

BIOSORPTION STUDIES OF CADMIUM (II) IONS FROM AQUEOUS SOLUTION BY *MUSA PARADISIACA* STALK

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ABSTRACT

This study is an investigation of the sorption of Cd(II) ions from aqueous solution by *Musa paradisiaca* stalk through a series of batch experiments conducted under varied pH, contact time, and initial metal ion concentration. The optimum pH is found to be 7.0, at which over 85% Cd(II) adsorption efficiency was achieved. The time - dependent study shows that the sorption is rapid within the first 10 minutes and when tested on the pseudo first-order and pseudo second-order kinetic models, the latter appears to be more appropriate having a linear coefficient of correlation (R^2) value of 0.999 and a rate constant, k_2 of $0.123 \text{ g mg}^{-1} \text{ min}^{-1}$. The Langmuir and Freundlich isotherm models are both suitable in describing the adsorbate-adsorbent relationship, however the Langmuir model gives a better fit as indicated by the R^2 value of 0.9823 with q_{max} equal to 980.40 mg g^{-1} . Fourier Transform Infra-Red spectroscopy study shows that -OH, -C=O and -C-O groups majorly make up the functionality of the biomass. The results in this study indicates that the biomass could be used for the removal of Cd(II) ions from aqueous systems.

Keywords: *Musa paradisiaca*, cadmium(II), adsorption isotherms, kinetics

INTRODUCTION

One of the characteristics of developing nations is industrialization. However, industrialization though advantageous has its attendant problems such as heavy metal pollution, as most industrial effluents are a source of heavy metal pollution in the aquatic environment wherein they are discharged. Sadly, in most of the developing countries, the treatment of municipal water does not include facilities to remove heavy metals; thereby exposing the inhabitants to possible heavy metal - contamination of the water in their reach (Okuo and Oviawe, 2007). Cadmium is one of these heavy metals and is among the priority pollutants listed by the Department of Environment, UK because it is poisonous. Besides, being carcinogenic, it can cause both acute and chronic levels of gastrointestinal disorders, abdominal pains, vomiting, diarrhea, renal disorders, hypertension and testicular degeneration (Ensafi and Ghaderi, 2007). Cadmium gains final entrance into the aquatic environment through wastes from metal plating, smelting, phosphate fertilizers, cadmium - nickel batteries,

mining, stabilizers, pigments and alloy industries (Sher *et al.*, 2013). There are several conventional methods for the removal of metal ions from aqueous solutions namely: chemical precipitation, ion exchange, chemical oxidation and reduction, electrochemical treatment, filtration, evaporative recovery, reverse osmosis and solvent extraction (Salem and Alliam, 2008; Isik, 2008; Dundar *et al.*, 2008).

A biological method known as biosorption is an upcoming and attractive, alternative method for the removal of trace metal ions from effluents because they are more feasible economically and environmentally friendly (Pagnanelli *et al.*, 2002). It involves the use of special solids known as adsorbents to remove substances (adsorbates) from either gaseous or liquid phase mixture (Asiagwu *et al.*, 2012). Plantain stalk (*Musa paradisiaca*) is the adsorbent used in this study. Plantain is a source of food all over the world. Developing nations like Nigeria face solid waste disposal problem after harvest and consumption

of the plantain because the stalk are left as waste. Therefore, this study is aimed at the conversion of this plantain stalk waste to useful product for the removal of Cd(II) ions from aqueous solution. The mechanism of the process by which the metal ions or adsorbate ions are adsorbed to the adsorbent will be necessary, hence this work looks at the kinetics and the equilibrium isotherm modeling of the process as well as the chemical make-up of the active sites.

MATERIALS AND METHODS

The various chemicals used in this work were of analytical grade produced by JHD Chemical Company. Atomic Absorption Spectrophotometer (AAS) (model Philip Pu9100x) was used for the residual metal ion analysis. The instrument was calibrated and periodically checked for response with a spectroscopic grade standard. All measurements were done with a hollow cathode lamp and fuel rich flame. The batch experiments were done in duplicates so that the mean was taken to maintain accuracy and ensure reproducibility. Fourier Transform Infrared (FTIR) study was carried out using Nicolet Avator 330, England, for functional group analysis of the adsorbent at pH 6 recorded in the range $4000 - 500 \text{ cm}^{-1}$. A Jenway pH meter was used for measuring the pH values in the aqueous phase. The pH meter was first calibrated before each use using buffer 4.0, 7.0 and 9.0 solutions.

