



Kinetic and Equilibrium Adsorption Studies of Pb and Zn ions from Aqueous Solution onto Plantain Stalk (*Musa Paradisiaca*)

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ABSTRACT: This study presents the sorption of Pb^{2+} and Zn^{2+} from aqueous solution by *Musa Paradisiaca* (plantain stalk) with respect to its equilibrium and kinetic behaviour. The optimum pH was found to be 5.0 and 6.0 for the sorption of the Pb (II) and Zn (II) ions respectively. At pH 5.0, the biomass sorbed 91.7% Pb(II) and 89.85% Zn(II), while at pH 6.0, the biomass sorbed 91.16% Pb (II) and 89.55% Zn (II). The contact time for the sorption was found to be rapid in the first 5mins. The amount of metal ion sorbed was found to be rapid from the lowest concentration, 10 mg/L to the highest concentration, 500 mg/L. The Langmuir and Freundlich model for dynamic metal ion uptake proposed were well fitted for Pb^{2+} and not well fitted for Zn^{2+} . The structural groups of the adsorbent were characterized by Fourier transform infrared (FTIR) Spectrometry. The following were present; hydroxyl, carbonyl, amine and phosphate groups which confirms the potential processes of sorption of the biosorbent. The first order and second order rate equation were tested and it was found that pseudo-second-order is more suitable for the sorption, having the linear coefficient of determination R^2 value of 0.999. The result obtained from this study indicated that plantain stalk (*Musa Paradisiaca*) could be employed for the removal of heavy metal contaminants from industrial effluents.

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Despite a lot of benefits obtained from industrialization, it has been shown that is one of the major environmental pollution (Osakwe and Ibrahim, 2017). The conventional technologies for effluent treatment are not economically feasible for small scale industries prevalent in developing economics due to huge capital investment. It is therefore important to carefully sort for different adsorbents which are of cheap value, and are naturally occurring products that have good sorbent properties and are of no important to people (Enemose and Osakwe, 2014). Since the olden days methods are neither efficient nor economical, especially, when used for the reduction of the concentrations of heavy metal ions, new separation methods are required to reduce heavy metal concentration to environmentally approved levels at affordable cost. Biosorption has the potential to contribute to the achievement of this goal especially because it is environmental friendly. (Klimmek *et al.*, 2001). In addition to being environmental friendly, it has other good advantages such as low cost, high efficiency, regeneration of biosorbent and possibility of metal recovery (Rajput, 2015). The biosorption of Pb(II) and Zn(II) ions from aqueous solutions has been analysed by different researchers. Hynda and Rachida, (2008), studied the biosorption of lead (II) ions from

aqueous solution by biological activated dates stems, by carrying out batch experiment under varied conditions of pH, contact time, initial Pb^{2+} dates stems concentration and temperature. The biosorption capacity was found to increase in the free parameters studied. The biosorption for Pb^{2+} onto activated dates stems obeyed the Langmuir and Freundlich isotherms models. It was indicated that the biosorption for Pb^{2+} onto activated date stems could be described by the pseudo-second-order kinetics. Opeolu *et al.*, (2011), reported zinc abatement from simulated and industrial wastewater using sugarcane biomass under varied conditions of contact time, biomass weight, metal concentration, pH, agitation's speed, temperature and particles size. The physicochemical characteristics of biomass were also studied. They observed that as zinc adsorption is increasing, contact time, biomass weight, pH and agitation speed are also increasing, while adsorption efficiency decreased with increasing particles sizes for temperature above 50°C.

