

Litchi chinensis peel biomass as green adsorbent for cadmium (Cd) ions removal from aqueous solutions

Tariq Mahmood Ansari^a, Shazia Shaheen^a, Suryyia Manzoor^{a,*}, Saima Naz^b, Muhammad Asif Hanif^c

^aInstitute of Chemical Sciences, Bahauddin Zakaryia University, Multan 60800, Pakistan, Tel. +92 3484138516; email: Suryyia878@gmail.com (S. Manzoor), Tel. +92 3007315941; emails: Drtariq2000@gmail.com (T.M. Ansari), sshaheen61@yahoo.com (S. Shaheen)

^bDepartment of Chemistry, University of Education, Multan Campus, Pakistan, Tel. +92 3167428490; email: saimanazuaf@gmail.com

^cNano and Biomaterials Lab, Department of Chemistry, University of Agriculture, Faisalabad 38040, Pakistan, email: drmuhammadasifhanif@gmail.com

Received 8 March 2019; Accepted 24 July 2019

ABSTRACT

An intensive research of the use of waste biomass for water remediation has led to the exploration of novel founts of these materials. One example is *Litchi chinensis* peels. This work describes the use of Litchi peels biowaste for the effective removal of Cadmium(II) ions and the influence of environmental variables like pH, sorbent dose, concentration of sorbate and physico-chemical treatments on Cd(II) adsorption capacity. The optimum experimental conditions for the maximum metal uptake were based on pH 5, particle size 43 μm and a contact time of 60 min. Cd(II) sorption data fitted well to pseudo-second-order kinetic model and Freundlich adsorption isotherms model. According to Freundlich adsorption isotherm the q_{max} was found to be 15.27 mg/g. Fourier-transforms infrared spectroscopic analysis of waste biomass exposed the presence of various alkane, alcohol, amines, and oxygen bound groups. The results of this study revealed that Litchi waste biomass is a promising, inexpensive and eco-friendly bio-material that can easily be used for removing high levels of Cd ions from wastewater.

Keywords: Sorption; *Litchi chinensis*; Water pollution; Cadmium ions; Atomic absorption spectroscopy

1. Introduction

Water being the basic matrix of life is referred as a universal solvent. The quality of the water is greatly influenced by human activities. Pollution, deterioration and depletion of natural water resources occur primarily due to anthropogenic activities. Water pollution mostly occurs when water is adversely affected due to addition of some external materials [1]. When industrial wastewater seeps down, the ground water is also contaminated, which contains a variety of toxic heavy metals (As, Hg, Cu, Cd, Ni, Pb and Cr) [2]. This makes the water unsuitable for the drinking and other household

purposes. The microbial and chemical transformations of these wastes create a negative impact on environment and human health. Heavy metals such as Cd, Zn, Cr, Mn, Fe, Ba, As, Cu, Ag, Pb and Co are the major pollutants especially in water [3–5]. Cadmium is neither essential nor biologically needed metal and occurs in the form of compounds instead of element in nature. Cadmium is very widely used in almost all industries diverse processes. Cadmium is used in nickel-cadmium batteries that are rechargeable and are most widely used in batteries, airplanes, computers, telephones and radar stations [6,7]. Cadmium is also used as stabilizer in plastic, pigment, as an anticorrosive, in electroplating and alloying metal in solders.

* Corresponding author.

