



## Pb(II) biosorption from hazardous aqueous streams using *Gossypium hirsutum* (Cotton) waste biomass

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### ABSTRACT

Studies on the biosorptive ability of *Gossypium hirsutum* (Cotton) waste biomass outlined that smaller size of biosorbent (0.355 mm), higher biomass dose (0.20 g), 5 pH and 100 mg/L initial Pb(II) concentration were more suitable for enhanced Pb(II) biosorption from aqueous medium. The Langmuir isotherm model and pseudo second order kinetic model fitted well to the data of Pb(II) biosorption. Highly negative magnitude of Gibbs free energy ( $\Delta G^\circ$ ) indicated that the process was spontaneous in nature. In addition to this surface coverage and distribution coefficient values of Pb(II) biosorption process were also determined. At optimized conditions Pb(II) uptake was more rapid in case of industrial effluents in comparison to synthetic solutions. FTIR spectroscopic analysis revealed that the main functional groups involved in the uptake of Pb(II) on the surface of *G. hirsutum* biomass were carboxyl, carbonyl, amino and alcoholic.

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## 1. Introduction

Heavy metals released into the environment have been increasing continuously as a result of industrial activities and technological development, posing a significant threat to the environment and public health because of their toxicity, accumulation in the food chain and persistence nature [1–3]. Exposure to heavy metal contamination has been found to cause kidney damage, liver damage, and anemia in low doses, and in high concentrations, heavy metals can be carcinogenic [4]. Pb(II) is neurotoxic. Numerous studies indicate that blood Pb(II) concentration above 100  $\mu\text{g/L}$  is associated with adverse outcomes on measures of intellectual functioning [5,6]. The conventional technologies used for removing heavy metal ions from aqueous streams include chemical precipitation; lime coagulation; ion exchange; membrane technologies; reverse osmosis and phytoremediation, but these are very costly [7–9].

Biosorption utilizes biological materials as adsorbents, and this method has been studied by several researchers as an alternative technique to conventional methods for the heavy metal removal from wastewater. The use of non-living biomaterial containing metal-binding compounds would have the advantage of not requiring utmost care and maintenance as well as being useful in

remediating areas with high levels of the heavy metal contaminants [10–14].

*Gossypium hirsutum* is grown all over the world as a major fiber crop. It is tropical/sub tropical crop, grown in summer [15]. *G. hirsutum* stem, leaves and bolls after the collection of cotton become waste. Similarly after the extraction of oil from cotton seeds, its cake (also used for animal feed) is low of cost and easily available throughout the world. According to our knowledge this most abundant waste material was never used for the biosorption of any heavy metal including Pb(II). So the present study was undertaken with the aim to access the maximum adsorption capacity ( $q_{\text{max}}$ ) of *G. hirsutum* waste biomass for Pb(II) from aqueous solution and industrial effluents. The functional groups involved in the adsorption process were determined by FTIR spectroscopic analysis. The effect of other important parameters such as biosorbent dose, biosorbent size, effect of initial Pb(II) concentration and sorption time on Pb(II) uptake by *G. hirsutum* waste biomass was also studied to completely understand the sorption process.

## 2. Materials and methods

### 2.1. Reagents

All the chemicals used in the present study were of analytical reagent grade, including  $\text{Pb}(\text{NO}_3)_2$ , concentrated HCl, concentrated

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H<sub>2</sub>SO<sub>4</sub>, NaOH (Merck) and Pb(II) atomic absorption spectrometry standard solution (1000 mg/L) (Fluka Chemicals).

## 2.2. *G. hirsutum* waste biomass

Different parts of *G. hirsutum* (Cotton) waste biomass viz. stem, leaves, bolls and seed cake were collected randomly from local fields near Faisalabad, Pakistan. Collected samples were washed with distilled water extensively, to remove the dust particles. Subsequently samples were oven dried for 72 h. The dried biomass was ground using food processor (Moulinex, France) and sieved through mechanical sieve (OCTA-DIGITAL 4527-01) to obtain biosorbent with known particle size. The sizes selected for the present study were 0.255, 0.355, 0.710 and 0.100 mm. These fractions were stored in air tight plastic bottles at standard temperature and pressure (STP) for further experiments [16].