The plantain stalk (*Musa paradisiaca*) was collected from different dump sites in Abraka community in Ethiope East Local Government Area of Delta State, Nigeria. It was first washed severally with tap water to remove sand and debris and then with deionised water, then cut into small pieces and sun-dried for five days. After drying, it was pulverized using an electric blender and homogenized by passing through 75 μm sieve and then stored in an air-tight polythene bag ready for use in the sorption experiments.

The aqueous stock solution of Cd(II) ions was prepared with the hydrated nitrate salts following the method of Jimoh *et al.*, (2012) in which 2.74 g of $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was carefully weighed and dissolved in de-ionized water in a beaker, quantitatively transferred into a 1000 mL standard volumetric flask and made up to the mark, which gave a concentration of 1000 mg/L.

Dilution of the stock solution was made from the 1000 mg/L to 50 mg/L for the analysis of pH and contact time and serial dilutions of 10, 30, 50, 100, 150, 300 and 500 mg/L of the adsorbate solution was also made for concentration-dependent analysis.

Batch adsorption experiments in which the effect of pH (ranging from 2 - 8) on the removal of Cd(II) ions by *Musa paradisiaca* was studied following the work of Babalola *et al.*, (2011). A 1 M HNO_3 and 1 M NaOH or 0.05 M HNO_3 and 0.05 M NaOH solutions were used for adjusting the solution pH to the required value. The adsorbent was then mixed with 25 mL of the adsorbate solution and shaken on a rotary shaker at 240 rpm for 1 hour. After shaking, the suspensions were filtered with whatman no. 45 filter papers and the concentrations of the metal ions were determined using Atomic Absorption Spectrophotometer (AAS).

The time-dependent experiment of the metal ions binding capacity on the adsorbent was done following the method of Babalola *et al.*, (2011). A 25 mL aliquot of the aqueous solution of the metal ion at optimum pH of 7.0 was measured into several bottles and mixed with 50 mg of the adsorbent, well corked and then the mixtures were consistently agitated on a rotary shaker at 240 rpm, at the time intervals of 5, 10, 30, 60, 90, 120 and 180 minutes. Thereafter, the mixtures were filtered in the same way earlier described and the residual concentrations of the Cd(II) ions were measured using AAS. This experiment was carried out in duplicate.

The concentration-dependent experiment for the Cd(II) ions binding capacity on the adsorbent was carried out by repeating the same procedure but at varied initial adsorbate concentrations at 27 °C prepared by serial dilution of the stock Cd(II) solution and at the pH and time which have been pre-determined. The results of the time - dependent study and concentration - dependent study were subjected to two kinetic models namely: pseudo first order and pseudo second order models and two isotherm models: Langmuir and Freundlich models respectively. Eligible plots were made using origin 6.0 statistical software and the R^2 values were generated from which the model which fitted better was determined. The relevant kinetic and

equilibrium parameters were deduced accordingly from the slopes and intercepts of the plots.

FTIR analysis was performed by weighing 50 mg of the biomass and mixing with 25 mL of de-ionized water at pH 7 to determine the functionality of the biosorbent. Also, to enable the determination of functional groups which make up the active sites and are involved in Cd(II) binding, another 50 mg of the *Musa paradisiaca* was weighed and mixed with 25 mL of the metal ion solution at pH 7 in a plastic bottle. Both mixtures were agitated for 1 hour at the speed of 240 rpm using a rotary shaker. After the shaking, the mixture was filtered using the same filter paper to obtain the residues for FTIR analysis. The finely pulverized dried *Musa paradisiaca* was mixed with KBr in the ratio 1 : 200 and pressed into a mould with a hydraulic press and mould. The resulting pellet was then analyzed with an FTIR spectrometer in the wave number range of 4000-500 cm^{-1} .

