Pak. j. sci. ind. res. Ser. A: phys. sci. 2011 **54** (3) 132-139

Biosorptive Removal of Cadmium from Aqueous Solutions by *Pleurotus ferulae:* Equilibrium, Kinetic and Thermodynamic Studies

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(received September 6, 2010; revised January 19, 2011; accepted April 12, 2011)

Abstract. Equilibrium, kinetics and thermodynamic parameters were evaluated to establish the potential usefulness of the *Pleurotus ferulae* biomass for biosorption of cadmium from aqueous solutions. Maximum biosorption was observed at initial pH of 4.5, temperature of 30 °C and at the initial cadmium concentration of 100 mg/L. Pseudo- second order rate expression well fitted the experimental data for cadmium when compared to pseudo-first order kinetic model. Equilibrium analysis using Langmuir and Freundlich models showed that the biosorption process is Langmuir model. The process was exothermic and ΔG^{o} was negative showing spontaneity of the process within the studied temperature range. The possible functional groups on the dried *Pleurotus ferulae* biomass, responsible for the sorption of Cd (II), are: – OH, – NH, – COO^- and – C– O.

Keywords: biomass, biosorption, cadmium, *Pleurotus ferulae*

Introduction

Heavy metal pollution in wastewaters is an extremely important environmental problem. Discharge of heavy metals from various industrial operations such as mining, ore processing, smelting and metal plating can easily cause metal pollution and hazardous effects on humans, animals as well as environmental imbalance. Due to metal accumulation through food chain and persistence, it is necessary to remove such chemical agents from wastewater before discharging it to the environment. Cadmium is usually associated with some principal metal ores widely used in daily life such as zinc, copper, mercury, iron, lead etc. Cadmium is also a dangerous pollutant originating from metal plating, metallurgical alloying, mining, ceramics and other industrial operations. Chronic exposure to elevated levels of cadmium is known to cause renal dysfunction (Fanconi syndrome), bone degeneration (‘Itai–itai’ syndrome), liver damage and blood damage (ATSDR, 1993). The US Department of Health and Human Services has determined that cadmium and cadmium compounds may be carcinogens. Cadmium has been found in at least 388 of 1300 items of national priority list identified by the Environmental Protection Agency (ATSDR, 1993).

Though much efforts have been significantly put into curtail the amount of cadmium in the environment, these have not really yielded the results desired by the most world related health and environmental standard regulatory bodies possibly due to inefficient and uneconomical removal technology used in the past two to three decades (Roskill Information Services, 1995). Therefore, the release of Cd into the environment, and resultant risk of exposure at various trophic levels still remain substantial. Some of the bases of conventional technologies for removing metallic ions from wastewater are solvent extraction, ion exchange, chemical precipitation, adsorption and reverse osmosis techniques (Kapoor and Viraraghavan, 1995). Chemical precipitation, especially as metal hydroxide or sulphide, is widely practiced, being simple employing inexpensive chemicals. However, it is not effective to reduce toxic metal concentrations to the level of water quality standards; also the generation of voluminous toxic waste sludge is a major problem encountered. Therefore, during the last few decades, research yielded adsorption to be one of the attractive techniques for removing noxious substances and for water purification; it is fast becoming an alternative to conventional precipitation and other techniques, especially for wastewaters that contain low concentrations of metals (Sahu *et al.,* 2010; Jyotikusum *et al.,* 2009; Wahid, 2009; Sing *et al.,* 2008; Sheng *et al.,* 2004).

Activated carbon is the most widely used adsorbent in the wastewater treatment. Owing to high cost and

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ineffectiveness of activated carbon in the removal of aqueous soluble matter particularly heavy metals (Faust and Aly, 1987), research on low-cost and high affinity-bearing metal adsorbents such as biomass wastes and various other biological materials has been receiving much attention. Biomass has been advocated for removal of heavy metals for the following reasons: multiplicity of functional groups that have affinity for metals as ligands, lightness of the particles and so low bulk density, ability to be pulverized to very small particles and hence having large surface area per unit mass, low cost and easy availability, among other properties.