## 2.3. Pb(II) solutions

Stock aqueous solution of Pb(II) having concentration 1000 mg/L was prepared by dissolving 1.59 g of Pb(NO<sub>3</sub>)<sub>2</sub> in 100 mL of deionized distilled water (DDW) and quantitatively diluting to 1000 mL using DDW. Pb(II) solutions of the different concentrations on desire were prepared by adequate dilution of stock solution with DDW.

## 2.4. Batch biosorption studies

In all the sets of experiments, 100 mL of Pb(II) aqueous solution of varied concentrations (25, 50, 100, 200, 400 and 800 mg/L) was taken in conical flask (250 mL) and mixed with desired biosorbent dose (0.05, 0.1, 0.15 and 0.2 g) at different biosorbent sizes (0.355, 0.500, 0.710 and 1.00 mm), pH (1–6) for various contact times (15, 30, 45, 60, 120, 240, 360, 480, 600, 840 and 1440 min) at 30 ± 1 °C and finally agitated for 24 h on orbital rotatory shaker (PA 250/25. H). The solution pH was adjusted using 0.1 N HCl and 0.1 N NaOH to appropriate value. The samples were finally filtered through Whatman filter paper no. 1 and stored in plastic bottles at 4 °C to reduce the risk of the evaporation of the stored samples [17–19].

## 2.5. Sample collection from industrial effluents

The wastewater samples from different industries such as glass industry (G.I.), ceramic industry (C.I.) and paint industry (P.I.) were collected from Sheikhpura, Gujranwala and Lahore, Pakistan, respectively. Triplicate samples were collected and stored in pre-cleaned plastic bottles and placed in a cooler for transportation to the research laboratory [41]. For the biosorption of Pb(II) from the hazardous aqueous streams by *G. hirsutum* (Cotton) seed cake biomass experiments were performed in triplicates at optimized conditions of biosorbent size (0.355 mm), biosorbent dose (0.2 g), pH (5.0) and contact time up to 360 min taking 100 mL of each industrial effluent sample.

## 2.6. Pb(II) analysis

Pb(II) concentration was analyzed using PerkinElmer Analyst 300 Atomic Absorption Spectrophotometer (AAS) using an air-acetylene flame [18,25]. The hollow cathode lamp was operated at 15 mA and analytical wavelength was set at 283.3 nm.

## 2.7. Metal uptake (*q*)

Metal uptake (*q*) was calculated using mass balance equation *q* (mg/g dry biomass) (Eq. (1)) [20].

$$q = \frac{V(C_i - C_e)}{m} \quad (1)$$

where “*V*” is the sample volume (mL), “*C<sub>i</sub>*” and “*C<sub>e</sub>*” are the initial and equilibrium metal concentrations (mg/L), respectively, and “*m*” is the amount of dry biomass (g).

## 2.8. Fourier Transform Infrared (FTIR) spectroscopy

FTIR spectroscopy was used to detect vibration frequency changes in *G. hirsutum* seed cake biomass before and after Pb(II) uptake. The spectra were collected by PerkinElmer spectrometer within the range 4000–400 cm<sup>-1</sup> using chloroform as mulling agent. The background obtained from the scan of pure chloroform was automatically subtracted from the sample spectra.

## 2.9. Statistical analysis

All experiments were run in triplicate and the arithmetic mean of the results was considered in data analysis. All statistical analysis was done using Microsoft Excel 2004, version Office Xp (Microsoft Corporation, USA) and Slidewrite software.

## 3. Results and discussion

### 3.1. Effect of biosorbent dose

The effect of biosorbent dosage was analyzed at pH 5.0, biosorbent size 0.355 mm and Pb(II) concentration 100 mg/L. The percentage adsorption of Pb(II) was increased on increasing biosorbent dose from 0.05 to 0.2 g/100 mL (Fig. 1). The increase in percentage Pb(II) adsorption with an increase in biosorbent concentration can be attributed to increase in binding sites. However, adsorption capacity (*q*) decreased with increase in biosorbent dose [21–23]. But these results were not surprising, as in all the biosorbent dosage experiments, percent metal adsorption is inversely related with metal uptake capacity of biosorbent. The decrease in adsorption capacity (*q*) was observed because higher biosorbent dose caused electrostatic interactions between cells and interference between the binding sites [24].