According to World Health Organization, the permissible limit of Cd in water is 0.003 mg/L [8]. Cadmium is being a highly toxic metal can lead to bone damage and cause bone pain, low bone mineralization, high rate of osteoporosis and bone fracture if it exceeds this value. Some organ in human body may also store cadmium especially kidney. Kidney damage occurs when chronic exposure of cadmium takes place [9–12]. Cadmium is carcinogenic; it causes lungs and prostate cancer in human. Liver functions are also badly affected by the cadmium inhalation [13–15]. Due to the toxic effects of the cadmium, it is imperative to remove it from the water and its exposure must be reduced. A variety of techniques have been reported for the removal of heavy metals from the water. Some of these conventional methods include ion exchange, solvent extraction, chemical oxidation and reduction, filtration, chemical precipitation, lime coagulation, reverse osmosis and evaporative recovery [16–20]. However in the recent decade, biosorption has gained a huge fame in the area of water purification. Biosorption process can be defined as ability of biological material to accumulate the heavy metal from the wastewater through metabolically mediated or physio-chemical pathway of uptake. Both dead and living biological materials can be used for the metal and pollutant removal from wastewater [21,22]. Different types of biowastes have been used for this purpose. Some of those are, biowaste such as weeds, the residues of plants, bark of trees, sawdust and also cellulose derived material, industrial waste like (food/beverage waste, anaerobic sludge and activated sludge) and waste from agriculture (wheat straw, rice straw, soybean hulls, ruin and vegetable peels) [23–27]. Other than these biowastes, a major progress has been seen in the utilization of algae, bacteria and fungi as biosorbents. These organisms are quite effective metal ion sequesters even at lower concentrations. Not only the living microorganisms with the capacity to accumulate heavy metals are being extensively studied but the dead microorganisms in different researches have also presented high capacity to adsorb metal ions through physical phenomena [28,29]. Another latest advancement in the field of biosorption is the development of biotrickling filters. Yang et al. [30,31] made use of the wasted biofilms from biotrickling filters and reutilized for the removal of heavy metals like Cd(II) and Pb.

Litchi is a fruit cultivated in tropical and subtropical areas. In different regions, it is called as litchi, lychee, laichi, leechie and lichee. It has more than 60 cultivars all over the world which differ in color, form and texture. It is also used in medicine. Many nutrients such as calcium, vitamin C, iron and thiamine are present in litchi [32,33].

Peels of different fruits are one of the important agricultural wastes which can be potential adsorbent for the removal of pollutants from water. For wastewater treatment, a variety of different fruit peels have been investigated [34,35]. Litchi peel is a biowaste which is mostly obtained from juice industry. A thorough literature survey indicated that litchi peel waste, a cheap and abundant agricultural waste, has not been previously investigated as an adsorbent for cadmium ions removal from wastewater.

The objective of this study was to evaluate the adsorption capacity (q) of Litchi waste biomass to develop a cost-effective method for Cd(II) removal from aqueous solutions. Different

parameters such as time, initial metal concentration, pH and temperature were studied in batch experimental process. The concentration of cadmium was determined by using atomic absorption spectrophotometer. Fourier transform infrared (FTIR) Spectroscopy was used to obtain different spectra of litchi waste biomass before and after adsorption.

2. Materials and methods

2.1. Preparation of biomass

Litchi was collected from the local market. The fruit was de-skinned and the collected peels were washed with de-ionized water several times to remove the dust, fruit residues and other soluble material. Afterwards, the peels underwent the drying step in an electric oven till constant weight at temperature 60°C was achieved. The dried biomass was ground using agate pestle and mortar until fine powder of homogenous particle size was obtained. Then sieving of this material was performed using a set of different sieves. Different particle sizes (43, 104, 175 and 246 μm) were obtained. The sieved sorbent was stored in an airtight plastic container wrapped with aluminum foil for further experiments.

2.1.1. Preparation of cadmium stock solution and working standards

Stock solution of the Cd(II) was prepared by dissolving weighed amount of cadmium salt in de-ionized distilled water. The stock solution of cadmium (1,000 mg/L) was prepared using cadmium nitrate. The working Cd standard solutions were prepared by appropriate dilutions of the 1,000 mg/L stock solution of the cadmium. The working standards were in the range of 0.2–10 mg/L.

2.2. Batch biosorption studies

In all the sets of experiments, fix volume of 100 ml with different initial metal concentrations was used. Initial pH of the solution was adjusted to required pH using 0.1 M HCl or ammonia solution accordingly. Weighed amount of the biosorbent was added to each flask. Then this solution was shaken in orbital shaker with speed of 150 rpm at 20°C for about 30 min. After shaking, the samples were filtered using Whatman 40 filter paper. The filtrate was then analyzed by atomic absorption spectrophotometer in order to determine the cadmium concentration in each sample.