RESULTS AND DISCUSSION

Effect of pH: The pH is a very relevant factor in the adsorption of heavy metals from aqueous solution because it affects the surface charge and speciation of the metal ions in solution. The effect of increase in pH on the percentage sorption of Cd(II) ion onto *Musa paradisiaca* stalk is shown in Figure 1.

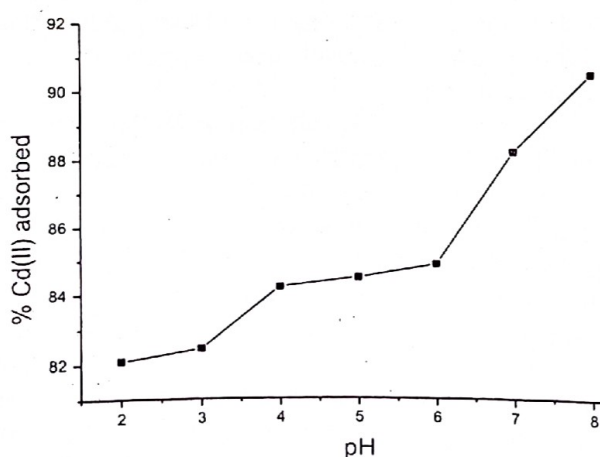
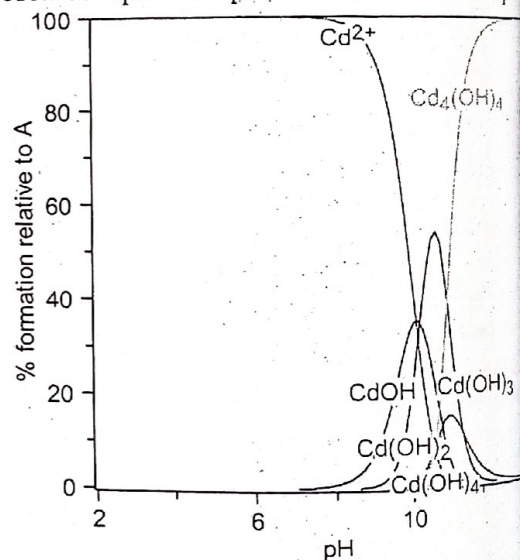


Fig.1: Effect of pH on the % Cd(II) ions adsorbed unto plantain stalk waste biomass

The data reveals that the solution pH has some effect on the percentage removal of Cd(II) ion from aqueous solution on to the adsorbent. The percentage removal increased steadily with increase in the solution pH from 2-8. In acidic pH, there is competition for binding sites between the Cd(II) and hydrogen ions which exist in acidic medium leading to lower removal in low pH (acidic) medium than in higher (alkaline) pH. A speciation diagram (Figure 1b) was generated using the Hyperquad Simulation and Speciation software to determine at what pH, insoluble precipitates of Cd(II) are precipitated under the given experimental conditions. A library of critical stability constants provides the likely species or forms of reagents present in solution as well as their stability constants at specific temperatures and ionic strengths. A plot of the percentage formation relative to A (where 'A' refers to Cd) against pH is shown in Figure 1b. From the Figure, Cd(II) ions begin to precipitate significantly from pH 8. Therefore pH 7 was chosen as optimum pH and used in subsequent



studies.

Fig. 1b. Cd^{2+} Speciation as a function of pH [log β of $\text{Cd}(\text{OH})^+$, 10.097; $\text{Cd}(\text{OH})_2$, 20.29; $\text{Cd}(\text{OH})_3^-$, 31.691; $\text{Cd}(\text{OH})_4^{2-}$, 47.28; $\text{Cd}_4(\text{OH})_4^{4+}$, 32.788; $\text{Cd}_2\text{OH}^{3+}$, 9.397; $\text{Cd}(\text{OH})_4$ (s), 14.35; Cd (s), 14.1; pK_w , 13.77, ionic strength of about 0.1 mol dm^{-3} , Cd concentration - $4.448 \times 10^{-4} \text{ mol dm}^{-3}$ at 25 $^\circ\text{C}$]

Effect of contact time: This is another important factor in the treatment of waste water. The rate at which Cd(II) ions are sorbed onto the adsorbent

was investigated and the result is presented in Figure 2.