### 3.2. Effect of biosorbent size

Fig. 2 shows the effect of different sizes of biosorbent on adsorption of Pb(II) at pH 5.0, biosorbent dose 0.2 g/100 mL, concentration

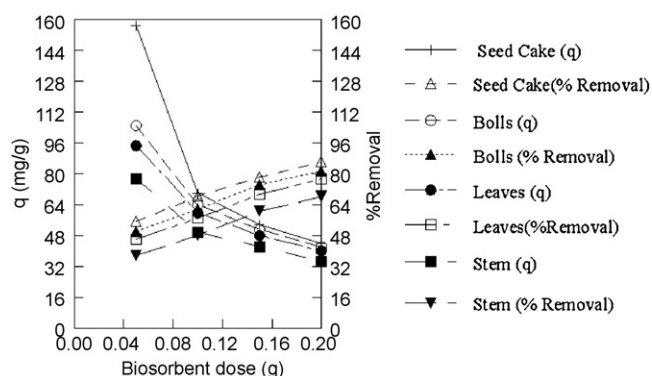


Fig. 1. Effect of biosorbent dose on Pb(II) biosorption by *G. hirsutum* waste biomass.

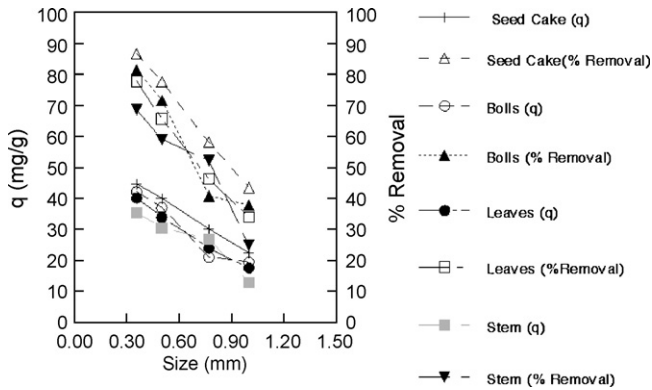


Fig. 2. Effect of biosorbent size on Pb(II) biosorption by *G. hirsutum* waste biomass.

100 mg/L for contact time of 24 h. From the same figure it is evident that, smaller the granular size of biosorbent, larger the adsorption capacity ( $q$ ). The optimum adsorption capacity ( $q$ ) was observed at smallest biosorbent size (0.355 mm) was 44.67, 35.44, 40.12 and 42.08 mg/g for seed cake, stem, leaves and bolls of *G. hirsutum* waste biomass, respectively. Smaller the size of biosorbent greater its surfaces area, resultant increase in the binding sites for adsorption [18].

### 3.3. Effect of pH

The pH dependence of metal uptake could be related to the functional groups of the biomass and solution chemistry. Fig. 3 shows the effect of pH on the adsorption of Pb(II) by *G. hirsutum* waste biomass. Adsorption capacity ( $q$ ) was analyzed over a pH range 1.0–6.0. It was apparent from the results that the metal adsorption was a function of pH; as the pH increased from 1.0 to 5.0, both adsorption capacity and percentage adsorption increased. The experiments beyond pH 5 were not carried as the Pb(II) precipitates at pH higher than 5.0. The maximum adsorption capacities (mg/g) for the different parts of *G. hirsutum* waste biomass were seed cake (45.01) > bolls (41.56) > leaves (38.62) > stem (34.42). Biosorbent materials primarily contain weak acidic and basic functional groups. It follows the theory of acid-base equilibria, in the pH range 1.0–5.0, and in this range the binding of heavy metal cations is determined primarily by the state of dissociation of the weak acidic groups. Carboxyl groups ( $-\text{COOH}$ ) are the important groups for the metal uptake by biological materials [25]. The ionic states of cell wall functional groups can be used to explain the pH dependency of the biosorption. Low pH conditions allow hydrogen and hydronium ions to compete with Pb(II) for metal-binding sites on the biomass, causing poor Pb(II) uptake. At higher pH values 3–5, there are lower