Biomass consists of polysaccharides and proteins carrying many functional groups, such as carboxylate, hydroxyl, sulphonate, phosphate and amino groups, which can bind metal ions. Different types of biomass have been investigated for biosorption of cadmium and other heavy metal ions. Those include bacteria (Bang *et al.*, 2000), fungi (Zafar *et al.*, 2007), wood bark (Al-Asheh and Duvnjak, 1998), red algae (Sarý and Tuzen, 2008), agricultural by-products (Krishnani *et al.*, 2008; Schiewer and Patil, 2008), marine algae (Freitas *et al.*, 2008; Lodeiro *et al.*, 2006) and aquatic mosses (Sarý and Tuzen, 2008; Herrero *et al.*, 2006). Literature review showed that little work had been reported on sorption potential of some notable basidiomycete, a fungi class mostly used as food supplement in Africa and some parts of Asia.

The characteristics of the biosorption behaviour are generally analyzed by means of both biosorption kinetics and equilibrium isotherms (Herrero *et al.*, 2006; Lodeiro *et al.*, 2006). The biosorption isotherm is an important tool for understanding the biosorption mechanism for theoretical evaluation as well. These basic data are useful in the design of suitable reactors for the adsorption process technology. The objective of this research was to investigate the removal of cadmium metal ions from simulated aqueous solutions by the fungus *Pleurotus ferulae*. It is widely available, growing on dead decaying mango trees and some other dead trees. In this study, it was chosen as a biosorbent material due to the lack of information on its biosorption capabilities and such basic data required for reactor design. The biosorption capacity was determined using various kinetic models; the well-known equations of Langmuir and Freundlich were used for the equilibrium analysis while the kinetic was analysed by using Lagergren expressions. The effect of biosorbent dosage, initial metal concentration, contact time, temperature and pH was examined. The thermodynamic parameters were also deduced from the adsorption measurements to further strengthen the useful sorbent potentials.

Materials and Methods

Reagents and equipments. The reagents used in this study are cadmium nitrate, nitric acid and sodium hydroxide procured from BDH Chemicals, England; they are all analytical grade reagents. Atomic absorption spectrophotometer (AAS Alpha 4 model) was used for analysis of metal ions and Fourier Transformation Infra-Red (FTIR Buck 500M) for determination of functional groups of the adsorbent.

Preparation of *Pleurotus ferulae* biosorbent and Cd (II) ion solution. *P. ferulae* was collected from an old cocoa plantation in Apatapiti area, off Federal University of Technology, Akure Road, Ondo State, Nigeria. The collected material was identified in the Department of Microbiology of the Federal University of Technology, Akure, Nigeria. The material was washed with deionised water to remove dirt particles. The washed material was first air dried and later oven dried at 70 °C for 48 h. Dry biomass was crushed into powder and sieved to 100 µm particle size. The stock solution containing 1000 mg/L of Cd (II) was prepared by dissolving 2.103 g of cadmium nitrate in 10 mL of concentrated nitric acid and then diluted up to 1000 mL in a volumetric flask with distilled water. The working solutions were prepared by diluting cadmium nitrate stock solution in accurate proportions to the required concentrations.

Batch biosorption experimental process. The experiments were conducted in 600 cm^3 Erlenmeyer flasks containing appropriate amount of dried *P. ferulae* and 50 mL of Cd (II) ion synthetic solutions having different concentrations and pH. At the end of the predetermined contact period, the mixtures were filtered and the filtrates were analysed for Cd (II) using atomic absorption spectroscopy and the equilibrium concentration were calculated (equation 1) while the percentage of the removed cadmium (R%) was obtained as shown in equation 2:

$$q = \frac{(C_0 - C_e)V}{M} \qquad (1)$$

$$R\% = \frac{C_e}{C_0} \times 100 \qquad (2)$$

where, M is the biosorbent mass (g), q the adsorbed metal ions (mg per gram of biosorbent at equilibrium), C_0 the initial metal concentration (mg/L), C_e the metal concentration at equilibrium (mg/L) and V is volume of the the working solution.

Spectrometric determination of the functional groups on dried *Pleurotus ferulae*. Infra-red spectroscopy was used for the determination of functional groups on the dried *P. ferulae*. The spectra were collected by using a Buck 500M FT-IR spectrometer within the range 400–4000 cm^{-1} using a KBr window. The background obtained from the scan of pure KBr was automatically subtracted from the sample spectra.

Results and Discussion

Effect of pH on biosorption of Cd (II) using *P. ferulae*. The effects of pH were investigated in the range of 1 to 7.5 at a constant contact time of 20 min, biosorbent dosage of 2.5 g, initial metal concentration of 100 mg/L. The percentage of the removed cadmium against pH of the solution, plotted in Fig. 1, showing that uptake of Cd (II) increases with pH.

