

## Biosorption of Cu(II) Ions from Aqueous Solution using Plantain Stalk (*Musa Paradisiaca L.*)

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<https://doi.org/10.36263/nijest.2020.02.0202>

### ABSTRACT

The study presents the adsorption of Cu (II) ions from aqueous solution by plantain stalk (*musa paradisiaca*) in relation to its equilibrium and kinetics characteristics, using batch adsorption method. The adsorbent was characterized using FTIR Spectrometry. The following functional groups were observed; hydroxyl, carbonyl, amine and phosphate groups which confirms the potential capability of sorption of the biosorbent. The study shows that there was optimum adsorption at pH 6.0 (75.41%) for Cu (II) ions. There was significant adsorption from the lowest to the highest time (5-180mins) and also there is a significant removal of the metal ions from the lowest to the highest concentration (10 mg/l-500 mg/l). The study also revealed that adsorption of Cu(II) ions by plantain stalk from aqueous solution is best described by Langmuir isotherm due to its  $R^2$  values of 0.950. When both order rate equations were tested, data showed a good compliance with the pseudo-second-order rate constant for Cu(II) ions due to its  $R^2$  value of 0.999, while the data for pseudo-first-order did not show a good compliance with Cu(II) ions due to its  $R^2$  value of 0.549. The results obtained from this study show that plantain stalk is a good adsorbent for the removal of Cu (II) ions from aqueous solution.

**Keywords:** Adsorption, Cu(II) ions, Plantain stalk, Kinetic models, Sorption equilibrium

### 1.0. Introduction

Copper is among the major pollutants, which enter the water bodies through various industrial operations such as mining, farming, metal plating etc. According to Environmental Protection Agency (EPA), the permissible level of copper (II) ions in the wastewater is 1.3 mg/l, and its toxicity lead to different diseases such as lung disorders, nausea, liver and kidney failure (Hans and Febe, 2018). When an individual is exposed to copper, it can cause liver damage leading to gastrointestinal problems (Chadrudee *et al.*, 2011). It has also been reported that accumulation of copper into human body causes erythrocyte destruction, diarrhoea, salivation, nausea and muscular cramps, chronic pulmonary problems, renal degradation and skeletal deformity (Jarup *et al.*, 1998). Therefore the reduction of copper concentration from industrial wastewaters, before discharging them into the body of water, becomes necessary (Tharanitharan *et al.*, 2014). There have been several environmental methods used for the removal of metal ions from wastewater. Some of these methods include chemical precipitation, ion exchange reverse osmosis and others. These methods were found to have some disadvantages such as generation of toxic waste, high cost, partial metal removal and requirement of high energy. This necessitates the use of biosorption methods which was found to be less costly, more efficient and does not generate toxic waste.

Many researcher have made use of various plant waste biomass (Horsfall and Abia, 2003; Enemose and Osakwe, 2012; Enemose *et al.*, 2014; Osakwe and Ibrahim, 2017) for adsorption study, but report of studies using plantain stalk as biosorbent for removal of copper ion from waste water is scanty. Plantain is one of the staple foods in Nigeria and the stalks are discarded as waste, littered indiscriminately thereby causing environmental menace. This call for the need to convert the plantain stalks waste biomass into useful products, for the removal of heavy metals ion from aqueous solution

(Eyenubo, 2018). This study aims to investigate how a native agricultural by-product such as plantain stalk can be used for adsorbing heavy metals from aqueous solution. It also intends to propose the modelling kinetics of the biosorption process.

## 2.0. Methodology

### 2.1. Sample collection and preparation

All the chemicals used in this study were manufactured by JHD and they are all of analytical grade. Atomic Absorption Spectrophotometer (AAS), model Philip PU9100x with a hollow cathode lamp and fuel rich flame (air acetylene), was used for heavy metal analysis. Calibration of the instrument was done with spectroscopy grade standard and monitored regularly for the response of the instrument. The batch experiment was conducted in duplicate and the mean for set of value were computed for the experimental accuracy.

The waste plantain stalk biomass was obtained in Abraka community in Ethiope East Local Government Area of Delta State Nigeria, from different dump sites. They were washed thoroughly with deionized water and then cut into pieces. Thereafter, they were dried in the sun for five days and ground into fine particles before they were filtered with 75  $\mu\text{m}$  sieve, and kept in a polythene bag prior to sorption analysis.

The preparation of the aqueous stock solution of  $\text{Cu}^{2+}$  was done following the method of Jimoh *et al.* (2012). About 3.80 g of  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  was dissolved in deionised water with a beaker and then poured into a 1000  $\text{cm}^3$  volumetric flask and was made up to the mark which is 1000 mg/l concentration. It was corked and shaken thoroughly for homogeneity. About 50 mg/l dilution was made from the 1000 mg/l concentration for the pH and contact time experiment. Other subsequent dilution of 10, 30, 50, 100, 150, 300 and 500 mg/l were made for the dependent concentration analysis.