2.2.1. Effect of pH

The effect of pH on the cadmium ion adsorption by biomass of Litchi was studied. 100 mL of 25 mg/L solution was taken in 11 different flasks. The pH of each solution was adjusted from 1–11 using 0.1 N HCl and ammonia solution. After adjusting the pH, about 50 mL volume of each solution was taken to a conical flask, 0.5 g of litchi biomass was added to each flask and these samples were shaken using shaker for 30 min with agitation speed of 150 rpm. After filtration, the filtrate was run through atomic absorption spectrophotometer to determine the cadmium ion concentration.

2.2.2. Effect of dose

To check the effect of biosorbent dose on the cadmium ion adsorption by litchi biomass, biosorbent dose of 0.1, 0.2, 0.3, 0.4 and 0.5 g was added to five different solutions having 50 mL of 25 mg/L Cd(II) solution. Then these samples were shaken on orbital shaker for 30 min at 20°C with speed 150 rpm. These solutions were filtered, and the amount of cadmium adsorbed was thus determined.

2.2.3. Effect of particle size and metal ion concentration

To determine the effect of particle size, different particle sizes (43, 104, 175 and 246 μm) were used. About 0.5 g of biosorbent of each particle size was added to 50 mL of 25 mg/L Cd(II) solution having pH 5. These solutions were then shaken for 30 min at room temperature and then these solutions were filtered.

Similarly, 50 mL of 10, 15, 25, 50, 75 and 100 mg/L of Cd(II) solution having pH 5 and biosorbent dose 0.5 g was taken in each flask. The solutions were shaken using orbital shaker for 30 min at room temperature with 150 rpm speed. Solutions were filtered and the quantitative analysis of cadmium was performed.

2.3. Determination of uptake capacity and percentage removal

The Cd(II) uptake was calculated by the simple concentration difference method. Adsorption capacity “ q_e ” is amount of metal ion (mg) that is adsorbed per gram (dry weight) of biomass. The q_e is the adsorption capacity and its maximum value is known as q_{max} . Maximum metal sorption capacity (q_e) was determined by the decrease in metal concentration in the solution after addition of different amounts of biomass and equilibration using Eq. (1). The initial concentration, C_i (mg/L) and equilibrium metal concentration C_e (mg/L) respectively were determined. Eq. (2) was used to compute the percent metal uptake (adsorption efficiency)

$$q_e = \left(\frac{C_i - C_e}{M} \right) V \tag{1}$$

$$\% \text{ Adsorption} = \left(\frac{C_i - C_e}{C_i} \right) \times 100 \tag{2}$$

where V is the volume of the solution in L and M is the mass of the sorbent in g.

2.4. Kinetic and isothermal models

Kinetic models have been used to analyze the experimental data. This kinetics modeling was used to investigate the rate controlling steps like chemical reaction processes, mass transport and mechanism of biosorption.

The pseudo-first-order and pseudo-second-order kinetic models can be used, assuming in case if the measured concentrations are equal to powder surface concentrations. The kinetics study was performed for the data with fix metal ion concentration (25 mg/L) and biosorbent dose (0.5 g) but different contact time. The adsorption kinetics for biological material widely follows first order and pseudo-second-order kinetic model. The first order model expression is:

$$\log (q_e - q) = \log q_e - k_{1,ads} \frac{t}{2.303} \tag{3}$$

The pseudo-second-order expression is:

$$\frac{t}{q} = \frac{1}{k_{2,ads} q_e^2} + \frac{t}{q_i} \tag{4}$$

where q_e is the maximum adsorption capacity at equilibrium (mg/g) and q_t is metal ion concentration at time t (min), $k_{1,ads}$ is rate constant for first order kinetics model and $k_{2,ads}$ is the rate constant for pseudo-second-order kinetics model expressed as (mg/g min).

To understand the adsorption phenomenon, different types of adsorption isotherms are applied. These isotherms suggest the type of adsorption taking place on biosorbent when metal is adsorbed on it. In present study, the adsorption of Cd(II) by Litchi peel waste biomass through the Langmuir and Freundlich isotherms model were studied.

The general form of Langmuir adsorption expression is,

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{(q \cdot q_{max})} C_e \tag{5}$$

where q_e is the adsorption capacity at equilibrium and C_e is concentration at equilibrium. $1/q_{max}$ and b are constants which are obtained from regression equation where $1/q_{max}$ are intercept and b is slope.

