0.945	0.944	0.931	0.954	0.998
Langmuir III	q_m, mg/g	0.009	0.003	0.0915	0.1199	0.1346	0.1317	0.1466	0.109
	K_a, dm^3/mg	102.8	18.5	97.5	38.14	40.6	29.74	108.6	65.62
	R^2	0.971	0.979	0.906	0.904	0.956	0.964	0.987	0.942
Langmuir IV	q_m, mg/g	4.54	18.11	90.58	34.473	26.93	28.72	95.41	61.84
	K_a, dm^3/mg	0.011	0.0043	0.095	0.125	0.1244	0.134	0.154	0.111
	R^2	0.97	0.979	0.906	0.904	0.663	0.966	0.879	0.967
Freundlich	K_f, (mg/g)(dm^3/mg)$^{1/n}$	0.206	0.456	1.027	0.348	0.24	0.332	0.396	0.3
	N	0.747	1.1198	1.478	2.15	2.32	2.008	2.572	1.896
	R^2	0.97	0.972	0.982	0.865	0.889	0.981	0.974	0.966

water stream is 1 µg/L. Comparison of commercial adsorbents for water treatment and the adsorbent used in the present study at low metal ion concentrations was done as shown in Table 6. Although direct comparison of the *Borassus flabliffer* as an adsorbent with other sorbent materials is not feasible owing to different applied experimental conditions it was found that in the case of Table 6 it was mentioned that they are good at removing mercury ions at higher concentrations but at lower concentrations they even do not reach 50% removal. The performance data reported in the literature suggest that activated carbons can achieve a residual level of 0.5 µg/L, which is not permissible. Whereas the Purolite s-920 Hg specific chelating resin, which is specifically prepared to remove Hg ions, can go up to a residual level of 0.34 µg/L, which is again not permissible. In the present study *Borassus Flabeliffer* could achieve 100% removal at low concentrations and is comparable to other non-conventional and commercial adsorbents at higher concentrations.

4. Conclusion

Palm shell powder has been effectively used as adsorbent for the removal of Hg(II) from a solution. The adsorp-

Table 6
Comparison table

No.	Material	Mercury concentration (µg/L)		Percentage removal	References
		Initial	Final		
1.	Powdered activated carbon	10, 000	4, 000	60	[24]
2.	Powdered activated carbon	10	0.5	50	[25]
3.	Gas activated carbon	0–100	<1.0	>41	[26]
4.	Gas activated carbon	1.5	0.8	47	[25]
5.	Purolite s-920 Hg specific chelating resin	10.67	0.34	96.8	[27]
6.	Carbon aerogel	1000	—	≈100	[28]
7.	Mustard husk	4000	—	56.2	[29]
8.	Sawdust	4000	—	68.3	[29]
7.	*Borassus Flabeliffer*	100	0.0	100	Present
8.	*Borassus Flabeliffer*	800	0.0	100	Present
9.	*Borassus Flabeliffer*	10,000	3760	62.4	Present

tion was found to be influenced by various parameters like initial pH, adsorbent dosage, contact time and initial Hg(II) concentration. The maximum uptake of Hg(II) occurred at initial pH of 3.0–7.0. The adsorption increased with increasing the dose of the adsorbent and decreased with the initial mercury(II) concentration. Equilibrium was achieved after 120 min and ≈ 98% removal of mercury was possible at the working pH of 5.0 and adsorbent dosage of 4 g/L. Kinetic and isotherm studies reveal that palm shell powder can be effectively employed for the adsorption of Hg(II). Adsorption follows both Freundlich and Langmuir isotherm models. Column adsorption experiments demonstrate the potential capability of palm shell powder to be used for removing mercury from aqueous solutions. The experimental sorption capacity of palm shell powder is comparable to other low-cost conventional sorbents.

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