Sugarcane biomass was responsible for over 90% adsorption of Zn^{2+} in both effluents. Under condition of agitation, 100% adsorption was achieved. Percentage ash and cation exchange capacity were positively correlated to percentage adsorption while

particles density and porosity were negatively correlated. Percentage desorption was over 90% for both effluents. In this study, plantain stalk (*Musa paradisiacal*) was used. Plantain has been source of food all over the world. Developing nation like Nigeria faces solid waste disposal problem, this necessitate the need for the conversion of this plantain stalk serving as waste in the environment to useful products for removal of metal ions from aqueous solution, which is a cheap adsorbent and it will be of great benefit to the environment and as well as scientific community. This study is aimed at generating useful information for the effective utilization of native agricultural by-products such as plantain stalk (*Musa paradisiaca*) for the removal of metal ions from aqueous solution. Hence this work has looked at the kinetics of the sorption and the equilibrium modeling of the process.

MATERIALS AND METHODS

Chemicals and sample preparation: Chemicals used in this study, were of analytical grade made by JHD Chemicals Company. Atomic Absorption Spectrophotometer (AAS) model Philip PU9100X with a hollow cathode lamp and fuel rich flame (air acetylene) was used for residual metal ion analysis. The instrument was calibrated with spectroscopy grade standard, which was checked periodically for instrument response. All measurements were done in air/acetylene flame.

The batch experiments were carried out in duplicate and the average computed for each set of values to maintain accuracy. Fourier Transform Infrared Spectrometer (Nicolet Avator 330, England) was used for functional group analysis.

Fourier-Transform Infrared (FT-IR) spectra of unloaded biomass at pH 4.0 and 5.0 and metal loaded biomass was recorded at 500-4000 cm^{-1} range. Jenway model pH meter was used to measure pH in the aqueous phase.

The plantain stalk (*Musa paradisiaca*), was collected from different dump sites in Abraka community in Ethiope East Local Government Area of Delta State, Nigeria. The biomaterial was washed with deionised water and then cut into small pieces and sun-dried for five days. After drying, it was pulverized using an electric blender, passed through a 75 μm sieve and stored in an air-tight polythene bag ready for the sorption experiment.

The aqueous stock solution of Pb^{2+} and Zn^{2+} was prepared with their various salts, following the method of Jimoh *et al.*, (2012). 4.55 g of $\text{Zn}(\text{NO}_3)_2$ and 1.60 g of $\text{Pb}(\text{NO}_3)_2$, were carefully weighed, dissolved in

deionised water in a beaker, quantitatively transferred into a 1000 cm^3 standard volumetric flask and made up to the mark, which gave a concentration of 1000 mg/L. Dilution of each stock solution was made from 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 each was also made for the concentration dependent study.

Sample characterization: FTIR analysis of plantain stalk (*Musa Paradisiaca*) was performed by weighing 50 mg of the adsorbent and mixed with 25 cm^3 of each metal solution for an adsorption study at pH 4.0 for Pb^{2+} and pH 5.0 for Zn^{2+} in several flasks. The blank was prepared using the same 50 mg and 25 cm^3 of deionized water at pH 4.0 for Pb^{2+} and pH 5.0 for Zn^{2+} . The mixture was shaken for 1 hr at the speed of 240 rpm using a rotary shaker, thereafter, it was filtered to obtain the residues for Fourier Transform Infrared (FTIR) analysis. One milligram of dried, powdered adsorbent was mixed with 200 mg of KBr and pressed using a hydraulic press and mould. The mixture obtained was immediately analyzed with FTIR spectrometer in the range of 4000 to 500 cm^{-1} .

Batch equilibrium experiment: Using 50mg of the biosorbent for each batch equilibrium experiment, the effect of pH (ranging from 2 to 8) on sorption of Pb(II) and Zn(II) ions by *Musa Paradisiaca* was investigated. 1 M HNO_3 and 1 M NaOH or 0.05 M HNO_3 and 0.05 M NaOH were used to adjust the pH of solution to the corresponding pH under investigation. The biomass was then mixed with 25 ml solution of the salt and agitated on a rotary shaker at 240 rpm for 1 hr. After shaken, the suspensions were then filtered with whatman no. 45 filter paper and the concentration of the metal ions were determined by Atomic Absorption Spectrophotometer (AAS). Each experiment was carried out in duplicate.