Freundlich equation shows the empirical relation between the adsorbate and adsorbent molecules. The generalized and linear form of Freundlich expression is given below:

$$q_e = K C_e \frac{1}{n} \tag{6}$$

$$\log q_e = \log K \pm \frac{1}{n} \log C_e \tag{7}$$

where q_e is the adsorption capacity at equilibrium and C_e is concentration at equilibrium, $1/n$ and K are constants which are obtained from regression equation where $1/n$ is slope and K is intercept.

3. Results and discussion

Fig. 1 depicts the experimental design of Cd(II) ions removal from aqueous solutions by adsorbing them on Litchi peel which is most probably due to the presence of polyphenols. The adsorbent surface contains a number of hydroxyl and carboxylic functional groups which are able to interact with the Cd(II) ions (Fig. 2).

3.1. Optimization of experimental conditions

The biosorption of cadmium by waste biomass of Litchi peel was studied to investigate the influence of different parameters. pH is one of the essential parameters to be evaluated in adsorption studies because it is not only affects the adsorption sites of the biosorbent but also the metal ion strength to be exchanged or to react with the bisorbent.

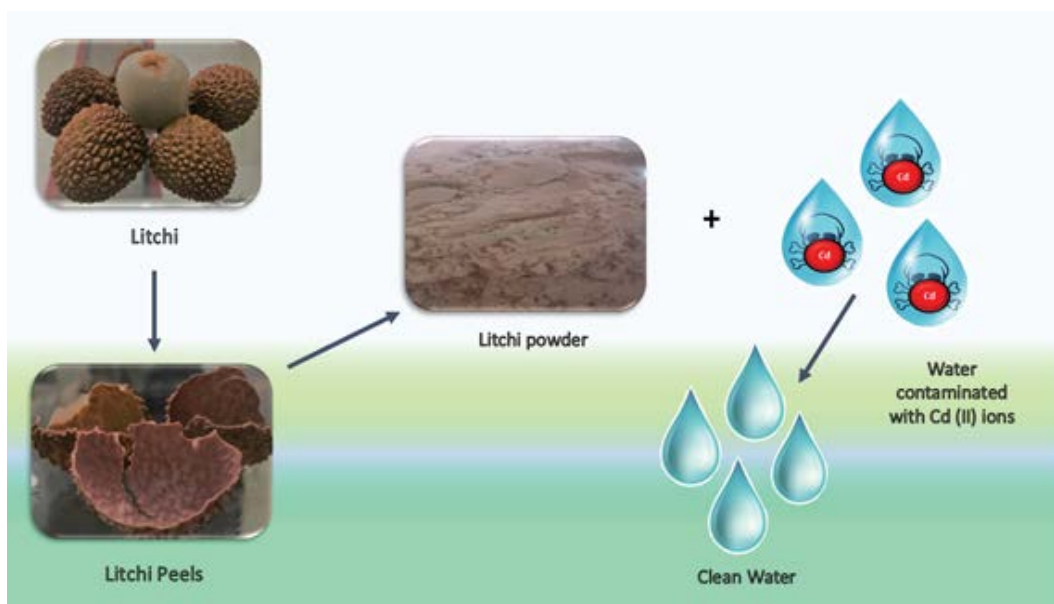


Fig. 1. Experimental scheme of the Cd(II) ions removal by applying Litchi peel.

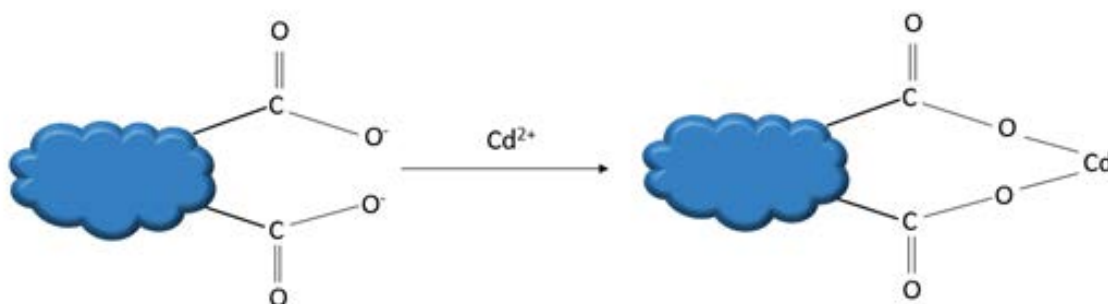


Fig. 2. Schematic representation of suggested adsorption mechanism of Cd(II) on Litchi peel.