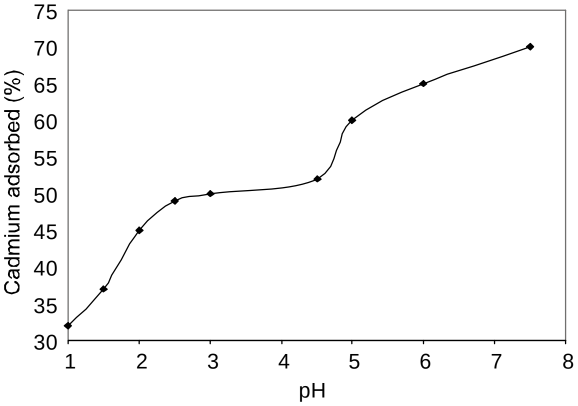

Fig. 1. Variation of pH with cadmium removed % by *P. ferulae*.

However, an inflection pH point was observed; the first region was between pH of 1 to 5 in which there was increase in uptake considerably between pH of 1 and 3 and, thereafter, almost remained constant till 4.5. Then another increase in the uptake was observed above pH of 5; in fact during the studies at this pH range (5 to 7.5), there was precipitate in the solution which most probably was due to formation of insoluble $Cd(OH)_2$. At pH between 1 and 4.5, the solution was obviously clear and no precipitate was observed. It could be deduced that true sorption took place between pH 1 and 4.5 whereas at pH between 5 and 7.5, there was precipitation in which removal of the metal is not really by adsorption alone. The removal of Cd (II) by *P. ferulae* as affected by the initial pH of the metal solution could probably be due to ionic attraction between the possible charges on the biomass and the metal ions, particularly at much lower pH. Earlier reports on heavy metal biosorption have shown that pH is one of the important parameters affecting most biosorption processes (Sarý and Tuzen, 2008; Bulut and Baysal, 2006; Sittig, 1973). The pH of medium affects the solubility of metals and the ionization state of the functional groups like carboxylate and amino groups of the biomass. The carboxylate groups carry negative charges that allow the biomass components to be potent scavengers of cations.

Effect of biosorbent dosage on biosorption of cadmium using *P. ferulae*. The effect of biosorbent dosage was monitored to attain the maximum uptake of Cd (II). The dosage was varied from 0.5 to 4 g at the predetermined initial Cd (II) concentration of 100 mg/L and 30 °C in 50 mL of the solution. Each dosage was monitored for a period of 1 h and 1 mL of the sample was taken at 10 min intervals, diluted to 25 mL with distilled water and analysed for cadmium. The percentage of cadmium removed was found to be increasing with increase in biosorbent dosage (Fig. 2). At low dosage, between 0.5 -1.5 g, the maximum removal at saturation was 20% for 1.5 g, which increased to 52% for 4 g after 20 min of contact. The removal of Cd (II) with sorbent between 2.5-4 g does not change appreciably thus 2.5 g of biosorbent dosage was taken as optimum value for the subsequent experiments. Various reasons have been suggested to explain the increased biosorption of metal with increasing biosorbent dosage including availability of more surfaces, electrostatic interactions and interference between binding sites (Sittig, 1973).

Effect of contact time on biosorption of cadmium using *P. ferulae*. The effect of contact time from 0 to 60 min on the removal of Cd (II) was investigated at a

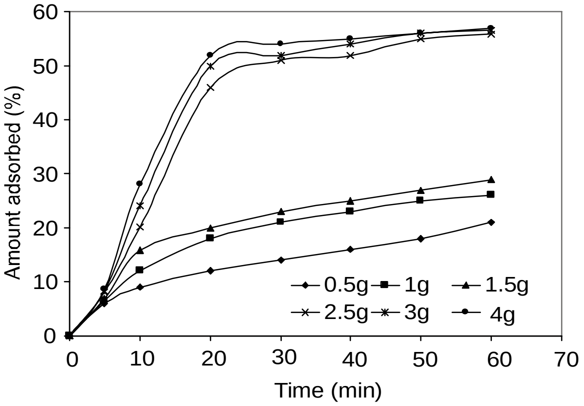

Fig. 2. Effect of adsorbent biomass on adsorption of Cadmium.

fixed predetermined initial Cd (II) concentration of 100 mg/L at 30 °C in 50 mL solution. The metal biosorption increased rapidly during the first 20 min and attained equilibrium as shown in Fig. 3.