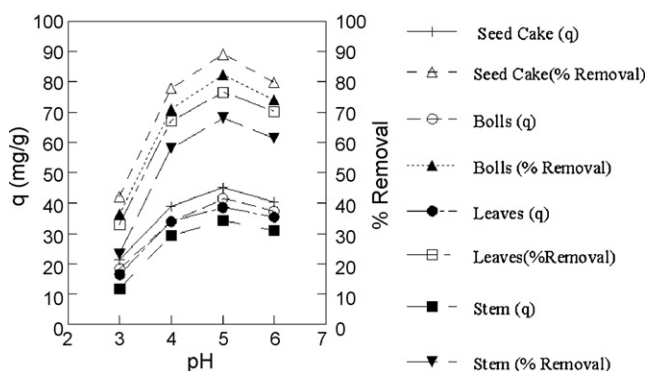


Fig. 3. Effect of pH on Pb(II) biosorption by *G. hirsutum* waste biomass.

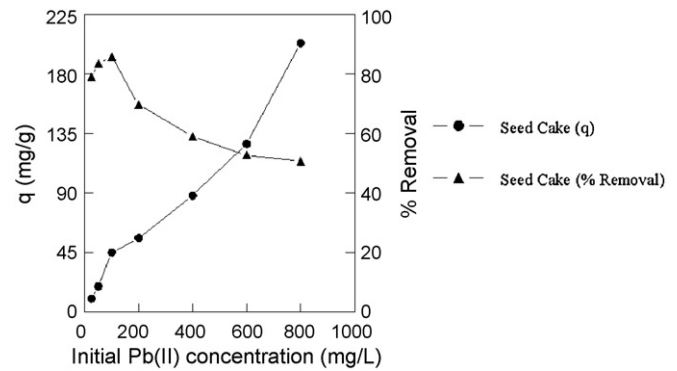


Fig. 4. Effect of initial Pb(II) concentrations on biosorption by *G. hirsutum* seed cake biomass.

numbers of competing hydrogen ions and more ligands are exposed with negative charges, resulting in the greater Pb(II) sorption. It may be explained that at low pH, the overall surface charge on the cells became positive, which inhibited the approach of positively charged Pb(II) cations [26–28]. At the pH higher than 5.0 the hydroxy complexes were formed [31] which caused decrease in adsorption capacity [29,30]. Maximum Pb(II) uptake (mg/g) was observed for seed cake (45.01) at pH 5.0, so all further experiments were conducted using seed cake of *G. hirsutum* at evaluated pH 5.0 [32].

### 3.4. Effect of initial Pb(II) concentrations

The rate of adsorption was a function of the initial Pb(II) concentration. Fig. 4 shows the effect of different initial Pb(II) concentrations on Pb(II) adsorption using seed cake biomass of *G. hirsutum*. The increase in initial Pb(II) concentration resulted in increase in the percentage adsorption from 25 to 800 mg/L with a maximum adsorption at 100 mg/L [16,33–35]. The Langmuir (Fig. 5) and Freundlich adsorption isotherms [36] are commonly used to model the biosorption studies based on metal concentration (Figs. 6).

Langmuir equation transforms to the linearized form (Eq. (2)) [21].

$$\frac{1}{q_e} = 1/q_{\max} + 1/(q_{\max}K_L)C_e \quad (2)$$

where  $q_e$  (mg/g) is the Pb(II) ion sorbed,  $C_e$  (mg/L) is the equilibrium concentration of Pb(II) ion solution,  $q_{\max}$  (mg/g) is the maximum amount of Pb(II) ion which can be taken up by biosorbent, and  $K_L$  ( $\text{L mg}^{-1}$ ) is the Langmuir constant. The Langmuir equation assumes that (i) the surface consists of adsorption sites (ii) all adsorbed

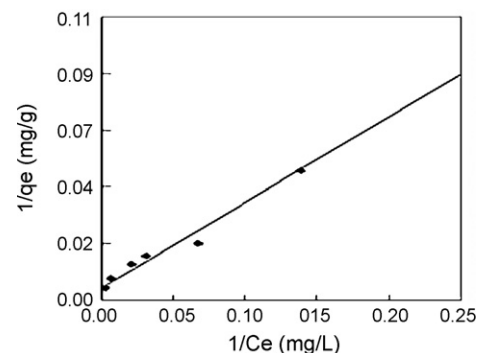


Fig. 5. Langmuir isotherm for Pb(II) biosorption by *G. hirsutum* seed cake biomass.