Biosorption of Cd(II) by Litchi peel from aqueous solution was studied at pH ranges 3–8 (Fig. 3). At pH 3 which is highly acidic, Cd(II) ions uptake by Litchi peel was the lowest and the %removal was 44%. The %removal was increased to 71.87% at pH 4. At pH 5, the uptake was 76.64% which was maximum for this parameter. The biosorbent sites at very low pH are much protonated because of high concentration and mobility of the hydrogen ions (H^+). So the metal ions are in competition with hydrogen ions for adsorption sites of the adsorbent. The sites which have been protonated were incapable of binding metal ions due to electrostatic repulsion existing between sites having similar charge. So the metal uptake is low at lower pH. When the solution pH is high, the concentration of H^+ ions is low and as a result more adsorption sites are available for metal ion to be adsorbed. An additional metal uptake was expected at further higher pH but to the contrary, a slight drop in % removal was observed at pH 6 which then turned constant afterwards. This behavior

can be due to the saturation of active sites on the surface of adsorbent.

The metal uptake depends upon to some extent on the dose of biosorbent used. This trend may vary for different metals. As assumed that the metal uptake decreases but %removal increases as the biosorbent dose increases and after some time it become constant. In present study, uptake of Cd(II) by Litchi peel followed this trend. The biosorbent dose was studied in the ranges of 0.1–0.5 g (0.1, 0.2, 0.3, 0.4 and 0.5 g) (Fig. 4). The %removal increases but uptake decreases as the biosorbent dose increases. At low biosorbent dose, the metal ion availability is greater so uptake is greater but %removal is low as dose is low. The observed behavior can be due to the reason that the metal ion competes for adsorbent active sites and uptake is high. But as biosorbent dose increases, the uptake decreases which can be probably due to electrostatic interactions among the cells of the biosorbent, preventing binding sites from metal occupation.

The biosorption of Cd(II) on Litchi peel waste biomass was also studied for four different particle sizes, that is, 43, 104, 175 and 246 μm (Fig. 5). The Cd(II) uptake decreased as the particle size was increased. The metal ion removal was about 92.66% when the particle size was 43 μm . The removal is 80.22%, 65.78% and 60.66% for particle size 104, 175 and 246 μm , respectively which is due to the fact that the smaller particle size has more surface area and ultimately more adsorption sites. So the more number of metal ions can be adsorbed on small size particles. The mass of the biosorbent was same in this experiment showing that adsorption is independent of the mass but depends on particle size.

For economical wastewater treatment, the effect of contact time is very important parameter to be determined. Fig. 6 shows the effect of contact time on biosorption of Cd(II) by Litchi peel, while all other parameters (pH, particle size, dose of biosorbent and initial concentration of metal) were kept constant and the experiment was carried out by varying the time intervals. The uptake of Cd(II) increased by increasing contact time of biosorbent with metal ions. The increase was

very sharp in first 30 min. Below these time limits, the uptake came to be constant approximately. In a system, the active adsorption sites are fixing and initially the ions bind the active adsorption site to form a monolayer. When the process commences, the uptake of metal by the sorbent surface is rapid but slows down as the active sites become occupied by the metal ions in the solution.

The initial metal ion concentration also significantly affects adsorption capacity. The adsorption study of Cd(II) by Litchi peel biomass with different initial concentration (10, 25, 50, 75 and 100) was performed. The other factor such as pH, biosorbent dose, shaking time and speed was kept constant.

The Cd(II) ions uptake at equilibrium (q_e) increased from 1.84 to 14.53 mg/g when metal ion concentration was increased from 10 to 100 mg/L. It is due to the reason that the concentration gradient increases as concentration increases. Fig. 7 shows the effect of different metal ion concentrations on the Cd(II) uptake by Litchi peel waste biomass.