After attaining the equilibrium, the amount of biosorbed metal ions did not appreciably changed with time. The removal of Cadmium increased to about 50% during 20 min and was less than 60% in 60 min of continuous contact. This might be due to saturation of the surface of the adsorbent in 20 min.

Effect of initial metal concentration on removal of Cd (II) using *P. ferulae*. The effect of initial metal concentration on the removal of Cd (II) was conducted using *P. ferulae* at different Cd (II) concentrations of 20 to 300 mg/L for contact time of 20 min, 2.5 g biosorbent material at 30 °C. It was observed that the amount of removed cadmium initially increased linearly with the Cd (II) concentration and then finally attained saturation after 20 min. (Fig. 4).

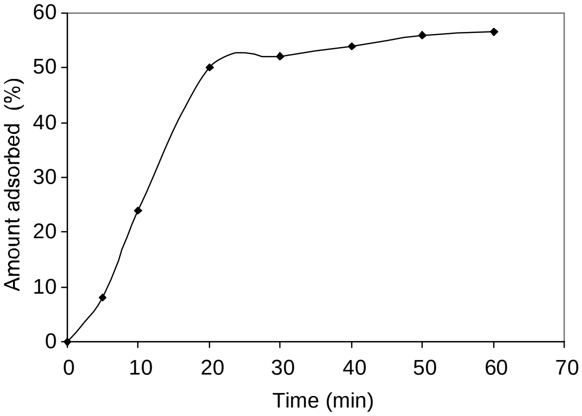

Fig. 3. Effect of contact time on removal of cadmium using *P. ferulae*.

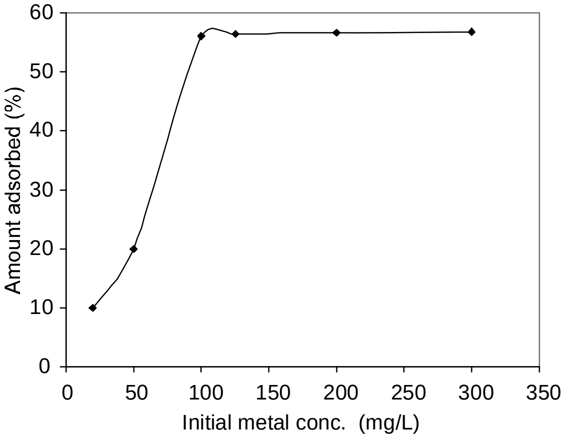

Fig. 4. Effect of initial metal concentration on removal of cadmium using. *P. ferulae*.

The saturation concentration was observed at 100 mg/L of the metal solution from which only 56% was removed. There are many factors which can contribute to the sorbate concentration effect. The first and important one is that adsorption sites remain unsaturated during the adsorption process. The second cause can be the aggregation/ agglomeration of sorbent particles at higher concentrations. Such aggregation would lead to a decrease in the total surface area of the sorbent particles available for metal adsorption and an increase in the diffusional path length. The particle interaction brought about at high sorbent concentrations may also desorb some of the metal ions, which are loosely and reversibly bound to the sorbent surface (Bulut and Baysal, 2006).

Effect of temperature and thermodynamic consideration of biosorption of Cd (II) on *P. ferulae*. The effect of temperature on the equilibrium sorption capacity of dried *P. ferulae* biomass was investigated in the temperature range of 30-50 °C. It was observed that biosorption capacity of the biomass decreased with increasing temperature. The reduction of the metal removal in this temperature range means that the process of cadmium sorption by *P. ferulae* is exothermic as it is the case for the majority of gas adsorption processes. Similar results have been reported for the sorption of lead by *Cephalosporium aphidicola* from aqueous solution at temperature 20-40 °C (Tunali *et al.*, 2006) and biosorption of lead and copper with dried activated sludge (Katsumata *et al.*, 2003).

The effect of temperature in the range of 30-50 °C was used to determine the effect of some important thermodynamic parameters on the biosorption of Cd (II). The values of equilibrium constants (K_d) at 30, 40 and 50 °C were calculated from the relation in Equation 3:

$$k_d = \frac{C_s}{C_e} \qquad (3)$$

where, C_e and C_s are the equilibrium concentrations (mg/L) of Cd (II) on the biosorbent and in solution, respectively.

$$\Delta G^o = - RT \ln k_d \qquad (4)$$

where, T is the absolute temperature, R gas constant and ΔG^o is the standard free energy change. The values of enthalpy change (ΔH^o) and entropy change (ΔS^o) were calculated from the following relation in equation 5:

$$\ln k_d = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \qquad (5)$$

where, ΔS^o and ΔH^o were calculated from the slope and intercept of Van't Hoff plot of ln k_d versus $1/T$ (Fig. 5).