The effect of contact time on metal ions binding capacity of the adsorbent was performed by measuring 25 cm^3 of the aqueous solution of each metal ions at the optimum pH of 5.0 for Pb (II) and Zn (II) into several bottles and mixed with 50 mg of the biomass. These were well corked and the mixture was constantly shaken in a rotary shaker at 240 rpm, at time intervals of 5, 10, 30, 60, 90, 120 and 180 minutes. After each contact time, the mixture was filtered using Whatman no. 45 filter paper and the concentration of each metal ions was determined using AAS. The analysis were done in duplicates.

Kinetic data analysis: The results were imputed into the pseudo-first order and pseudo-second order kinetic models. Appropriate plots were made from which the

correlation co-efficient R^2 values were obtained to determine the most fitting model. The corresponding kinetic parameters calculated from the slope and intercept of such plots. The effect of concentration on metal ions binding capacity of the adsorbent were performed by measuring 25 cm³ of serial dilutions of 10, 30, 50, 100, 150, 300 and 500 mg/L made from the stock solution and mixed with 50 mg of the biomass in several plastics bottles. These were well corked and the mixture was constantly shaken at optimum time for the metal ions solution in a rotary shaker at 240 rpm. After shaking, the mixture was filtered using Whatman no. 45 filter paper and the concentration of each metal ion was determine using AAS.

Adsorption Isotherm: Two of the most sorption models were used to fit the experimental data. The Langmuir model which assumes that equilibrium is achieved when a monolayer of the adsorbate molecules saturates the adsorbent. This model can be presented as in equation 1

$$q_e = \frac{X_m K_L C_e}{1 + K_L C_e} \quad \text{--- (1)}$$

Where X_m and K are the Langmuir constants and specifically X_m is the monolayer and sorption capacity of the biomass, q_e is the concentration of metal ion on the biomass (mg/g) at equilibrium and C_e is the concentration (in mg/L) remaining in solution at equilibrium. The linear form of the Langmuir model is given in equa

$$\frac{C_e}{q_e} = \frac{1}{X_m K_L} + \frac{C_e}{X_m} \quad \text{--- (2)}$$

The capacity of the biomass can be obtained if a plot of C_e/q_e against C_e is made.

The second model is Freundlich model which can be written as in equation 3. The mathematical equation is given as

$$\frac{X}{m} = K C_e^{\frac{1}{n}} \quad \text{--- (3)}$$

Where: X is the mass of metal ion adsorbed (mg), m is the mass of biomass used (g), C_e is the concentration of metal ion at equilibrium, n is the adsorption intensity and K is the adsorption constant. The linear form of equation 3 take the form of equation 4.

$$\ln \frac{X}{m} = \ln k + \frac{1}{n} \ln C_e \quad \text{--- (4)}$$

A plot $\ln \frac{X}{m}$ against $\ln C_e$ will give a straight line which confirm the Freundlich isotherm.

Pseudo-first-order kinetics: The rate law is shown below

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

Where q_e and q_t are the amount of each Pb (II) and Zn (II) ions sorbed at equilibrium and time t , respectively, K_1 , is the rate constant for the Pseudo-first-order biosorption. The integrated rate law becomes

$$\ln (q_e - q_t) = \frac{(q_e - k_1 t)}{2.303} \quad \text{--- (6)}$$

A plot of $\ln (q_e - q_t)$ against time t , was made and values of K_1 , and q_e were obtained from slope and intercept, respectively.

Pseudo-second-order kinetics: Applicability of the second-order kinetic is tested with the rate equation.

$$\left[\frac{dq_t}{dt} \right] = k_2 (q_e - q_t)^2 \quad \text{--- (7)}$$

Where K_2 is rate law for pseudo-second order biosorption. On integrating between the boundary condition of $t = 0, t = t$ and $q = 0, q = q_t$, the following expression was obtained.

$$q_t \frac{qt}{(q_e - q_t)} = k_2 t \quad \text{--- (8)}$$

On linearizing

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

A plot of t/q_e against t gives $(1/q_e)$ as slope and $(1/K_2 q_e^2)$ as intercept from which K_2 can be obtained. Both models tested for suitability using their correlation of coefficient R^2 .