### 2.2. Sample characterization

The adsorbent was characterized using FTIR analysis with FTIR spectrophotometer in the range of 4000 – 500  $\text{cm}^{-1}$ .

### 2.3. Batch biosorption experiment

About 50 mg of the biosorbent was used for the batch biosorption experiment, in which the effect of pH (2-8) analysis was done on sorption of Cu(II) ions from its aqueous solution. About 1M  $\text{HNO}_3$  and 1M  $\text{NaOH}$  was used to regulate the solution to the required pH under study. About 25  $\text{cm}^3$  of the solution of the salt was mixed with the biomass and shaken at 240 rpm for 60 mins. The suspension was filtered using WhatMan Number 45 filter paper and the filtrate was analysed using atomic absorption spectrophotometer (AAS) in duplicate.

Contact time effect on the metal ion binding capacity of the biosorbent was also determined. About 50 mg of the biomass was added to 25  $\text{cm}^3$  of Cu(II) ion aqueous solution in different bottles. The bottles were tightly corked and subjected to shaking at different time of 5, 10, 30, 60, 90, 120 and 180 minutes. The mixtures were then filtered with WhatMan Number 45 filter paper and analysed with AAS. These were done in duplicate.

### 2.4. Kinetics data analysis

The results were subjected to pseudo-first order and second-order kinetic model. From their various equations, plots were made to obtain the correlation coefficient  $R^2$  values to ascertain the appropriate model for the sorption. The slope and intercept were used to determine the kinetic parameter for each model. About 25  $\text{cm}^3$  of the serial dilution of 10, 30, 50, 100, 150, 300 and 500 mg/l were measured out and mixed with 50mg of the biomass in different plastic bottles for the conduction of the effect of concentration of the  $\text{Cu}^{2+}$  binding capacity. It was shaken with a rotary shaker at the optimum time for 240 rpm and then filtered with number 45 WhatMan filter paper and analysed using Atomic Absorption Spectrophotometer.

### 2.5. Data evaluation

During the batch process, the amount of metal ions sorbed by the biomass was calculated using the equation shown below:

$$q_e = \frac{v}{m} (C_0 - C_e) \quad (1)$$

Where:

- $q_e$  amount of metal ion adsorbed on the biomass in mg/g at equilibrium
- $C_0$  initial metal ion concentration in mg/l
- $C_e$  final metal ion concentration in mg/l
- $v$  volume of metal ion solution in litres
- $m$  mass of adsorbent

### 2.6. Adsorption isotherm

Langmuir and Freundlich models were used to fit the experimental data due to their ability of detecting adsorbate that bind to more than one surfaces because of the monolayer and multilayer properties possessed by them. They also have the capability of describing adequately the same set of liquid-solid adsorption data at certain concentration range.

The Langmuir model can be presented as in the equation below:

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

Where:

- $X_m$  and  $K$  are the Langmuir constant and specifically  $X_m$  is the monolayer and sorption capacity of the biomass,  $q_e$  is the concentration of metal ion on biomass (mg/g) at equilibrium.

Linearizing the above equation it becomes:

$$\frac{C_e}{q_e} = \frac{1}{X_m K_L} + \frac{C_e}{X_m} \quad (3)$$

The sorption capacity of the biomass can be obtained by plotting  $C_e/q_e$  against  $C_e$ .

The Freundlich model equation can be written as in the equation below:

$$\frac{X}{m} = K C_e^{\frac{1}{n}} \quad (4)$$

Where:

- $X$  mass of metal ion adsorbed (mg)
- $m$  mass of biomass used (g)
- $C_e$  concentration of metal ion at equilibrium
- $n$  adsorption intensity
- $K$  adsorption constant

Linearizing the equation, it becomes:

$$\ln \frac{x}{m} = \ln K + \frac{1}{n} \ln C_e \quad (5)$$

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

### 2.7. Pseudo-first-order kinetics

The rate law equations are as follows:

$$\frac{dq_t}{dt} = K_1(q_e - q_t) \quad (6)$$

Where:

$q_e$  and  $q_t$  are the amount of each Cu(II) and Cr(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:

$$\ln(q_e - q_t) = \frac{(q_e - k_1^t)}{2.303} \quad (7)$$

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.

### 2.8. Pseudo-second-order kinetics

Applicability of the second order kinetics is tested with the rate equation:

$$\left[ \frac{dq_t}{dt} \right] = k_2(q_e - q_t)^2 \quad (8)$$

Where:

$k_2$  is the rate law for pseudo-second-order biosorption.