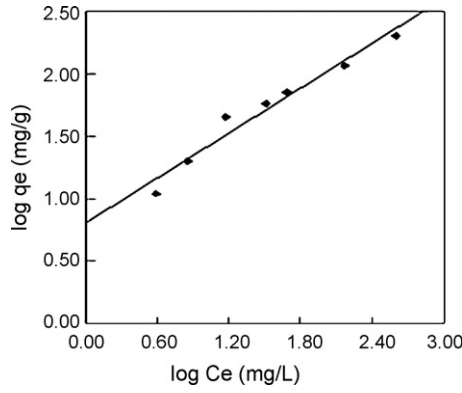


Fig. 6. Freundlich isotherm for Pb(II) biosorption by *G. hirsutum* seed cake biomass.

species interact only with a site and not with each other and (iii) the adsorption is limited to monolayer. It is then assumed that once a metal ion occupies a site, no further sorption can take place. This suggested that all binding sites are energetically equivalent and there is no migration or interaction between the adsorbed ions on the surface area [37].

The Freundlich equation is an empirical relationship describing the adsorption of solutes from a liquid to a solid surface. The general form of this model is:

$$q_e = K_F C^{1/n}$$

This can be linearized by taking logarithm of both sides of the equation to give:

$$\log q_e = \log K_F + 1/n \log C_e \quad (3)$$

where  $K_F$  and  $1/n$  are the Freundlich constants.

The Langmuir model fitted better to experimental data in comparison to the model of Freundlich as represented from the value of its constants,  $q_{max}$  and  $R^2$  (Table 1).

### 3.5. Separation factor ( $R_L$ )

A dimensionless constant, separation factor ( $R_L$ ) can be used to predict whether a sorption system is favorable or unfavorable in batch adsorption process [38].  $R_L$  values between 0 and 1 represent the favorable isotherm.  $R_L$  was calculated from Langmuir isotherm based equation (Eq. (4)) [18].

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

where  $K_L$  is the constant from Langmuir equation and  $C_i$  is the initial Pb(II) concentration. The separation factor ( $R_L$ ) profile of adsorption of Pb(II) on seed cake of *G. hirsutum* is presented in Fig. 7. The parameter,  $R_L$ , indicates the shape of the isotherm and the nature of the sorption process as given below:

- $R_L > 1$  = unfavorable isotherm.
- $R_L = 1$  = linear isotherm.
- $R_L = 0$  = irreversible isotherm.
- $0 < R_L < 1$  = favorable isotherm.

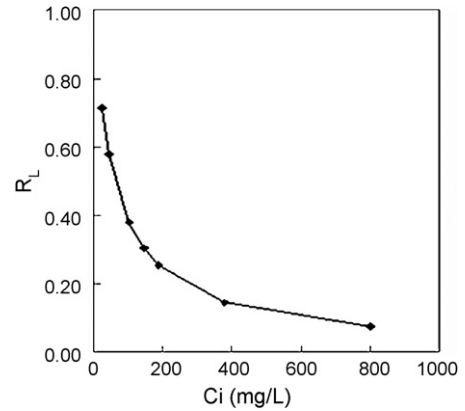


Fig. 7. Separation factor profile for biosorption of Pb(II) as function of initial metal concentration by *G. hirsutum* seed cake biomass.

The values of  $R_L$  for Pb(II) were calculated and plotted against initial metal ion concentration. Favorable isotherm (Fig. 7) showed that, the sorption of Pb(II) on the seed cake biomass of *G. hirsutum* increased as the initial metal ion concentration increased from 25 to 800 mg/L, indicating that adsorption is even favorable for the higher initial metal ion concentrations. The sorption process was favorable for Pb(II) removal at all concentrations investigated. From the figure it can be noted that adsorption process is favorable on low as well as high concentrations [3].

### 3.6. Surface coverage ( $\theta$ )

The fraction of biomass surface covered by Pb(II) was studied using Langmuir type equation (Eq. (5)). By plotting the surface coverage value ( $\theta$ ) against Pb(II) concentration (Fig. 8) [18].

$$\theta = K_L C_i (1 - \theta) \quad (5)$$

where  $K_L$  is the Langmuir adsorption coefficient and  $C_i$  is the initial Pb(II) concentration.