RESULTS AND DISCUSSION

FTIR spectroscopy: The physical and chemical characteristics of biosorbent are important for understanding the metal binding mechanisms on the biomass surfaces and these are presented in fig 4 and 5. The percentage transmission for various wave number of the FTIR analysis of plantain stalk (*Musa paradisiaca*) is presented in fig. 4 (a & b) and 5 (a & b), the FTIR spectra of the adsorbent show the presence of hydroxyl, amine, carbonyl, Esters, Alkenes, phosphate, nitro and azides for the metal

adsorbed by the biomass at pH 4.0 and 5.0 for those passed through metal ions solution and the blank sample ie biomass passed through deionized water. It was found that there was no significant different. The presence of -OH group, along with carbonyl group confirm the presence of carboxylic acid group in the biosorbent. The -OH, -NH, carbonyl and carboxylic group are important sorption site (Volesky, 2003).

Table 1: Textural characterization of plantain stalk (*Musa Paradisiaca*) on lead at pH 4.0

Wavelength (cm ⁻¹)	Assigned functional groups
3383.26	O - H stretching vibration of cellulose
3288.74	N - H stretching vibration of cellulose
2922.25	C - H stretching vibration of cellulose
2161.95	C = H stretching vibration of cellulose
1951.28	C - C stretching vibration of cellulose
1728.28	-CO - O - Aryl and αβ - unsaturated
1631.83	C = O non-conjugate
1107.18	C - O stretching vibration of cellulose

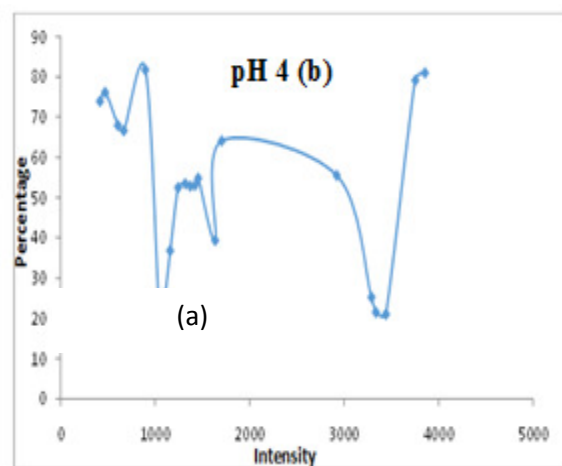
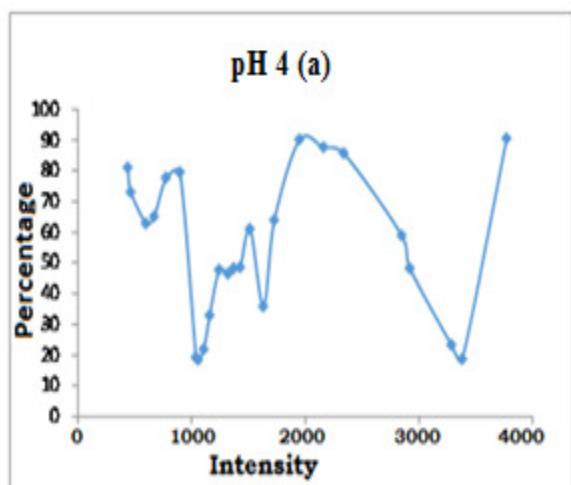


Fig. 4 (a and b): The FTIR spectrum patterns of plantain stalk (*Musa paradisiaca*) on lead at pH 4.0 (a), and on deionised water at pH 4.0 (b)

Table 2: Textural characterization of plantain stalk (*Musa paradisiaca*) on zinc at pH 5.0