On integrating between boundary condition of  $t = 0$ ,  $t=t$  and  $q=0$ ,  $q_e=q_t$ , the following expression was obtained:

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

On linearizing,

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (10)$$

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 model tested for suitability using their correlation of coefficient  $R^2$ .

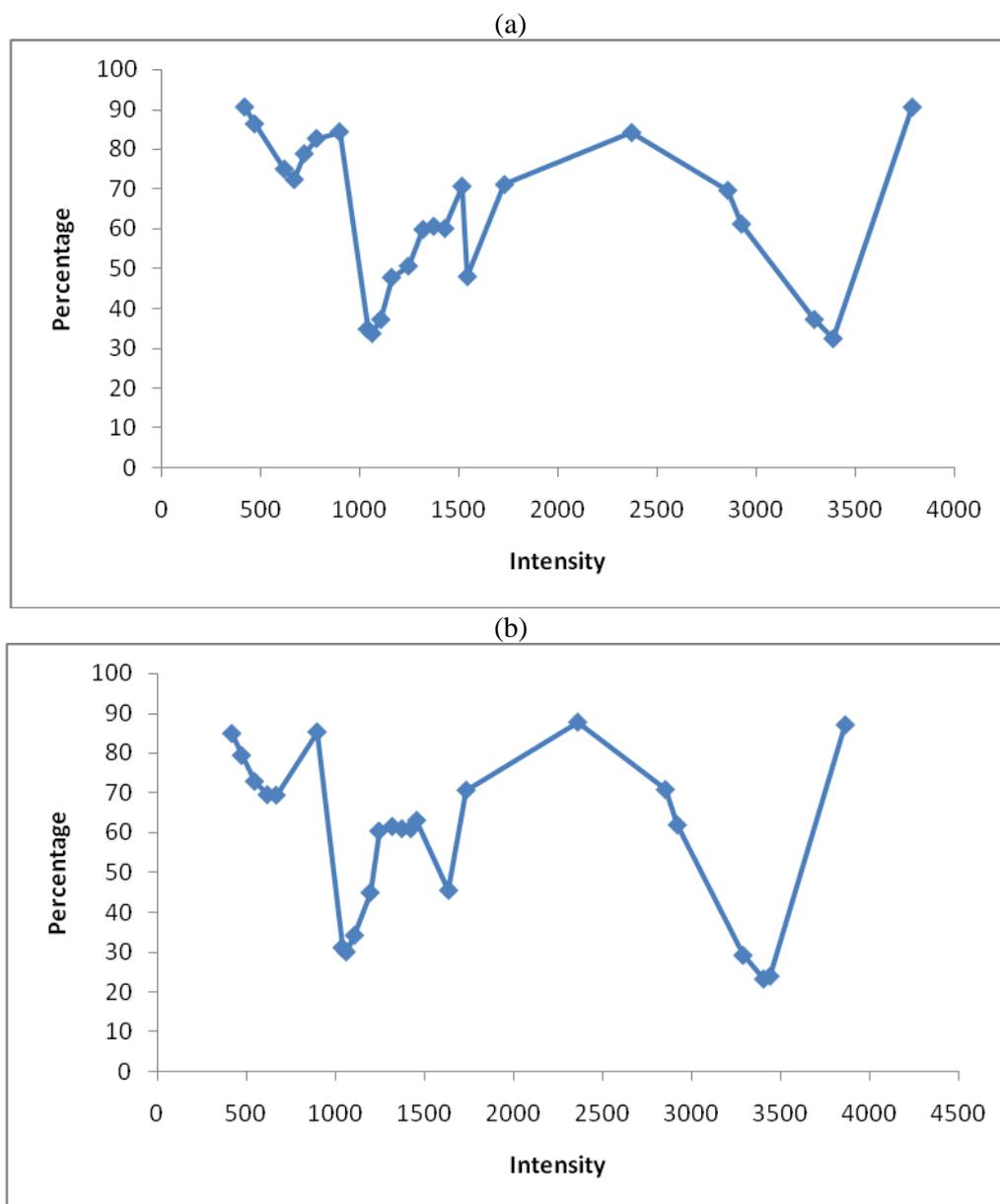
## 3.0. Results and Discussion

### 3.1. FTIR analysis

There is need to understand the metal binding mechanism on the biomass surfaces, hence the analysis of the physical and chemical characteristics of the adsorbent that is presented in Figure 1a and 1b. Table 1 presents the various functional groups of the adsorbent, analysed from its textural characterization, which provide information of the chemical structure of the adsorbent materials by describing the various stretching vibration associated with the adsorbent.