The increase in initial Pb(II) concentration from 25 to 500 mg/L for *G. hirsutum* seed cake biomass increases the surface coverage on the biomass until the surface is nearly fully covered with a mono-

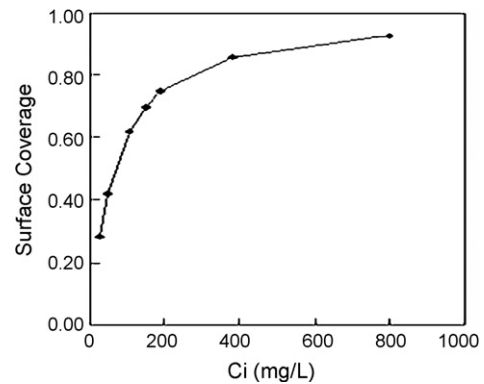


Fig. 8. A plot of surface coverage ( $\theta$ ) against initial Pb(II) ion concentration ( $\text{mg L}^{-1}$ ) for *G. hirsutum* seed cake biomass.

Table 1  
Langmuir and Freundlich isotherm parameters for Pb(II) uptake by *G. hirsutum* seed cake biomass

Experimental	Langmuir isotherm parameters			Freundlich isotherm parameters			
	$K_L$ (L/mg)	$q_{max}$ (mg/g)	$R^2$	$q_{max}$ (mg/g)	$K_F$ (mg/g)	$1/n$	$R^2$
203.05	0.01546	196.07	0.9918	228.77	6.42	0.597	0.9604

**Table 2**Comparison between the Lagergren pseudo first order and pseudo second order kinetic models for Pb(II) uptake by *G. hirsutum* seed cake biomass

Experimental	Pseudo first order kinetic model			Pseudo second order kinetic model		
	$q_e$ (mg/g)	$k_{1,ads}$ ( $\text{min}^{-1}$ )	$R^2$	$q_e$ (mg/g)	$k_{2,ads}$ (mg/g min)	$R^2$
44.67	35.23	$1.24 \times 10^{-2}$	0.971	50	$4.86 \times 10^{-3}$	0.997

layer (Fig. 8). At concentration higher than 500 mg/L, the metal adsorption rate on biomass surface becomes independent of the Pb(II) concentration. This happens because the surface coverage ceases very significantly with concentration of Pb(II) at higher levels.

### 3.7. Distribution coefficient ( $D$ )

Distribution coefficient ( $D$ ) can be used to predict the ratio of Pb(II) concentration in aqueous phase to the Pb(II) concentration adsorbed on *G. hirsutum* surface (Table 2). A value of  $D$  greater than 0.50 suggests that, the more number of Pb(II) ions are adsorbed on biomass surface in comparison to the concentration of Pb(II) in aqueous phase. Distribution coefficient ( $D$ ) is “the ratio of metal ion concentration in the adsorbent phase, to the metal ion concentration in aqueous phase” [18].

### 3.8. Gibbs free energy ( $\Delta G^\circ_{ads}$ )

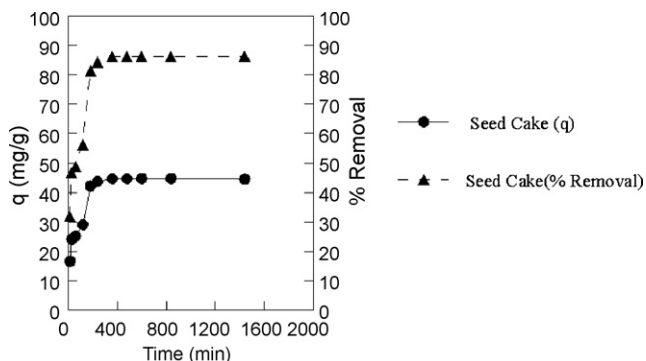
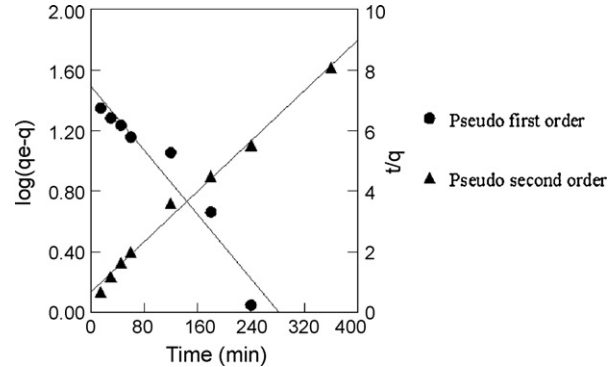
To judge whether the adsorption process is spontaneous or non-spontaneous, the value of  $\Delta G^\circ_{ads}$  was calculated using Bockers Swinkel's adsorption isotherm (Eq. (6)) [39]