3.2. Kinetic and isothermal models

The comparison of the calculated parameters based on equilibrium adsorption determine the type of kinetic

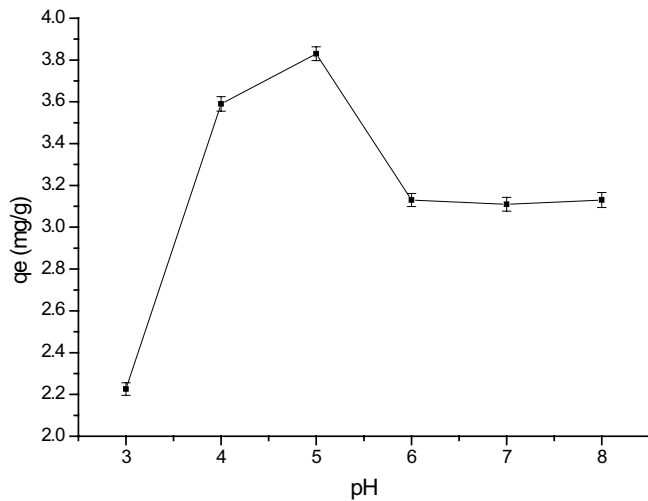


Fig. 3. Effect of pH on uptake of Cd(II) by Litchi peel.

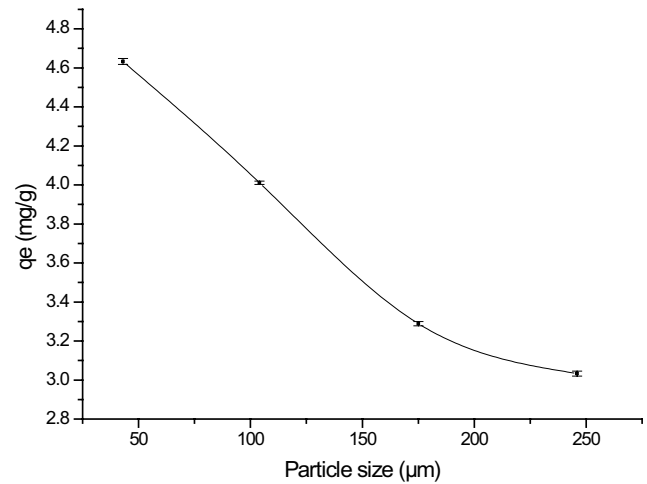


Fig. 5. Effect of particle size on uptake of cadmium by Litchi peel.

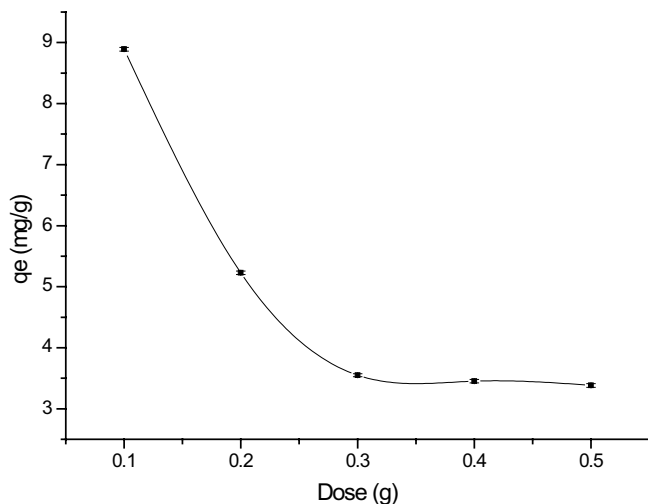


Fig. 4. Effect of biosorbent dose on uptake of Cd(II) by Litchi peel.

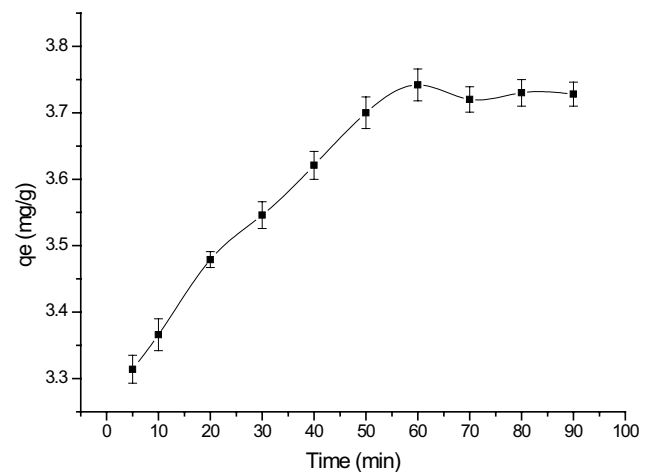


Fig. 6. Effect of contact time on uptake of Cd(II) by Litchi peel.