**Table 1:** Textural characterization of plantain stalk (*Musa paradisiaca*) with copper solution at pH 6.0

Wavelength (cm <sup>-1</sup> )	Assigned functional groups
3782.53	O – H stretching vibration
3288.74	N – H stretching vibration
2852.81	C – H stretching vibration
2359.02	– N <sub>3</sub> stretching vibration
1728.28	– CO – O – Aryl and αβ – unsaturated
1641.48	$\begin{matrix} \diagup & & \diagdown \\ & C = C & \\ \diagdown & & \diagup \end{matrix}$ non – conjugated
1105.25	C – O stretching vibration



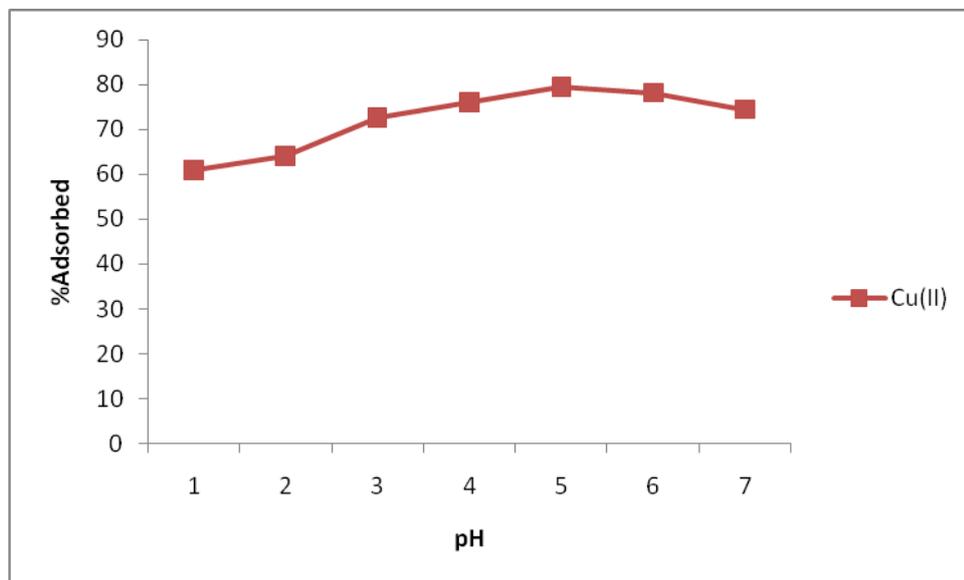
**Figure 1:** The FTIR spectrum patterns of plantain stalk (*Musa paradisiaca*) on copper at (a) pH 6.0 (b) on deionised water at pH 6.0

The Fourier Transformed Infrared (FTIR) analysis showing the various wave numbers were presented in Figure 1a and 1b. From the FTIR spectra, the adsorbent display the presence of ester, alkenes, phosphate, hydroxyl, azide, carbonyl, amine and nitro for the adsorption of the metal at pH 6.0, when it is passed through the metal ions solution and also through the deionized water (i.e. the blank without metal ions solution). And as it can be seen from the plot, there is no much difference between the two spectra.

The confirmation of carboxylic acid group present as a result of the presence of hydroxyl and carbonyl group with the presence of amine group is important sorption site (Volesky, 2003; Eyenubo, 2018).

### 3.2. Effect of pH

The effect of pH is one of the essential parameter that affects the adsorption of heavy metals with biomass from aqueous solutions. The percentage variation of Cu(II) ions sorbed at different pH by *Musa paradisiaca L.* is displayed in Figure 2.



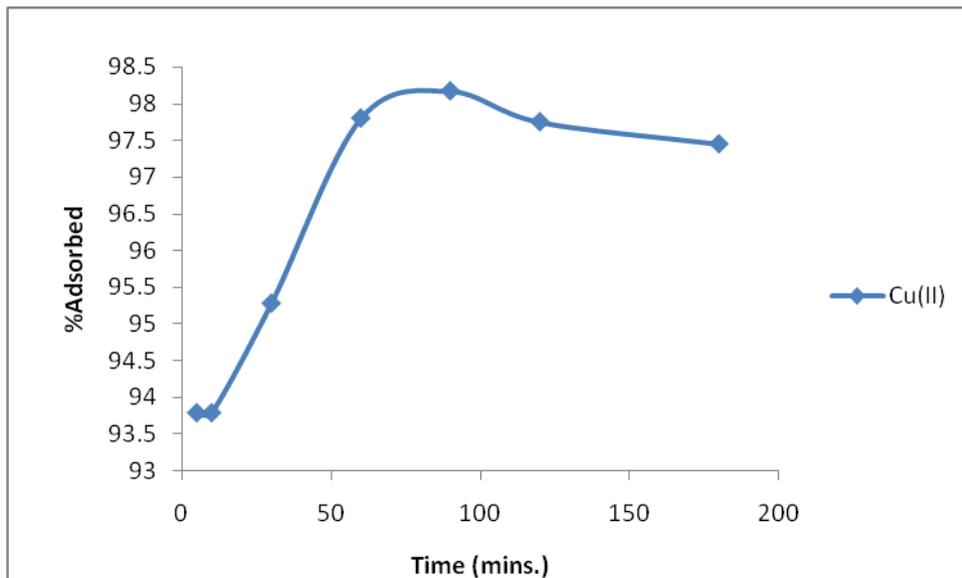
**Figure 2:** Effect of pH on Cu(II) ions biosorption on plantain stalk

