Chemical Modification of Pseudo stem of *Musa Paradisiaca l.* in Treating Copper(II) ions from Wastewater and Isotherm Studies

Fazlina Roslan, Sitinoor Adeib Idris

Faculty of Chemical Engineering, Universiti Teknologi MARA

Abstract—**The biosorption of Cu(II) from dilution of wastewater by using chemically modified Pseudo stem with sodium hydroxide (NaOH), calcium chloride (CaCl) and ethanol (EtOH) was investigated. Atomic absorption spectrophotometer (AAS) method was established to determine the final concentration of Cu(II). The batch experiments showed that biosorption efficiency was dependent on the initial metal ions concentration, contact time and dosage of biosorbent. The optimum pH and temperature for the removal of Cu(II) was 5.9 and 27° C respectively. The maximum adsorption of Cu(II) ions was 79% or 77.17 mg/h for 75mg/l of initial Cu(II) concentration. The optimum time of Cu(II) biosorption was within 60minutes. The results of experiments were analysed using three adsorption isotherm models which are Langmuir, Freundlich and Redlich-Peterson. Thus, the Redlich-Peterson model was the best isotherm to represent the data with R^2 value of 0.988. Heavy metals in the wastewater are classified as non-biodegradable and can give serious health disorder and environment which can cause anaemia, respiratory effect, bad smell and cancers.**

Keywords— Adsorption, Biosorption, Heavy metals, Chemical modification, Lignocellulose waste, Industrial wastewater.

I. INTRODUCTION

Metal contamination is considered to be one of the most ubiquitous and complex environmental today. Industrial activities produced wastewater may contain several of toxic metals such as Cadmium (Cd), Lead (Pb), Copper (Cu), Zinc (Zn), Nickel (Ni) and Chromium (Cr) in different concentration. The term 'heavy metal' is collectively applied to a group of metals with density greater than 5 g/cm^3 and atomic number above 20 (Charif Gakwisiri *et al.* 2012). Hence, the density and atomic number of copper are 8.96 g.cm-3 at 20°C and 29 respectively (Chemical Properties of Copper, 1996). A large volumes of wastewater was discharged from electroplating industries and contains a lot of heavy metal ions which are Pb(II), Cu(II), Ni(II), Cr(II) and Cd(II) ions. These metals are not biodegradable and very hazardous to the human health and environment which can cause respiratory effect, bad smell and cancers. Several methods that have been introduced for treating heavy metal ions are adsorption, ion exchange and chemical precipitation. However, adsorption method has many advantages over other methods such as recovery of metal value, selectivity, sludge free operation, cost effectiveness and meeting of strict discharge specifications.

Banana stem or Pseudo stem of *Musa Paradisiaca l.* is a commonly available and abundant natural adsorbent in both subtropical and tropical region in the world (Vinegswaran *et al*, 2015). It also has high adsorption capacity due to the organic composition in the stem like cellulose, hemicellulose and lignin (Li *et al*, 2010). However, the adsorption capacity of Pseudo stem can be enhance the capacity by chemical and physical treatment. Thus, chemical modification of bio-sorbents can make its surface become more stabilize, prevent organic leaching, create new adsorption site and protonation (Yargic *et al.* 2014), (Jimenez *et al*. 2013).

The aim of the study is to evaluate the effectiveness of chemically modified banana stem with sodium hydroxide, calcium chloride and ethanol in adsorption of Copper(II) ions from wastewater. The effect of parameters such as the initial metal concentration, adsorbent dose and contact time between adsorbent and adsorbate to the adsorption process was studied. The adsorption isotherm models, namely Langmuir, Freundlich and Redlich-Peterson were also investigated to know the adsorption characteristics.

II. METHODOLOGY

A. Pre-treatment of adsorbent

The Pseudo stem was collected, washed, dried and sieved to 100 mm size. The dried Pseudo stem were chemically treated with the combination of sodium hydroxide (NaOH) with calcium chloride (CaCI) and ethanol (EtOH). The amount of NaOH, CaCI and EtOH were 280, 280 and 540 ml respectively. Then, 11 g of banana's particle was soaked in 1.1 L of the chemical mixture solution and placing it on the orbital shaker with speed of 155 rpm for 24 hours at room temperature. Next, the chemically modified bio-sorbent were filtered and rinsed with distilled water to remove any free chemicals until the neutral pH value was 5.9. After that, it is dried at 80°C of temperature in the drying oven for 24 h. All chemically modified banana stem were kept in a desiccator for a while before it used for the future experiments.

B. Stock solution of metal ions-Copper(II) ion solution

Cu(II) ion solution was used as the stock solution in the current study. The stock solution (196mg/l) of Cu(II) ion has been taken from electroplating industrial. The dilution of stock solution must be appropriate in order to obtain the desired concentration of Cu(II) solution used later in the experiment. Hence, the concentration of Cu(II) solution were prepared between 75, 100, 125, 150 and 175 mg/l.