model that fits experimental data. From the values of adsorption data, it can be noticed that adsorption of Cd(II) ions by Litchi peel waste biomass fitted well for pseudo-second-order kinetic model with an R^2 value of 0.999

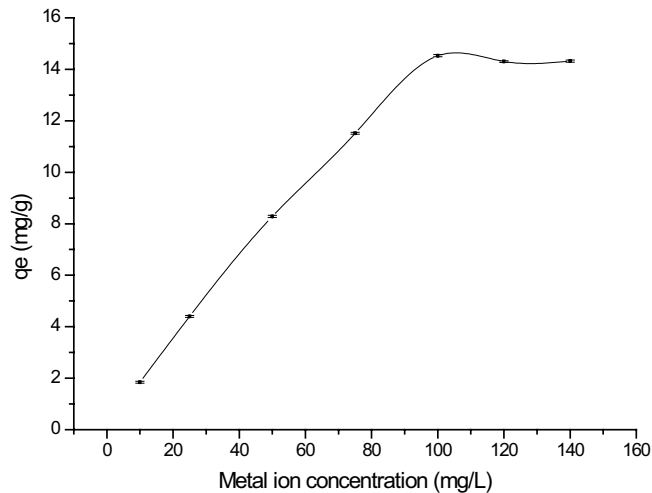


Fig. 7. Effect of initial metal ion concentration on uptake of Cd(II) by Litchi peel.

which was higher than pseudo-first-order kinetic model, that is, 0.990 (Table 1). The same result has been reported by biosorption study of Cd(II) using *Polypogon monspeliensis* waste biomass [36].

The Langmuir and Freundlich isotherm models were evaluated to determine the adsorption behavior (Table 2). The Freundlich isotherm model suited well to the adsorption data of Cd(II) by Litchi peel and a relatively high value of R^2 was achieved. Based on Freundlich isotherm, the adsorption capacity of 15.27 mg/g was observed which is quite close to experimental value, that is, 14.53 mg/g.

Biomasses like *Polypogon monspeliensis* have also been investigated for the adsorption of Cd(II) [32]. The biosorbent presented a q_{max} equal to 9.87 mg/g. Similarly, banana peels showed a q_{max} of 9.87 mg/g [37]. Hence, Litchi peel biomass can be considered a preferable biosorbent with an adsorption capacity of approximately 15 mg/g.

3.3. Spectral studies

FTIR analysis was carried out to study the functional groups present in Litchi peel that can serve as active sites to bind the metal ions. These functional groups are responsible to carry out the adsorption process. The FTIR spectra of Litchi fruit peel was taken before and after adsorption of Cd(II). These two spectra are shown in Figs. 8 and 9.

Table 1

Comparison between Lagergren pseudo-first-order and pseudo-second-order model parameters

Metal	Pseudo-first-order kinetic model			Experimental value	Pseudo-second-order kinetic model		
	q_e (mg/g)	$k_{1,ads}$	R^2		q_e (mg/g)	q_e (mg/g)	$k_{2,ads}$ (g/mg min)
Cd(II)	0.529	0.34	0.990	3.742	3.676	0.21	0.999

Table 2

Comparison between Langmuir and Freundlich isotherm models parameters

Metal	Langmuir isotherm parameters			Experimental value	Freunlich isotherm parameters			
	q_{max} (mg/g)	k_l (L/mg)	R^2		q_{max} (mg/g)	q_{max}	K (mg/g)	$1/n$
Cd(II)	18.87	0.1072	0.976	14.530	15.27	2.23	0.580	0.996