Wavelength (cm ⁻¹)	Assigned functional groups
3396.76	O - H stretching vibration of cellulose
3267.52	N - H stretching vibration of cellulose
2922.25	C - H stretching vibration of cellulose
2360.95	P - H stretching vibration of cellulose
1728.28	-CO = C - C Aryl and αβ - unsaturated
1631.83	C = O non-conjugate
1514.17	C - N = O stretching vibration of cellulose
1105.25	C - O stretching vibration
1037.75	P - O alkyl stretching vibration of cellulose

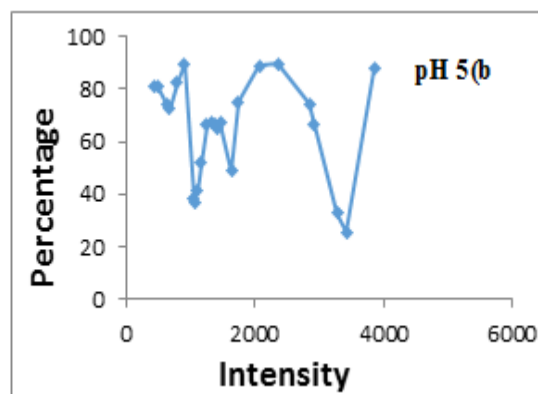
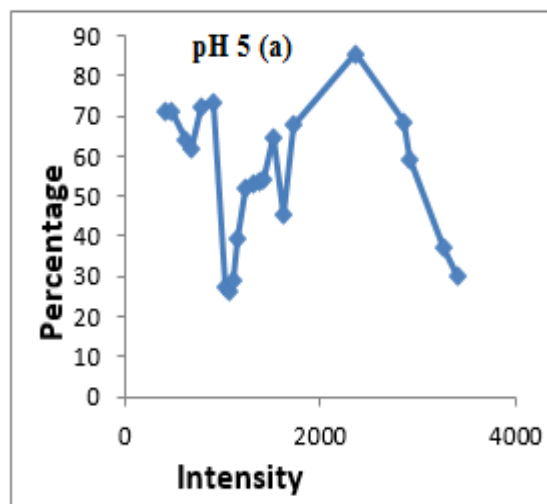


Fig 5: (a, and b): The FTIR spectrum patterns of plantain stalk (*Musa paradisiaca*) on zinc at pH 5 (a) and on deionised water at pH 5 (b)

Effect of pH: As important as pH, it affects the biosorption of heavy metals from aqueous solution. The variation of the percentage of Pb (II) and Zn (II) sorbed by *Musa paradisiaca* at various pH values (2 to 8) is shown on fig. 1.

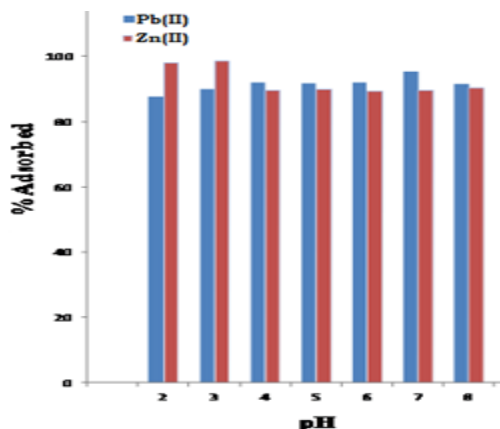


Fig. 1: Effect of pH on Pb (II) and Zn (II) ions biosorption on plantain stalk (*Musa paradisiaca*)

The pH dependence data for the sorption of the two metals under investigation with plantain stalk (*Musa Paradisiaca*) are presented on figure 1. The data revealed that at pH 2.0, there was a significant removal of the metal ions by the biomass: 87.76% of Pb (II) and 98.03% of Zn (II). However as the pH increase to 5.0 there was increase in the amount of metal removed from Pb (II) ions and a slight decrease in the amount remove from Zn (II) ions by the biomass 91.79% Pb (II) and 89.85% Z (II). The amount of metal ions removed by the biomass at low pH of 2.0 were a little bit high in Pb (II) ions and a little bit low in Zn (II) metal ions compared to those removed at pH 5.0. This might be as a result of decrease or an increase in competition between the hydrogen ion and the metal ion, because at this pH, the concentration of hydrogen ion is high (Pamukoglu, 2007).