$$G^\circ_{ads} = -2.303 RT \log \left[ \frac{55.4\theta}{C_i(1-\theta)} \right] \{ \theta + n(1-\theta)n^{-1}/n^n \} \quad (6)$$

where  $C_i$  is the initial concentration of Pb(II) ion in the solution and  $n$  is charge on metal. The values of  $\Delta G^\circ_{ads}$  are highly negative on all concentrations suggesting that the Pb(II) adsorption process by *G. hirsutum* seed cake is spontaneous in nature on low as well as high concentrations (Table 2).

### 3.9. Effect of kinetics on biosorption of Pb(II)

Fig. 9 indicates the effect of contact time on adsorption of Pb(II) by seed cake biomass. Percentage adsorption at different time intervals ranging from zero time to 24 h was analyzed. Optimum adsorption capacity ( $q$ ) and percentage adsorption were 44.67 mg/g and 86.46% respectively. Kinetic study revealed that adsorption took place in two phases, a rapid surface adsorption within 15 min followed by slow intracellular adsorption before the equilibrium was established after 6 h [40].

**Fig. 9.** Effect of kinetics on Pb(II) biosorption by *G. hirsutum* seed cake biomass.**Fig. 10.** Comparison between pseudo first order and pseudo second order for Pb(II) biosorption by *G. hirsutum* seed cake biomass.

Kinetics of adsorption by any biological material has been widely tested by the first order expression given by Lagergren and pseudo second order approach (Fig. 10) [41,42]. The first order Lagergren equation (Eq. (8)):

$$\log(q_e - q) = [\log q_e - \{(k_{1,ads}t)/2.303\}] \quad (8)$$

The pseudo second order equation (Eq. (9)) is given as follows.

$$t/q = 1/k_{2,ads} q_e^2 = t/q_t \quad (9)$$

where  $q_e$  is the mass of metal adsorbed at equilibrium (mg/g),  $q_t$  is the mass of metal at time  $t$  (min),  $k_{1,ads}$  is the first order reaction rate constant of adsorption ( $\text{min}^{-1}$ ),  $k_{2,ads}$  is the pseudo second order rate constant of adsorption ( $\text{mg/g min}^{-1}$ ). A comparison between Lagergren first order and to pseudo second order kinetic model is expressed in Table 3. Pseudo second order kinetic model with higher value of regression coefficient ( $R^2 = 0.997$ ) fitted well to the data.

### 3.10. Pb(II) biosorption from hazardous aqueous streams

Sorption kinetic experiments were performed at optimized conditions in order to investigate sorption time for Pb(II) biosorption from hazardous aqueous streams. Experiments indicated that sorption equilibrium reached much faster in case of industrial wastewater samples (120 min) in comparison to synthetic wastewater (360 min) using same biosorbent (Fig. 11). This may be due to the presence of co-metal ions in the industrial effluents. The percentage sorption from hazardous industrial effluents was in

**Table 3**Apparent Gibbs free energy  $\Delta G^\circ_{ads}$  (kJ/mol) and distribution ratio ( $D$ ) for Pb(II) uptake by *G. hirsutum* seed cake biomass

Ci (mg/L)	$\Delta G^\circ_{ads}$ (kJ/mol)	$D$
25	-28.3160	0.8495
50	-28.1095	0.8468
100	-27.7719	0.8571
150	-27.6267	0.7824
200	-27.5285	0.7460
400	-27.2998	0.6101
800	-27.1399	0.5069