Figure 2 shows the sorption of Cu(II) ion onto plantain stalk as biosorbent. The data in the plot indicate that there was a rapid removal of the metal ions by the biosorbent at the lowest pH of 2.0; 61.0 of Cu(II). The result therefore indicate that weak acidic pH favour the sorption of Cu(II) ions onto the plantain stalk, this may be attributed to the presence of sufficient  $H^+$  ions in the solution which compete with the metal ions and are tightly held to the negatively charged active site on the biosorbent, creating less available site for the other cations. Similar observations were reported by Osakwe and Ibrahim (2017). Maximum adsorption efficiency for Cu(II) ion was observed at pH 6.0 ion.

As can be seen from the plot, metal ions sorption increase with increase in pH i.e. from pH 2.0 – 6.0 for Cu(II) ions where the maximum pH was observed. Similar results were reported by some other researcher in similar studies (Enemose and Osakwe, 2012; Pandey *et al.*, 2014). Idowu *et al.* (2016) suggested that the lower removal efficiency of  $Cu^{2+}$  at low pH could be attributed to surface protonation leading to repulsion of metal ions or competition between  $H^+$  and the metal ions for appropriate sites on the adsorbent surface. This result can also be explained from the fact that as the pH increased, the competition between the  $H^+$  and the copper ions at low pH weakens and eventually the metal ions will replace the  $H^+$  ion bound to the adsorbent (Raju *et al.*, 2012). However, as the pH increases further to pH 7.0 and 8.0, there was a decrease in the metal ion sorbed (78.09 and 74.45 respectively).

### 3.3. Effect of contact time

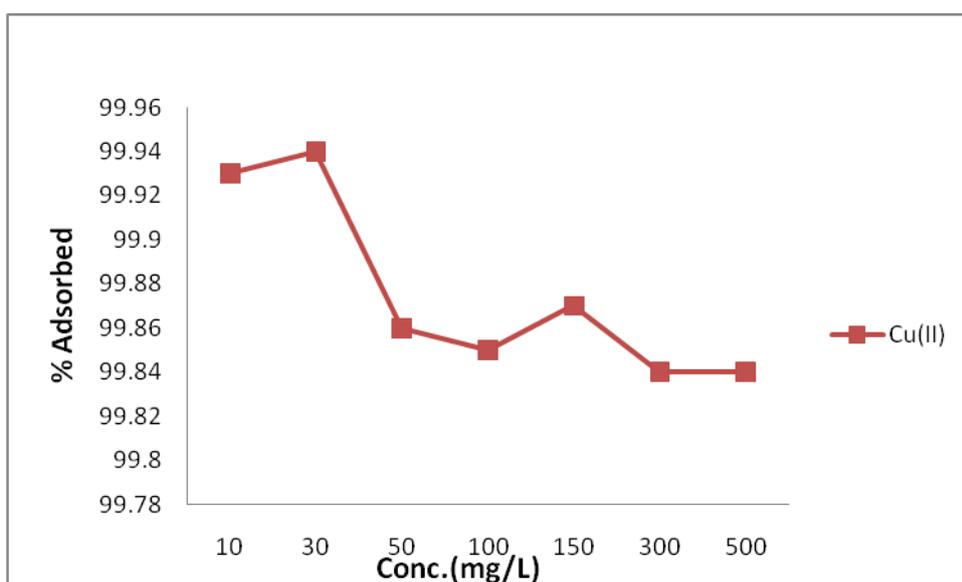
The results presented in Figure 3, shows the rate at which Cu(II) ions were sorbed onto plantain stalk, which indicate percentage adsorbed against time  $t$ . There was a significant amount of Cu(II) and Cr(II) ions sorbed by the adsorbent in all the times investigated, with a little difference between them. For 5 mins, 93.73 and 98.77 %; for 10 mins, 93.79 and 100 %; for 30 mins, 95.28 and 99.96; respectively. This may be as a result of the presence of sufficient reactive sorption site on the biomass cell wall (Dauda *et al.*, 2015; Osakwe and Ibrahim, 2017). Cu(II) ion removal gradually increased from 5-90mins where it obtained its maximum sorption and then slightly decreased at 120 and 180mins; 97.75 and 97.45 respectively.