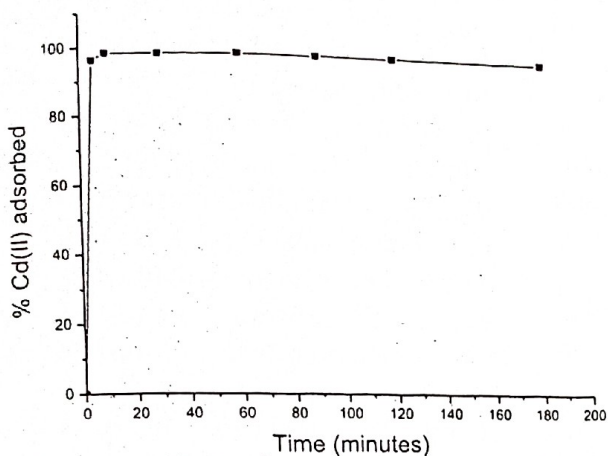


Fig. 2: Effect of contact time on the Cd(II) ions, biosorption unto plantain stalk waste biomass

There is a rapid initial sorption of Cd(II) ions by plantain stalk at the first 10 minutes: (above 90%). This may be due to strong attractive force between the metal and the adsorbent. As the time increases there is a little rise in the percentage of Cd(II) ions sorbed by the biomass. Since there is a significant removal of Cd(II) ions within the first 10 minutes, extending the time would be insignificant. The results are in line with those of Hynda and Rachida (2008).

The effect of Cd(II) concentration: This effect was investigated in the concentration range from 10 - 500 mg/L, and the result is shown in Figure 3.

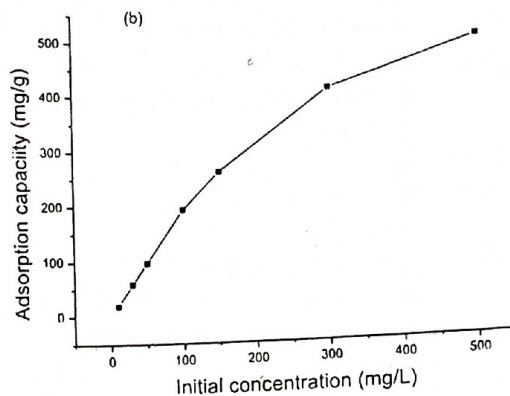
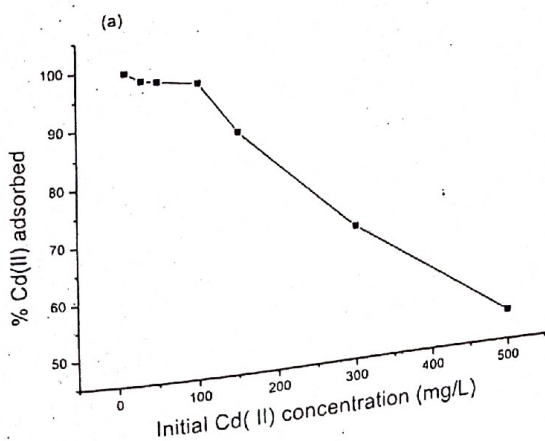


Fig. 3: Effect of initial metal ion concentration on the (a) percentage adsorption of Cd(II) ions and (b) adsorption capacity of plantain stalk waste biomass for Cd(II) ions.

When the initial adsorbate concentration increases the adsorption efficiency decreases. This may be due to the quick saturation of the adsorbent since a constant dose was used even at low adsorbate concentration. Nevertheless, the adsorption capacity increases when initial adsorbate concentration is increased. This outcome suggests that there is competition of metal ions for the binding sites which are available. Moreover, the higher the initial concentration of the adsorbate ions, the more the force to overpower likely resistance to mass transfer of the Cd(II) ions from the bulk aqueous phase to the solid phase (Onyancha *et al.*, 2008). From the data, there is rapid sorption from the lowest concentration to the highest concentration without any significant difference. The rapid sorption at the lower concentration can be credited to the surplus of the active sites on which the sorbate could easily attach (Inamullah *et al.*, 2007).