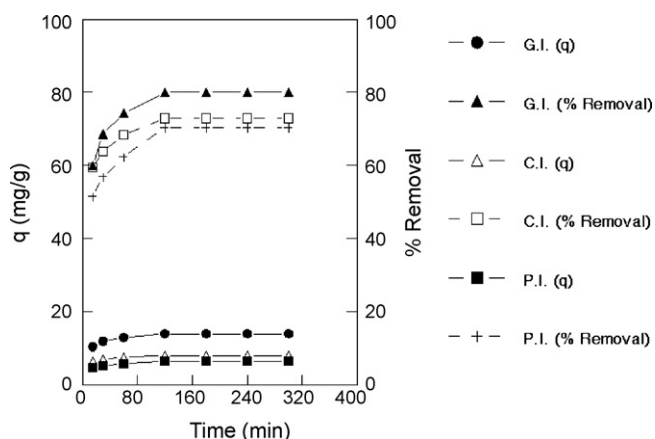


Fig. 11. Pb(II) biosorption from hazardous aqueous streams by *G. hirsutum* seed cake biomass at optimized conditions.

Table 4

Pb(II) biosorption from hazardous industrial effluents by *G. hirsutum* seed cake biomass at optimized conditions

Industries	Initial Pb(II) concentration (mg/L)	q (mg/g)	% Removal
G.I.	35.21 ± 0.25	14.04 ± 0.014	80.85 ± 1.19
C.I.	22.15 ± 0.19	8.075 ± 0.021	72.91 ± 0.85
P.I.	18.57 ± 0.21	6.0543 ± 0.011	70.38 ± 0.92

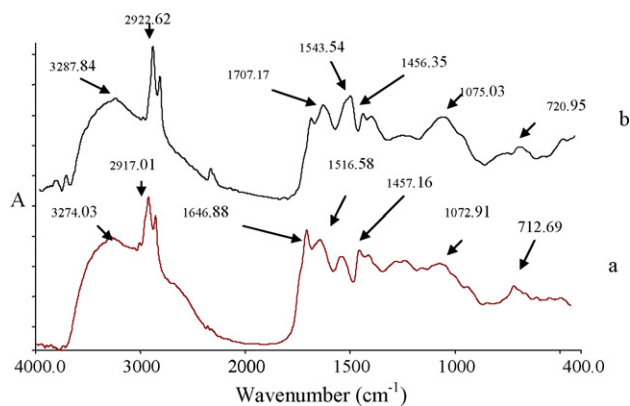


Fig. 12. FTIR spectra of *G. hirsutum* seed cake biomass: (a) before and (b) after Pb(II) sorption.

the following order: G.I. (80.85 ± 1.19%) > C.I. (72.9 ± 0.85%) > P.I. (70.78 ± 0.92%) (Table 4) [18].

### 3.11. FTIR analysis

The functional groups involved in the adsorption of Pb(II) by *G. hirsutum* seed cake waste biomass mass were determined using FTIR spectroscopic analysis. The main functional groups involved in adsorption process were found to be carbonyl, carboxyl, alcoholic and amino groups (Fig. 12). The involvement of these functional groups in metal adsorption process can be judged from change in frequency of absorbing groups (Fig. 12a and b). The absorbance of the peaks in Pb(II) loaded sample was subsequently lower than in unloaded sample [18].

## 4. Conclusions

*G. hirsutum* (Cotton) waste biomass is easily available throughout the world, so it was selected for the removal of Pb(II) from

aqueous solutions. From the results, it was concluded that maximum adsorption of Pb(II) ions from aqueous solutions occurred at pH 5.0, 0.355 mm biosorbent particle size and 0.20 g biosorbent dose of seed cake biomass. At equilibrium and pH 5.0 the maximum Pb(II) uptake (45.01 mg/g) was observed for seed cake. The Pb(II) adsorption on *G. hirsutum* waste biomass was well described by Langmuir adsorption isotherm. The uptake of Pb(II) by *G. hirsutum* seed cake biomass occurred in two phases, a rapid surface adsorption within 15 min followed by slow intracellular adsorption till equilibrium. The whole adsorption process was well described by pseudo second order. The percentage sorption from hazardous aqueous streams was in the following order: G.I. (80.85 ± 1.19%) > C.I. (72.9 ± 0.85%) > P.I. (70.78 ± 0.92%).

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