As the pH increase from 5.0 to 6.0, more respectively, sorption sites become available and this facilitate increase in amount of metal ions sorbed. The maximum sorption at pH 5.0 for Pb (II) and Zn (II) ions respectively may be attributed to the negative charge density on the biosorbent surface which increased due to deprotonation of the metal-binding and thus increases the sorption of metal ions (Hynda and Radida, (2008). Furthermore, adsorption was reported to depend on factors other than pH, which include flow rate residence time, effects of dissolution of some other materials from adsorbate, amongst others (Shukia and Pai 2005). This may explain the continued increase in adsorption of metal by biomass after pH 6.0 when precipitation of metal is expected to start.

Effect of Contact Time: The rate at which Pb (II) and Zn (II) was sorbed onto *Musa paradisiacal* was studied and the results are presented on figure 2 which shows the percentage adsorb against time t.

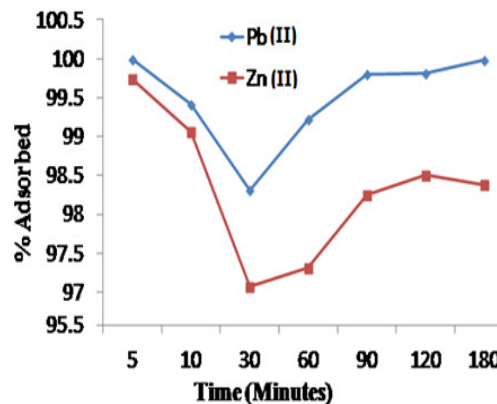


Fig. 2: Effect of contact time on the Pb (II) and Zn (II) ions, biosorption on plantain stalk (*Musa paradisiaca*)

There is a rapid sorption of Pb (II) and Zn (II) ions by the biomass at the first 5 and 10mins: 99.99% and 99.44% Pb (II), and 97.74% and 99.06% Zn (II) respectively. This may be due to strong attractive force between the two metal ions and adsorbent (Hynda Rachida 2008). Then the metal ion sorption for each of the metals did not significantly change.

The result demonstrate that maximum sorption is an important parameter for large – scale application in industrial process, this finding was ascribed to highly porous structures of adsorbent, which provides ready access and large surface area for sorption metals on the binding sites (Cabuk *et al.*, 2007). The maximum sorption of Pb(II), and Zn(II) ions was attained within 5 and 10 minutes. Therefore increasing the contact time further had no or little effect on the amount of the heavy metals sorbed. These results are in line with those of Hynda and Rachida (2008).

Effect of concentration: The results plotted on fig 3, with percentage adsorb against concentration, shows the rate of adsorption of Pb (II) and Zn (II) ions onto plantain stalk. The data in figure 3 showed the different adsorbate concentration ranging from 10, 30, 50, 100, 150, 300 and 500 mg/L.

The data in the figure revealed that there is a rapid sorption from the lowest concentration to the highest concentration without any significant difference on both metals. The rapid sorption at the lower concentration are ascribed to the sufficient active sites which the sorbent could easily occupy (Inamullah *et al.*, 2007).

The stability or the non-significant difference observe as the concentration increases at this stage, it is expected that the common ion effect must have set in (Yurt *et al.*, 2005).