Langmuir Isotherm Treatment: Langmuir theory proposes that sorption takes place at specific homogenous sites on the adsorbent. It assumes that the point of complete formation of a monolayer by the adsorbate ion is the point at which equilibrium is reached. This model is represented with Equation 1:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \dots \dots \dots (1)$$

Where K_L is the Langmuir constant, q_m is the monolayer adsorption capacity of the *Musa paradisiaca*, q_e is the amount of the cadmium ion

(mg) taken up per gram of the biosorbent at the point of equilibrium and C_e is the concentration remaining in solution (mg/L) at equilibrium. Equation 1 can be rearranged or made linear into Equation 2:

$$\frac{C_e}{q_e} = \frac{1}{x_m K_L} + \frac{C_e}{x_m} \dots \dots \dots (2)$$

The capacity of the biomass and Langmuir constant can be obtained from the slope and intercept respectively, where a plot of C_e/q_e against C_e is made.

Fruendlich Isotherm Treatment: Fruendlich model can be written mathematically as in equation (3):

$$q_e = K_f C_e^{1/n} \dots \dots \dots (3)$$

Where: q_e and C_e have been defined earlier, K_f is the Fruendlich constant and n is a measure of the intensity of adsorption. Again, Equation (3) can be made linear to take the form of equation (4):

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \dots \dots \dots (4)$$

Plotting $\log q_e$ versus $\log C_e$ results to a straight line confirming the Fruendlich isotherm. From the slope and intercept, the values of n and K_f may be estimated respectively.

The equilibrium sorption data obtained during the analysis from Langmuir and Fruendlich equations are shown on Table 1 and the plots are presented in Figures 4 and 5.

Table 1: Langmuir and Fruendlich isotherms parameters for Cd(II) biosorption on *Musa paradisiaca*

Langmuir Isotherm			Fruendlich Isotherm		
q_m (mg/g)	K_L (L/g)	R^2	K_f	n	R^2
980.4	0.0023	0.982	4.81	2.6	0.974
0		3	5	2	7

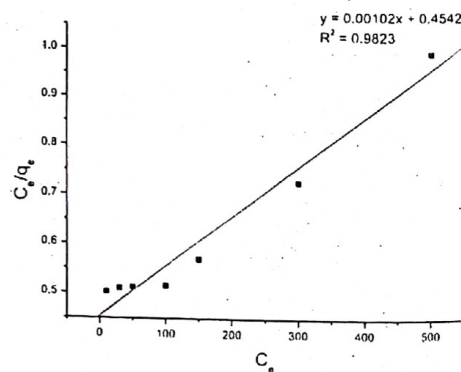
The data in Table 1 showed that the Langmuir isotherm provided a better for the equilibrium sorption data than the Fruendlich isotherm, due to the higher R^2 value of 0.9823. The data also revealed that the adsorbent has a great q_m of 980.4 mg/g. Shibi and Anirudhan (2006) reported a maximum monolayer adsorption capacity of 65.88 mg/g for Cd(II) adsorption onto polymer-grafted *Musa paradisiaca* while Ogunsile and Babarinde (2013) reported 7.73 mg/g for Cd(II) adsorption onto same adsorbent. In both cases,

the Langmuir model fitted better than the Fruendlich's, which is in line with the findings of this work. The enhancement in the value of q_m in this work over those earlier mentioned using the same adsorbent may be particularly due to the difference in the particle size of the *Musa paradisiaca* in each case and other factors summarized in Table 2. The particle size in this work is the smallest among the three cases, hence the amount adsorbed was highest in this work. This is because the smaller the particle size, the greater the surface area and the higher the amount adsorbed per gram of the adsorbent.