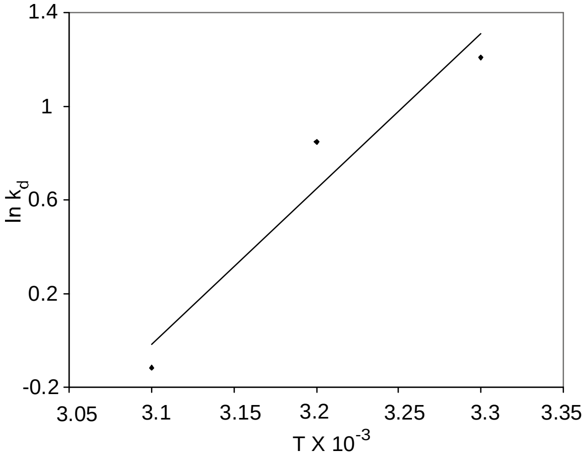

Fig. 5. Plot of ln k_d against 1/T.

Table 1 shows the values of k_d, ΔH^o, ΔS^o and ΔG^o obtained from eq. 4. Thermodynamic parameters are quite useful for evaluation of feasibility of any process.

Table 1. Thermodynamic parameters for biosorption of Cd (II) on *P. ferulae*

Temperature (K)	k_d	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (kJ/mol)
303	3.35	-3.046	-0.0551	0.1711
318	2.33	-2.236	-0.0551	0.0171
323	0.89	0.313	-0.0551	0.1711

The negative values of ΔG^oshow that the process is spontaneous and the spontaneity decreases with increase in temperature within the studied range. The positive value of ΔS^o suggests an increase in randomness at the solid–liquid interface during biosorption. The negative values of ΔH^oconfirm that the process under study is exothermic while the values of k_d indicate favoured process at 303 K but decreasing with increase in temperature. These parameters show that the process is favourable within the temperature range but could best be performed at lower temperatures.

Biosorption kinetic of Cd (II) on *P. ferulae*. The biosorption mechanism and potential rate controlling steps have been investigated, using the Pseudo-first and pseudo-second order kinetic models. The Pseudo-first order (Lodeiro *et al.*, 2006) rate expression of Lagergren is

$$\frac{dq}{dt} = k_{1,ad}\,(q_e - q), \qquad (6)$$

where, q is the amount of metal adsorbed on the biosorbent at time t and k_1, $_{\text{ad}}$ /min is the rate constant for pseudo-first order biosorption. The integral form of equation 6 is

$$\ln\,(q_e - q) = \mathrm{In}\; q_e - k_{1,ad}\,t \qquad (7)$$

A linear fit of ln (q_e - q) versus t shows the applicability of this kinetic model. Expression for the pseudo-second order (Herrero *et al.*, 2006) kinetic model is

$$\frac{dq}{dt} = k_{2,ad}\,(q_e - q)^2 \qquad (8)$$

where, k_2, $_{\text{ad}}$ (g/mg/min) is the rate constant of the pseudo-second order biosorption. The integrated linear form of equation 8 is

$$\frac{t}{q} = \frac{1}{k_{2,ad}\,q_e^2} + \frac{1}{q_e}\,t \qquad (9)$$

If the experimental data fits the plot of t/q versus t as linear relationship, the pseudo-second order kinetic model is valid.

From the slopes and intercepts of plots of log (q_e - q) against time t, the pseudo-first-order rate constants (k_1, ad) and theoretical q_{eq}, $_{\text{cal}}$ values were determined. The pseudo-second order biosorption rate constant (k_2, $_{ad}$) and theoretical q_e values were determined from the slope and intercept of the plots of t/q against t. The values of the parameters (k_2, $_{\text{ad}}$), q_e calculated and q_e experimental together with correlation coefficients are presented in Table 2.

The linearized form of the pseudo-first and pseudo-second order kinetic model at different initial metal concentrations for a period of 60 min was analysed. The correlation coefficients of the pseudo-first-order kinetic model obtained for metal using *P. ferulae* were found to be less as compared to the pseudo-second order kinetic model studied in the present investigation (Table 2.).