C. Batch adsorption experiments

Batch experiments were carried out at room temperature and pH value of 5.9 (Noeline, Manohar et al. 2005). A mixture of 0.1 g of banana stems powder and 50 ml aqueous Cu(II) solution of desired concentration range between 75 and 175 mg/l were placed in the 200 ml Erlenmeyer flask. The Erlenmeyer that contained mixture of Cu(II) solution with chemically modified banana stem powder were shake by an orbital shaker for 1 hour until equilibrium was reached with the speed of 155 rpm. All samples were carried out in duplicate under the same conditions and the average results were taken. Once the equilibrium achieved, the contents of the flask were filtered through filter paper and final concentration of Cu(II) was measured using atomic absorption spectrophotometer(AAS).

The effect of varying contact time range between 10 to 60 minutes and amount of bio-sorbent were studied. Three adsorption isotherm models were examined in the experiment. Uptake of Cu(II) ions and amount of Cu(II) ions adsorption (mg/g) were calculated by the Equation 1 and 2 respectively.

$$\text{Uptake } \% = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1) \text{ (Aloma } et\ al. \text{ 2012)}$$

$$q_e = \frac{C_0 - C_e}{m} V \quad (2) \text{ (Aloma } et\ al. \text{ 2012)}$$

where Co is the initial concentration of Cu(II) (mg/L), Ce is the concentration of Cu(II) at equilibrium state (mg/L), m is the amount of adsorbate used (g), and v is the volume of the solution (l).

All the bio-sorption experiments undergo in duplicate and the average values are calculated.

D. *Adsorption isotherm models*

Isotherm models are used to predict the maximum adsorption capacity of a sorption system. It helps to analyses of the feasibility of the treatment process for specific application, selection of most suitable sorbent and the require amount of bio-sorbent. Langmuir, Freundlich and Redlich-Peterson isotherm model are the three of another isotherm model that are most commonly used in research study.

Langmuir model

The Langmuir model (1918) assumes that the metal ion removal occurs on homogeneous (uniform) surface by monolayer adsorption. The adsorption does not involved ant interaction between adsorbed metal ions. The formulae of the Langmiur models are shown in Equation (3).

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_F} \quad (3) \text{ (Feng et al. 2014)}$$

Equation (3) can be expressed in its linear form as:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m} \quad (4) \text{ (Feng et al. 2014)}$$

where q_e is the amount of Cu(II) adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of Cu(II) (mg/l), q_m is the maximum amount of adsorbed Cu(II) per unit mass of biosorbent (mg/g), K_L is the Langmuir constant related to the adsorption energy.

Freundlich model

In 1906, Freundlich presented the earliest known sorption isotherm equation. It is based on the equilibrium sorption on heterogeneous surfaces. The Freundlich equation is represented as below:

$$q_e = K_F C_e^{1/n} \quad (5) \text{ (Feng et al. 2014)}$$

Equation (5) can be expressed in its linear form as:

$$\text{Ln} q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (6) \text{ (Feng et al. 2014)}$$

where q_e is the amount of adsorbed Cu(II) per unit mass of biosorbent, C_e is the equilibrium concentration of Cu(II) (mg/l), K_F (l/g) and 1/n are the Freundlich constants related to adsorption capacity and sorption intensity respectively.

Redlich-Peterson model

Jossens and co-workers modified the three parameter isotherm first proposed by Redlich and Peterson (1959) to incorporate features of both the Langmuir and Freundlich isotherm into single equations (Jossens *et al.* 1978). At low concentrations the Redlich-Peterson isotherm approximates to Henry's law and at high concentrations its behaviour approaches that of the Freundlich isotherm. The formulae of Redlich isotherm model are shown in Equation (7).

$$q_e = \frac{K_R C_e}{1 + \alpha_R C_e^g} \quad (7) \text{ (Feng et al. 2014)}$$

The linear form isotherm can be expressed as follows

$$\ln \frac{C_e}{q_e} = g \ln C_e - \ln K_R \quad (8) \text{ (Feng et al. 2014)}$$

where K_R and α_R (L/mg) are the Redlich-Peterson isotherm constant and g is the exponent between 0 and 1. Two limiting cases were involved in this isotherm: value of g (Henry's law) = 0 while value of g (Langmuir) = 1.

III. RESULTS AND DISCUSSION

A. *Effect of initial metal concentration*

The initial metal concentration was one of the important aspects that determine the biosorption efficiency of metal. It provides an important driving force to overcome the resistance to the mass transfer of Cu(II) between solid and aqueous phase. The effect was studied in batch adsorption experiments at constant time (1hour), agitation speed (170 rpm), pH of Cu(II) ions solution (5.9), bio-sorbent dosage (0.1g) and room temperature (27℃) using different initial concentrations (75,100,125,150 and 175 mg/l).