Table 2: Comparism of q_m and experimental conditions for Cd(II) adsorption onto *Musa paradisiaca*

Conditions	Ogunsile and Babarinde; (2013)	Shibi and Anirudhan (2006)	Present study
Adsorption capacity q_m (mg/g)	7.73	65.88	980.40
Particle size (um)	500 - 1000	80 - 230	≤ 75
Time (minutes)	60	180	10
pH	6.0	6.5	7.0
Adsorbent dose (mg)	150	100	50
Temperature of study (K)	300	303	300

(a)



(b)

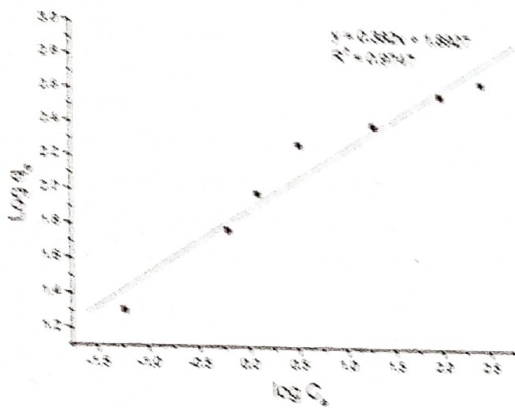


Fig. 4(a) Langmuir (b) Freundlich adsorption isotherms for Cd(II) ion removal from aqueous solution by plantain stalk waste biomass

Pseudo first-order kinetics:

The rate equation is shown below:

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \dots\dots\dots (5)$$

Where q_e and q_t are the milligrams of Cd(II) ions sorbed per gram of *Musa paradisiaca* at equilibrium and at time t , respectively and k_1 is the rate constant for the pseudo first-order mechanism. Integrating Equation 5 results in equation 6:

$$\log(q_e - q_t) = k_1 - \frac{k_1 t}{2.303} \dots\dots\dots (6)$$

The log of $(q_e - q_t)$ was plotted against time, t . The value of q_e was obtained from the intercept while k_1 was estimated from the slope of the graph.

Pseudo second-order kinetics:

The pseudo second-order kinetic model is represented by equation 7:

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \dots\dots\dots (7)$$

Where k_2 is rate constant for pseudo-second order process and integrating equation 7 leads to equation 8:

$$\frac{q_t}{q_e(q_e - q_t)} = k_2 t \dots\dots\dots (8)$$

On linearizing,

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \dots\dots\dots (9)$$

Plotting t/q_t against time results in a straight line with $1/q_e$ as slope and $1/k_2 q_e^2$ as intercept. Both models were checked for their suitability using the correlation coefficients R^2 and the

pseudo second - order model proved to be more suitable with a q_e of 254.88 mg/g, correlation coefficient of 0.9999 and K_2 of $0.123 \text{ mg g}^{-1} \text{ min}^{-1}$ which are displayed in Table 3.

Table 3: Pseudo second - order kinetic parameters for Cd(II) ions removal by *Musa paradisiaca* waste biomass

Metals ion	K_2 (mg/g/ mins)	Initial rate (mg g/min)	q_e (mg/g)	R^2
Cd(II)	0.123	76.14	24.88	0.999

The correlation of the experimental result with the pseudo second - order model suggests that chemisorption reaction would be the main adsorption mechanism here (Upendra, 2006). The adsorption of Cd(II) ions as well as other metal ions onto *Musa paradisiaca* has been established to be pseudo - second order (Ogunsile and Babarinde, 2013; Shibi and Anirudhan, 2006) and in this case, with an initial rate $h = 76.14 \text{ mg g}^{-1} \text{ min}^{-1}$.