**Figure 3:** Effect of contact time on Cu(II) ion sorption on plantain stalk

### 3.4. Effect of concentration

The plot of the result of percentage adsorbed against concentration on Figure 4, display the adsorption rate of Cu(II) ions onto the plantain stalk. The data display various adsorbate concentrations, from 10, 30, 50, 100, 150, 300 and 500 mg/l. From the data, it was observed that there was maximum sorption from the lowest to the highest concentration, which may be as a result of mass transfer which is the concentration gradient (Pandey *et al.*, 2014). The non-significant differences observed in the percentage adsorbed in Cu(II) ions, might be a result of common ion effect which must have set in (Yurt *et al.*, 2003). It may also be as a result of the large amount of metal ions ready for competition on the binding site. There was no significant difference in the percentage adsorbed even up to the highest concentration of 500 mg/l, rather there was a little decrease indicating saturation of all binding site on the adsorbent surfaces beyond a particular concentration (Ali *et al.*, 2014). It can also be seen from the plot that the amount of Cu(II) ions adsorbed onto the plantain stalk decreased with increase in concentration of the adsorbate. The highest percentage of Cu(II) ions adsorbed was about 99.94% at 30 mg/l concentration by the adsorbent.



**Figure 4:** Effect of concentration on Cu(II) ion sorption on plantain stalk

The sorption equilibrium data obtained were analysed using Langmuir and Freundlich isotherm. From the data in Table 2, Langmuir proved a better isotherm in Cu(II) ion due to its  $R^2$  value of 0.950. It

may also be due to the homogeneous distribution of active sites onto the plantain stalk (Hans and Febe, 2018).

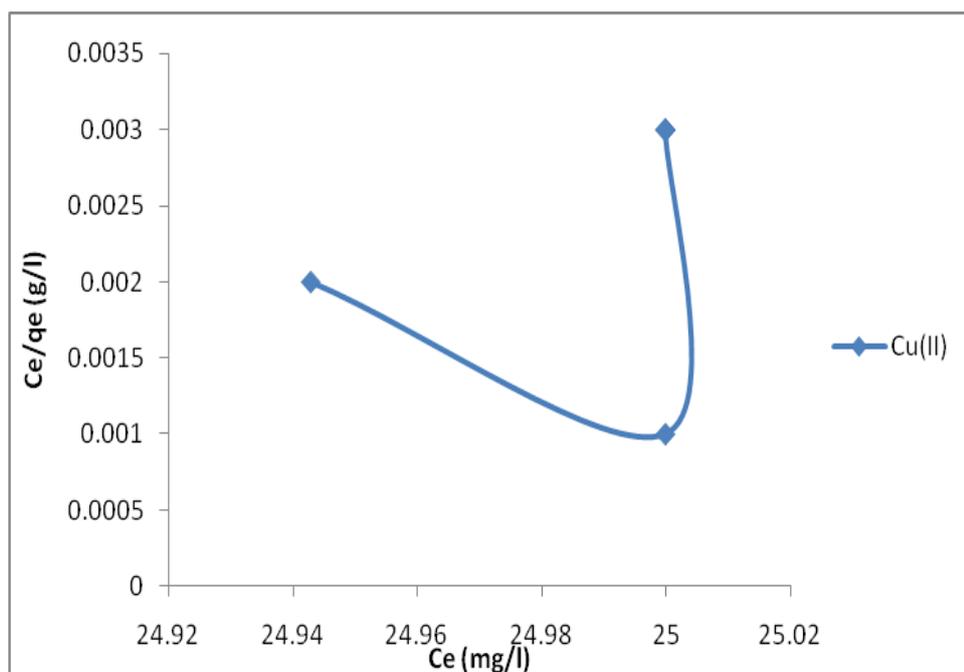
The data also showed a high adsorbent of 26.5 for the Cu(II) ion which is the mass capacity. Also, the metal had a sorption coefficient  $K_L$  of 189, which is the apparent energy for sorption.

From the data in Table 1, the  $K_f$  values of Cu(II) is 1.391. This indicates that it has a higher adsorptive capacity on the waste biomass since it is above unity. The  $R^2$  values of Cu(II) ion is 0.883, since the value is not up to 0.999, Freundlich isotherm is not too appropriate to describe the model. The result shows that plantain stalk is a good adsorbent for the sorption of Cu(II) ions from aqueous solution.