Biosorption Equilibrium of Cd (II) on *P. ferulae*.The biosorption of metal can be quantitatively evaluated by experimental equilibrium isotherms. The graphical expression of isotherm is a plot of the metal uptake by per unit weight of biosorbent against the residual metal ion concentration in the biosorption medium. There are two widely accepted and easily linearized adsorption isotherm models used in the literature, namely Freundlich and Langmuir models.

The Freundlich model based on the relationship between the metal uptake capacity "q" (mg/g) of biomass and the equilibrium metal ion concentration "C_e" (mg/L). The general Freundlich equation is as follows:

$$q = k_f\,C_e^{1/n} \qquad (10)$$

Table 2. The Pseudo-first and second order rate constants of Cd(II) using *P. ferulae*

C_o (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo-first order			Pseudo-second order		
		$q_{e,cal}$ (mg/g)	k_1 (1/min)	r^2	$q_{e,cal}$ (mg/g)	k_2(g/mg/min)	r^2
20	4.65	1.79	0.068	0.971	2.72	0.166	0.992
50	6.31	2.17	0.082	0.990	5.89	0.116	0.998
100	7.51	2.05	0.069	0.963	7.54	0.118	1.000
125	7.80	2.29	0.107	0.972	7.07	0.119	0.999
200	7.95	2.07	0.084	0.935	7.39	0.116	0.999
300	8.10	2.03	0.084	0.934	8.03	0.115	0.999

r^2, calculated and experimental; q_{eq},values for the biosorption of Cd(II).

and linearized form of the model is

$$\ln q_e = \ln k_f + \frac{1}{n}\ln C_e \qquad (11)$$

where intercept, $\ln k_f$, is a measure of adsorbent capacity, and the slope, $1/n$, is the intensity of adsorption.

The general Langmuir equation is commonly presented as:

$$q_e = \frac{Q_o bC_e}{1 + bC_e} \qquad (12)$$

and the equation may be linearized as follows:

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \qquad (13)$$

where, q_e is the amount of metal ion removed (mg/g), C_e the equilibrium concentration (mg/L), Q_o and b are the Langmuir constants related to adsorption capacity and affinity, respectively. Experimental data fitted both the Freundlich and the Langmuir isotherms. But the correlation coefficients of Langmuir adsorption isotherm showed that the Langmuir isotherm yielded the best fit to experimental data. The necessary equilibrium constants were calculated from the corresponding plots (figures not shown) for the biosorption of Cd (II) on the biosorbent; the results are presented in Table 3. Hence, the biosorption process in this study may be interpreted as monolayer adsorption. The values of *b* indicate the affinity of biosorbent to the investigated metals and imply strong binding of metal ions.

Fourier transform infrared analysis (FTIR). The FTIR spectra of dried *P. ferulae* biomass (Fig. 6) in the range of 400-4000/ cm were used to obtain information on the nature of possible functional groups that could be responsible for biomass-metal ion interactions. The broad stretching absorption band at 3441 cm^{-1} is assigned to both –NH and bonded – OH groups.

The band observed at 2926 cm^{-1} is an indication of symmetric/asymmetric stretching vibration of the $–CH_3$ and $–CH_2$ groups and their bending vibrations are 1458 and 1467 cm^{-1} for dried biomass. Carbonyl stretching band of un-ionized carboxylates was observed at 1792 cm^{-1}. The band at 1947 cm^{-1} is a consequence of –C–O stretching vibration conjugated to a –NH deformation and is indicative of amide-I band. The other band referred to amide group, namely amide-II, is present at 1541 cm^{-1} assigned to -NH deformation conjugated to –CN=N deformation for dried biomass. The 1077 cm^{-1} band was due to C–O stretching of carboxyl groups and the bending vibration band of hydroxyl groups. The functional

Table 3. Langmuir and Freundlich constants from Cd (II) isotherms

C_o (mg/L)	Langmuir			Freundlich		
	q_{max} (mg/g)	b (g/mg/min)	r^2	k_f	1/n	r^2
20	2.79	5.68	0.992	0.72	0.166	0.912
50	5.17	5.82	0.990	0.89	0.116	0.928
100	7.54	5.69	1.000	1.54	0.118	0.870
125	7.09	5.70	0.999	1.07	0.119	0.919
200	7.37	5.84	0.999	1.39	0.116	0.909
300	8.03	5.84	0.999	1.03	0.115	0.919