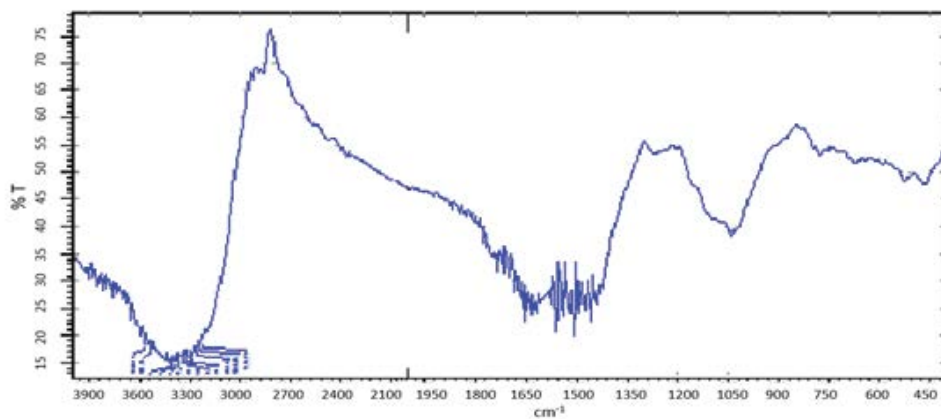


Fig. 8. FTIR spectra of Litchi peel before Cd(II) adsorption.

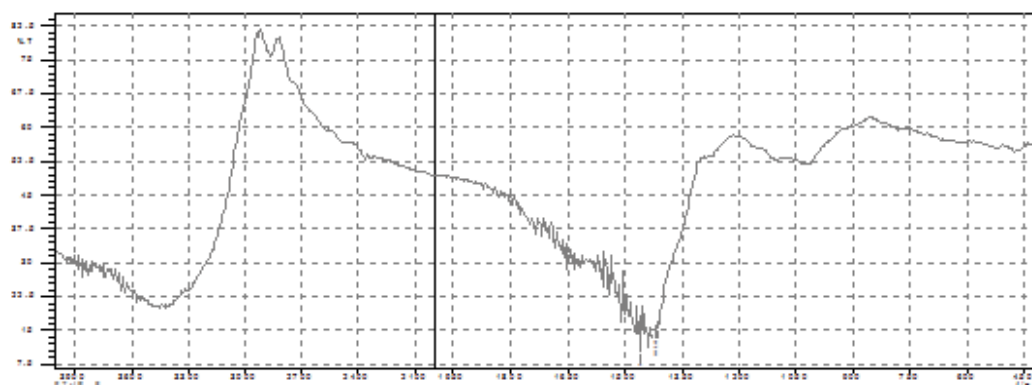


Fig. 9. FTIR spectra of Litchi peel after Cd(II) adsorption.

The broad and intense band from $3,200\text{--}3,500\text{ cm}^{-1}$ suggests the presence of hydroxyl (--OH) groups which are due to the alcohols and phenols found on the peel. The bands at $2,870$ and $2,960\text{ cm}^{-1}$ correspond to the symmetric and asymmetric --CH stretch of alkanes. The weaker --CH stretch occurred at $2,850\text{--}3,000\text{ cm}^{-1}$. A series of bands was observed from $1,400\text{--}1,650\text{ cm}^{-1}$ indicating the presence of the groups such as C=C , --COO and --NH . The band at 1050 cm^{-1} can be attributed to C--OH stretching vibrations. Fig. 9 presents a significant change in the position and complexity of the bands. The intensity of the bands lessened in the region of $1,200\text{--}900\text{ cm}^{-1}$. Moreover, a variation in the shape of the bands between $1,650\text{--}1,350\text{ cm}^{-1}$ was also noticed. These differences in the spectrum (Fig. 9) corroborate the adsorption of Cd(II) ions by the litchi peel.

4. Conclusion

Litchi waste biomass was found to be effective for Cd(II) removal from aqueous solutions. Cd(II) uptake by Litchi peel waste biomass was considerably affected by operational variables. The Freundlich isotherm model fitted to Cd(II) biosorption by litchi peel. Second order kinetics was found to be more suitable as compared to the first order due to inclusion of both adsorbent and adsorbate species in biosorption of Cd(II) ions onto litchi waste biomass. Litchi biomass can thus be considered as a non-hazardous and economical bio waste with a potential to be utilized in water treatment processes.

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