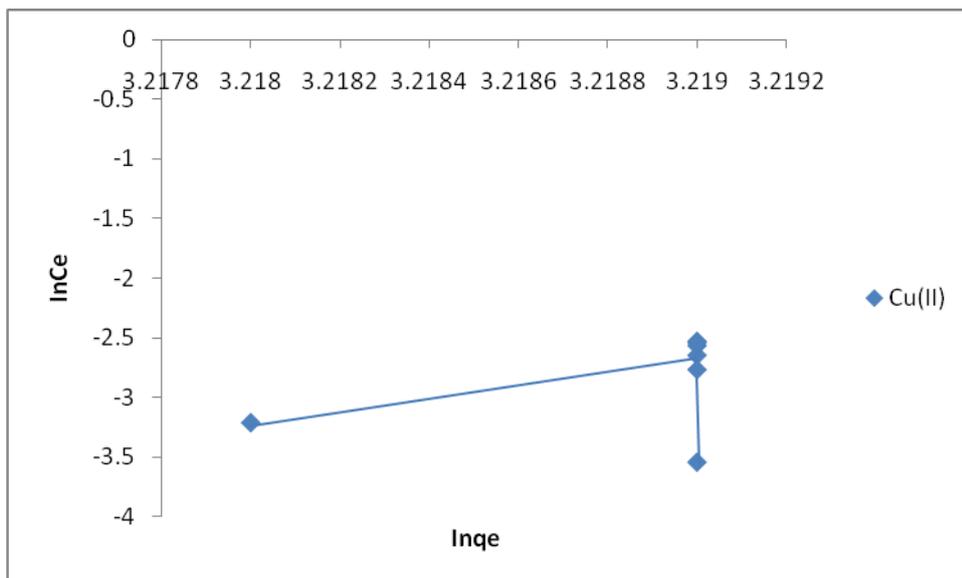
**Table 2:** Langmuir and Freundlich parameters

C			Freundlich isotherm		
$q_m$ (mg/g)	$K_L$ (L/g)	$R^2$	$K_F$	1/n	$R^2$
Cu (II) 26.5	189	0.950	1.391	-1.004	0.883

Figures 5 and 6 display the application of sorption patterns as a unit process determined with isotherm models. The equilibrium data obtained were analysed using Langmuir and Freundlich equations as it can be seen in Table 2 and the plots presented in Figures 5 and 6. Critical examination of the plot [ $C_e$  (mg/l) vs  $C_e/q_e$  (g/l)] via calculation of the slope and intercept of the curve, it shows that Langmuir isotherm was the appreciated model for the sorption of Cu(II) ions onto plantain stalk, since its  $R^2$  value is 0.950.



**Figure 5:** Linear Langmuir adsorption isotherm for Cu (II) ion removal from aqueous solution by plantain stalk



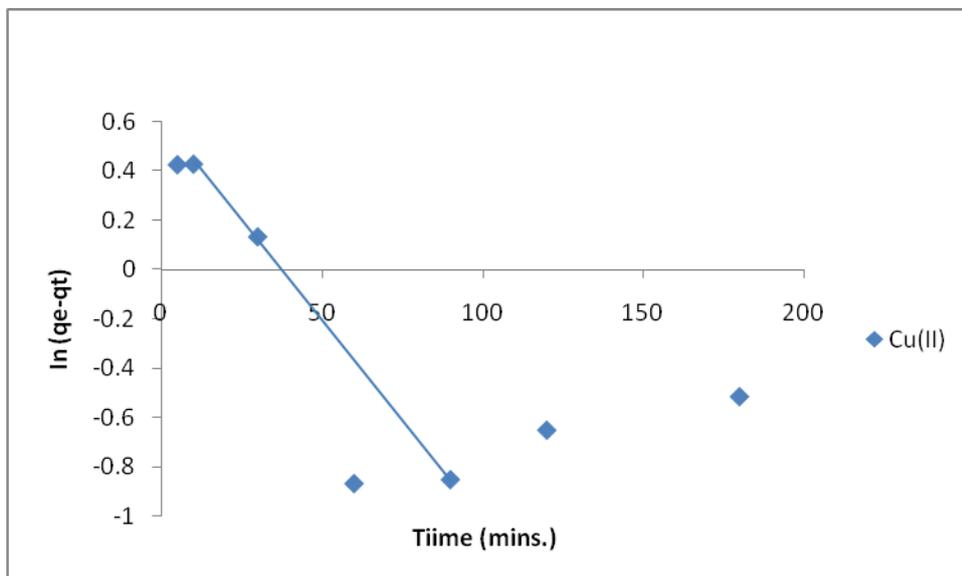
**Figure 6:** Freundlich equilibrium isotherm for the sorption of Cu (II) ions by plantain stalk

Table 3 displayed the resulting parameter obtained using pseudo-first and second order model, in which the  $R^2$  values are lower than 0.999 for the metal in first order 0.549 and in second order, the  $R^2$  value of Cu(II) ion is 0.999. This indicate that the model is more appropriate to describe the sorption of Cu(II) ion in second order and also is not appropriate in first order since the  $R^2$  value is not up to 0.999. This also shows that the predominant process is chemisorption, which involves the sharing of electron between the biomass surface and the metal ions (Hans and Febe, 2018).