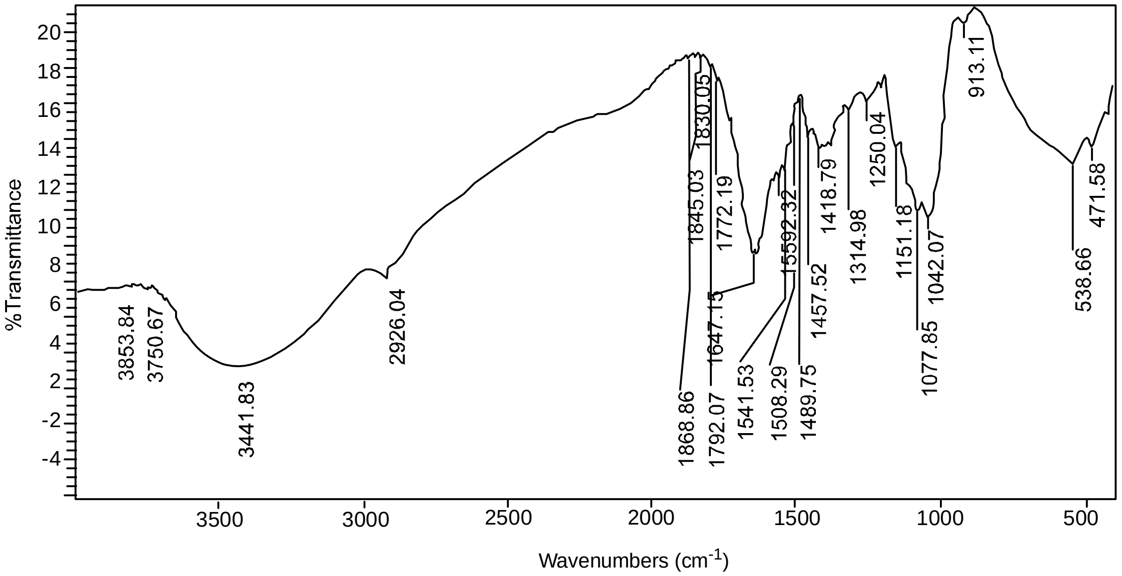

Fig. 6. FTIR of dried biomass.

groups which can adsorb Cd (II) ions are of the type–OH, –NH, –COO$^-$ and –C–O present on the biomass (Xuejiang *et al.,* 2006).

Conclusion

In this study, a dried powdered *Pleurotus ferulae* biomass was used for investigating the effect of contact time, biosorbent dosage, initial metal concentration, initial pH and temperature on the biosorption of Cd (II) from simulated solution. From these variables, kinetic, equilibrium and thermodynamic parameters were determined and conclusions were drawn. There was rapid biosorption of Cd (II) within the first 20 min with biosorption capacity of 6.5 mg/g and thereafter became very slow. There was increase in sorption of Cd^{2+} with increase in biosorbent dosage from 0.5 to 2.5 g and thereafter did not show appreciable sorption. Biosorption of the metal increased with increase in pH up to 3.0 and thereafter reached a plateau. Though removal of Cd (II) was still observed up to pH 7.5, but this was strongly assumed to be due to precipitation. Rapid biosorption was observed upto initial Cd (II) concentration of 100 mg/L but thereafter did not show appreciable sorption. Increase in temperature lead to decrease in sorption of Cd (II) on to *P. ferulae* biomass indicating an exothermic process. Kinetic analysis using rate expressions of Lagergren shows that the process is pseudo-second order reaction model. Initial biosorption rate increased with the increase in initial Cd (II) concentration and decreased with increase in temperature. Equilibrium analysis using Freundlich and Langmuir models showed that the biosorption process follows Langmuir model. Thermodynamic consideration shows that equilibrium constant (K_d) decreases with temperature; decrease in Gibb's free energy (ΔG^o) with increase in temperature shows that the biosorption of Cd (II) by *P. ferulae* is better performed at 30 °C. The possible functional groups on the dried *P. ferulae* responsible for the sorption of Cd (II) are: – OH, –NH, – COO$^-$ and –C–O.

Acknowledgement

The contributions of following people are highly acknowledged: Prof. K.A. Onifade and Dr. (Mrs.) B.J. Akinyele, both of Microbiology Department of the Federal University of Technology, Akure, for identification of the biomass. Appreciation is also due to Miss O.G. Fasuyi for her partial contribution in the experiment.

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