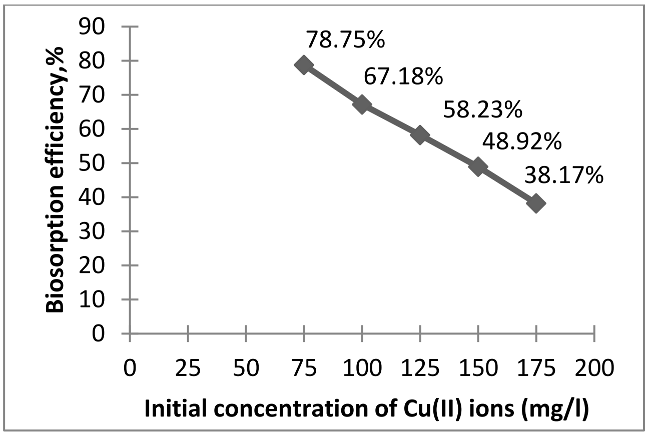

Figure 1: Effect of initial Cu(II) concentration on the bio-sorption efficiency of Cu(II).

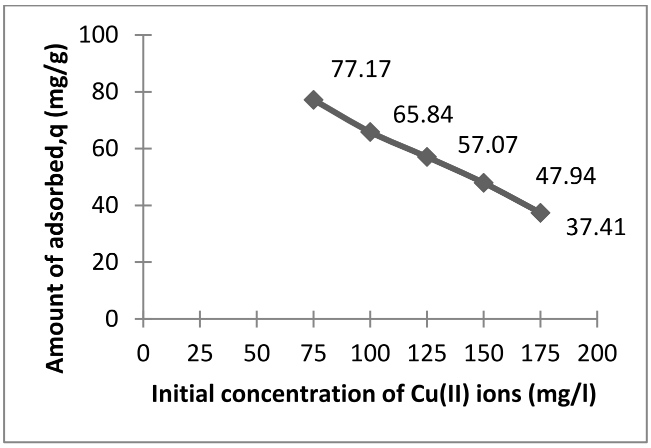

Figure 2: Effect of initial Cu(II) concentration on the amount of adsorbed Cu(II).

As seen in Figure 1 and 2, the bio-sorption efficiency of Cu(II) ions and the amount of Cu(II) ions adsorbed per unit mass of bio-sorbent respectively were decreased steadily when the initial of Cu(II) concentration increased. Thus, the increasing of initial Cu(II) ions concentration from 75 to 175 mg/l will resulted to the decreasing both of the amount of Cu(II) ions adsorbed per unit mass of bio-sorbent (77.17 to 37.41 mg/g) and the biosorption efficiency (78.75 to 38.17%) respectively. This is happened due to the fact that the number of vacant site on the biosorbent surface was limited. Moreover, ratio of Cu(II) moles initial number to the available adsorption site was higher that can lead to the lower biosorption efficiency. According to the (T.Fatima et al. 2013), more left un-adsorbed of higher Cu(II) ions in solution due to the saturation of binding site and take up of the bio-sorbent particles became sluggish. Decrease in driving force of the concentration gradient to overcome resistance of all mass transfer of Cu(II)ions between aqueous and solid phases was also one of the factors that contribute to the decrease of biosorption efficiency.

B. Effect of contact time

The effect of contact time on the biosorption efficiency of metal was studied in the batch adsorption experiment. It was studied in constant agitation speed (170 rpm), pH of Cu(II) ions solution (5.9), bio-sorbent dosage (0.1g) and room temperature (27℃) an initial concentrations of Cu(II) (75 mg/l). The differences of contact time were 10, 20, 30, 40 and 50 minutes.

Based on the Figure 3, it shows that the biosorption efficiency of Cu(II) ions was increased with increasing the contact time until optimum contact time was reached at 60 minutes. After 60 minutes, the biosorption efficiency of Cu(II) decreased and not significantly effective.

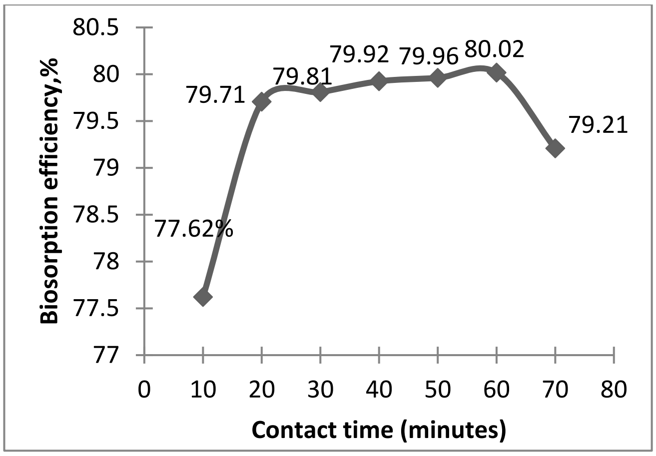

Figure 3: Effect of contact time on the bio-sorption efficiency of Cu(II).