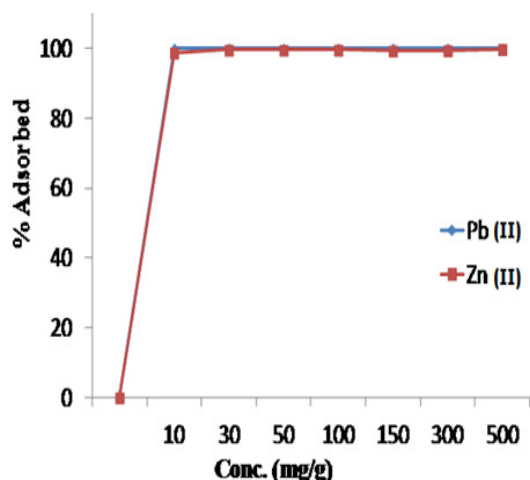


Fig. 3: Effect of concentration on the Pb (II) and Zn (II) ions biosorption on plantain stalk (*Musa paradisiaca*)

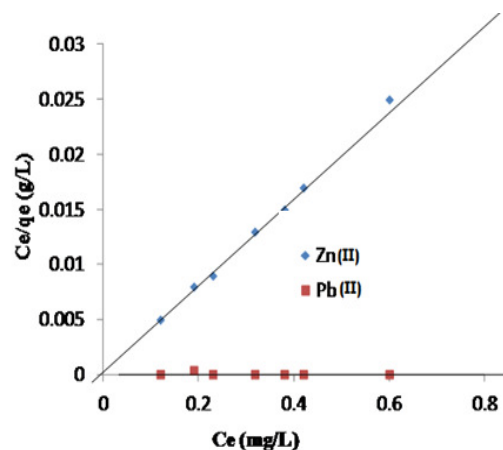


Fig. 6: Linear Langmuir Adsorption isotherm for metal ion removal from aqueous solution by plantain stalk (*Musa paradisiaca*)

Langmuir and Freundlich Isotherm: The data in table 3 showed that Pb(II) ions has the maximum monolayer adsorptive capacity from aqueous solution than Zn (II) ion. This showed that the plantain stalk has a higher mass capacity for Pb (II) than Zn (II) ions. The R² values suggested that the Langmuir isotherm provide a good model for Zn (II) ions than Pb (II) ions of the sorption system. The sorption coefficient K_L which is related to the apparent energy of the sorption, was greater for Zn (II) ions. The sorption capacity q_{max}, of the metal ions investigated on the biomass are of the order Pb(II) (333333mg/g) > Zn(II) (24.6mg/g).

Table 3: Langmuir and Freundlich parameters

	Langmuir Isotherm			Freundlich	
	qm (mg/g)	Kl (L/g)	R ²	K _F	1/n
Pb(ii)	333333	0.150	0.0033	1.00	-0.466
Zn(ii)	24.6	2033	0.998	22.278	0.898

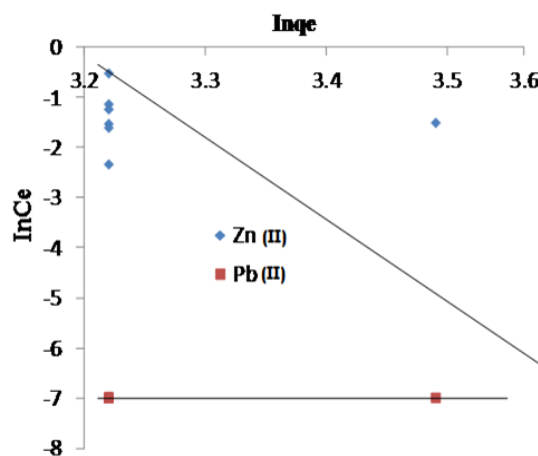


Fig. 7: Freundlich equilibrium isotherm for the sorption of Pb (II) and Zn (II) ions by plantain stalk (*Musa paradisiaca*)

The applicability of sorption process as a unit operation can be evaluated using isotherm models. The equilibrium sorption data obtained were analyzed in terms of Langmuir and Freundlich equations. As shown in table 3 and the plots are presented in figure 6. & 7.

The linear Freundlich isotherm for the sorption of the two metals onto *Musa paradisiaca* are presented in table 3. Examination of the plot (lnq_e Vs lnC_e) reveals that the Freundlich isotherm was an appropriate model for the sorption study of metal ions since the value of the coefficient R² values for Pb(II) ions is 1.000 and not appropriate for the sorption of Zn (II) ion since the R² values for Zn (II) ion is 0.071 on the biomass. The K_F values for Zn (II) ion (22.278L/g) is greater than that of Pb (II) ions (1.00 L/g), this suggest that Zn (II) ions has greater adsorption tendency towards the waste biomass than the Pb (II) ions.