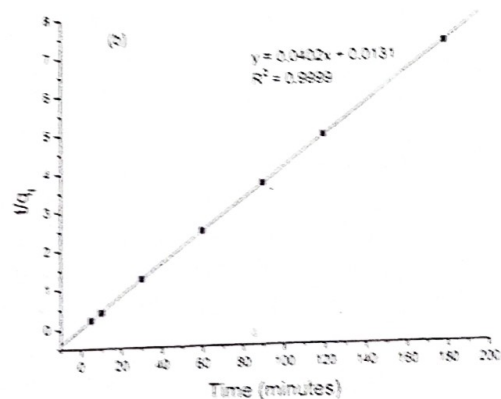
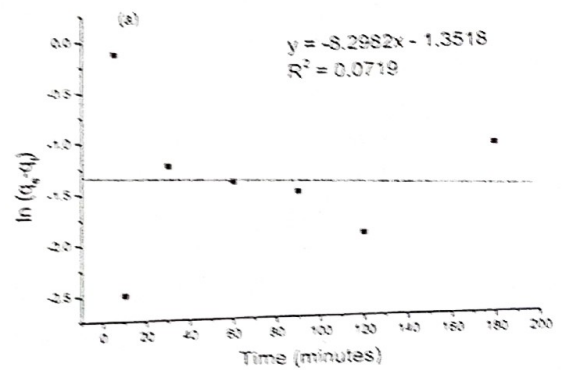
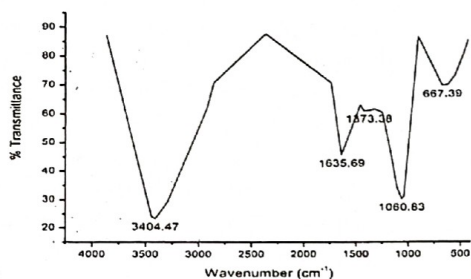


Fig. 5 (a) Pseudo first-order (b) Pseudo second-order kinetic plot for the sorption Cd(II) ion by plantain stalk waste biomass.

FTIR Studies:

The characterization of the biosorbent is important because it provides an understanding of the mechanism by which the metal ions are bound on the solid surface through the identification of the functional groups which make up the active sites. The results of the FTIR analysis of the biomass before and after binding with Cd(II) ions are shown in Figures 6 (a) and (b) respectively.

(a)



(b)

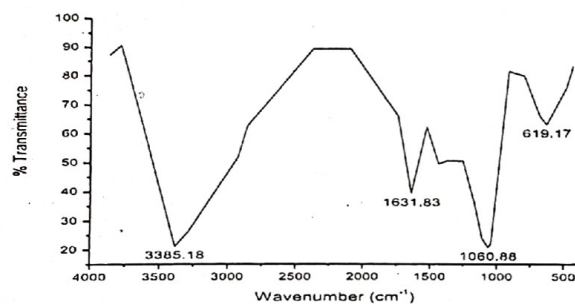


Fig. 6a: The FTIR spectrum patterns of (a) plantain stalk at pH 7 before metal binding (b) Cd(II) - loaded plantain stalk at pH 7.

The IR spectra of the *Musa paradisiaca* at pH 7 shows a few distinct absorption bands (Fig. 6a). For example the distinct and sharp absorption at 3404 cm⁻¹ which is indicative of -OH. The absorption band around 1635.69 cm⁻¹ depict mainly C=O stretch and suggests that the band around 1373 cm⁻¹ is due to C = C stretch. (Rifaqat and Moonis , 2002; and Kalsi, 2004). The band around 1060 cm⁻¹ could be attributed to C-OH stretch of sugar (Rifaqat and moonis, 2002), while the band around 667 cm⁻¹ may be due to metal-oxygen bond probably Si-O due to

trace impurities of sand. The Presence of C and carbonyl groups implies that the biosorbent contains carboxylic acid group and these groups are major active sites in biomaterials (Voleski, 2003). Comparing the spectrum of the Cd(II) laden biomass with that of the non laden at pH 7 it is seen that the band at 3404 cm⁻¹ broadened and moved to a lower wave number (3385.18 cm⁻¹) after Cd(II) adsorption. Also the previous bands around 1635.69 cm⁻¹ and 667 cm⁻¹ shifted to lower wave numbers while the band around 1373 cm⁻¹ shifted to a higher wave number. There are no new peaks formed after the adsorption of Cd(II) therefore it can be said that no new compound is formed (Kalsi, 2004).

CONCLUSION

From the study, it is found that *Musa paradisiaca* biomass is a good and affordable biosorbent for sorption of Cd(II) ions from aqueous solution. The process is affected by initial pH, contact time and initial concentration of the Cd(II) solution, following a pseudo second-order mechanism and being homogenous in nature as provided by the better fit into the Langmuir model with a Langmuir monolayer maximum adsorption capacity of 980.40 mg/g. The FTIR analysis reveals that the functional groups which make up the active sites are mainly hydroxyl and carbonyl groups.

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