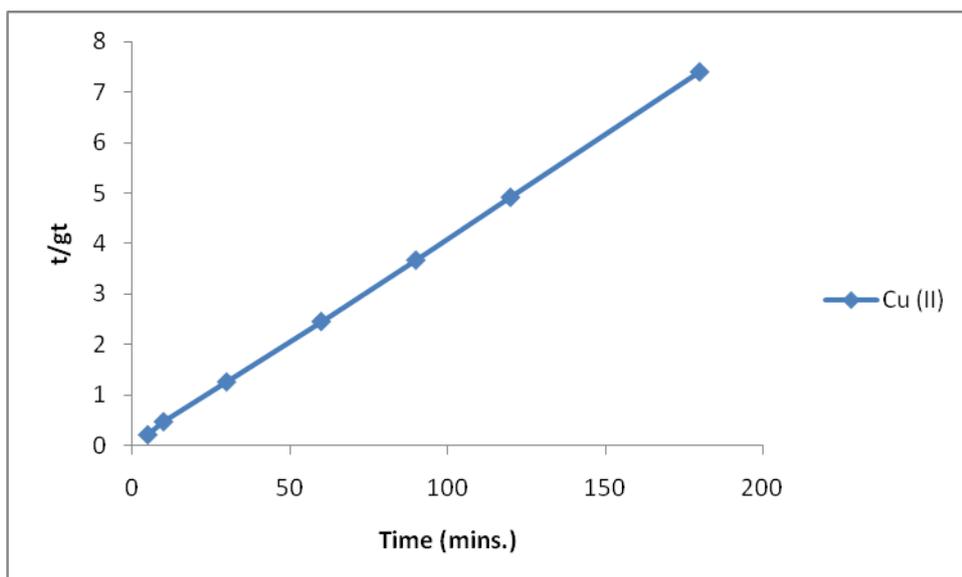
**Table 3:** Pseudo-first and second order kinetic parameter for Cu (II) ions removal by plantain stalk.

First order				Second order		
Metal	$K_L$	$q_e(\text{mg/g})$	$R^2$	$K_2$	$q_e(\text{mg/g})$	$R^2$
Cu(II)	-0.006	1.168	0.549	0.043	24.570	0.999

From Figures 7 and 8, the slope and intercept of these curves  $k_1$  and  $k_2$  and the equilibrium capacity were determined and it was observed that there is non-linearity in the metallic ion diffusivity, i.e.  $\ln(q_e - q_t)$  and time  $t$  for the plot, this shows that the diffusivity of the metal ion onto the biomass surface was film diffusion controlled (Eyenubo, 2018). Specifically, from the plot of the second order (fig.8), it was observed that the relationship between the diffusivity of the metallic ion,  $t/q_t$  and time  $t$  is a linear plot. As a result of non-linearity, the equation proposed was not appropriate.



**Figure 7:** Pseudo – first order kinetics for the sorption of Cu(II) ions by plantain stalk



**Figure 8:** Pseudo – second order kinetics for the sorption of Cu(II) ions by plantain stalk

#### 4.0. Conclusions

The process of adsorption of the metal ions is stable, fast, and a good percentage of the metal ions were absorbed by the adsorbent at initial pH, contact time and concentration, there were rapid absorption of metal ions. From the FTIR spectra, it was found that the adsorbent displayed the presence of ester, alkenes, phosphate, hydroxyl, azide, carbonyl, amine and nitro for the adsorption of the metal at pH 6.0. From the laboratory analysis, the data obtained showed that the plantain stalk has a good capacity of removing metal ions over a broad range of reaction condition. It also proves that linear Langmuir isotherm and pseudo second order kinetic are more appropriate for the description of the sorption of Cu(II) ion onto plantain stalk.

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**Cite this article as:**

Eyenubo O. B., Peretomode O. and Osakwe S. A., 2020. Biosorption of Cu(II) Ions from Aqueous Solution using Plantain Stalk (*Musa Paradisiaca L.*). *Nigerian Journal of Environmental Sciences and Technology*, 4(2), pp. 226-236.<https://doi.org/10.36263/nijest.2020.02.0202>