Table 4: Pseudo-first order and second-order rate constant and other kinetic parameter for metal ion removal by plantain stalk (*Musa paradisiaca*) waste biomass

Metal	first order			Second order			
	K ₁	q _e (mg/g)	R ²	h _o (mg/g mn)	K ₂ (mg/g min)	q _e (mg/g)	R ²
Pb (II)	-	0.01	0.2	25.580	0.041	25.1	0.9
	0.0	9	30			30	99
Zn (II)	-	0.41	0.2	29.500	0.048	24.7	0.9
	0.0	2	11			50	99

Table 4 list the resulting parameter obtained using pseudo-first and second-order model, from which the R² values of the first order are 0.230 and 0.211. This did not appropriately described the sorption of the two metal ions onto the biomass, since the values of coefficient of determination R² were all less than 0.999, while that of the second order provide a good

description for the sorption, since the R^2 values are up to 0.999 for the two metals.

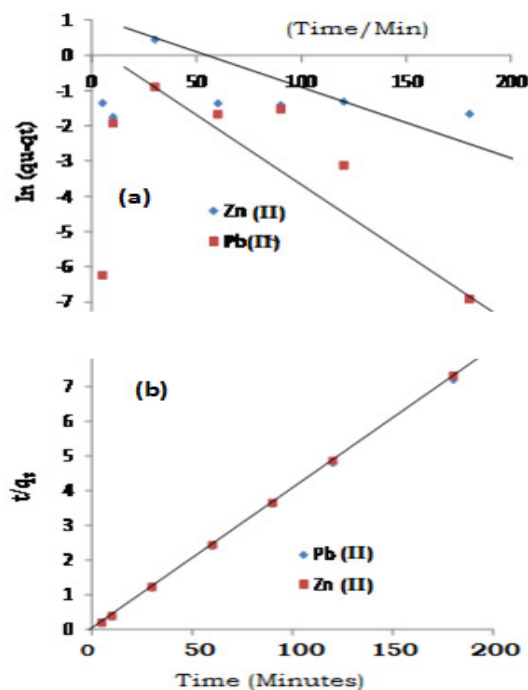


Fig. 8 (a) and (b): Pseudo-first and second-order kinetics for the sorption of Pb (II and Zn (II) ions by (*Musa Paradisiaca*)

From the slopes and intercepts of these curves, K_1 and K_2 and the equilibrium capacity q_e were determined. From the plot, it is observed that the relationship between the metallic ions diffusivity, $\ln(q_e - q_t)$ and time t is nonlinear for Pseudo-first-order plot, showing that the diffusivity of the metal ions onto the biomass surface was film diffusion controlled. Since there is a non-linearity of the diffusivity plot, in describing the reaction among the two metal, the equation proposed was not adequate. From the plot of the second-order, it was observed that the relationship between the metallic ions diffusivity, t/q_t and time t is a linear plot; and as the coefficient of metal ions on the biomass were all equal to 0.999 for the two metals, it is plausible to suggest that the main adsorption mechanism was chemisorptions reaction (Upendra, 2006).

Conclusion: The adsorption processes for the two metals is stable, rapid, there was a significant sorption of metal ions by the adsorbent. The binding capacity of the biomass was observed to be a function of initial pH of aqueous solution. In laboratory scale experiment, the data shown that the plantain stalk has some potential for the removal of metal ions from aqueous solution over a wide range of reaction condition. The FTIR study showed that the main surface functional group responsible for the adsorption

in this biomass were hydroxyl, amine, carboxyl and phosphate groups. The kinetic data clearly showed that the pseudo-second-order model is a more appropriate model for the description of Pb^{2+} and Zn^{2+} onto plantain stalk than pseudo-first order equation.

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