The two-phases metal uptake process occurred as reported in previous reports on the bio-sorption of heavy metals by bio-sorbent (Aloma et al., 2011). From Figure 4.3, there were two stages involved on the biosorption capacity of Cu(II) ions.The first stage happens in the first 20 minutes, the biosorption efficiency was increased rapidly with more than 78 % took place. After 20 minutes, the biosorption capacity of Cu(II) ions become slower until achieved the equilibrium time (60 minutes). According to Sathasivam (2010), the equilibrium time was achieved at 1 hour.

The binding sites on the biosorbent were more available in the first 20 minutes. Gradual occupancy of these sites within 20 minutes allowed faster binding of Cu(II) ions on the biosorbent. In addition, ion exchange followed by a slow chemical reaction of the metal ions with active functional groups on the wastewater sample will make the faster adsorption (Gin *et al.* 2014). After 20 minutes, there are more competition among of Cu(II) ions occurred to get available sites on the bio-sorbent compare in the first 20 minutes.

Nevertheless, after 60 minutes the percentage of Cu(II)ions biosorption decreased and not significantly effective as result of all the bio-sorbent's sites have been filled by Cu(II) ions and also accumulation of heavy metal ions species. Moreover, formation of repulsive forces between the heavy metal ions on the bio-sorbent were occurred when the majority of the binding site occupied by metal ions make it to be harder binding to the remaining vacant surface sites. In fact, the metal ions need to pass through the deeper pore surfaces of bio-sorbent which make the substantial resistance leading to adsorption decreased as well as the rate of ion transportation from outer to the inner of bio-sorbent sites actually determines the rate of adsorption of later phase (Arunakumara et al., 2013). Further increase in contact time also had insignificant effect on the amount of metal ions adsorbed (Nurzulaifa et al., 2015). Thus, the effect of contact time was one of the important parameters required for the wastewater treatment system.

C. Dosage of biosorbent

The effect of bio-sorbent dosage on the Cu(II) removal was investigated and has been shown in Figure 4. The graph indicates that the removal of Cu(II) increased from 76 to 87% with the increasing of bio-sorbent dosage(100 to 500 mg) while keeping other parameters such as pH (5.9), room temperature (27˚C), initial concentration of Cu(II) ions (75 mg/l), agitation time (1 hour) and agitation speed (170 rpm) constant.

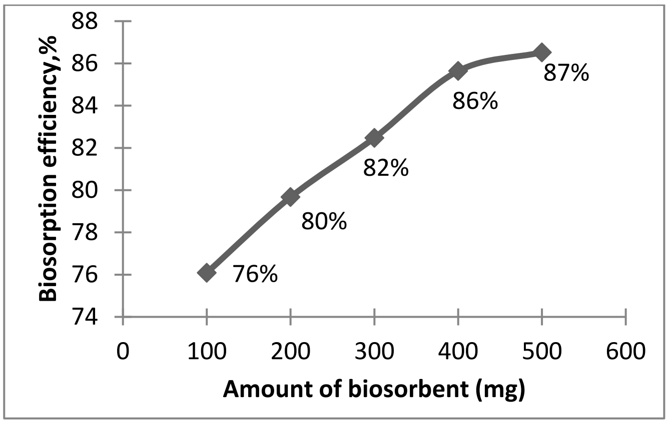

Figure 4: Effect of biosorbent dosage on the bio-sorption efficiency of Cu(II).

It can be observed that the bio-sorption efficiency of Cu(II) ions normally increased gradually by increasing biosorbent dosoge. Higher amount of bio-sorbent dosage will be effected by the partial aggregation among the available of the bio-sorbent binding sites and bigger contact surface area of bio-sorbent with Cu(II) ions metal (Arunakumara *et al.* 2013), thus increased the biosorption efficiency of Cu(II) ion and it will leads to the better adsorption. However, further increasing of bio-sorbent dosage probably (> 500 mg) not significantly affected on the removal of metal ions by reason of reaching of maximum uptake (equilibrium stage) of Cu(II) ions, overcrowding of adsorbent particles (Nurzulaifa *et al.* 2015) as well as the amount of heavy metal ions bound to the bio-sorbent or amount of residual ions in solution remains constant.

D. Adsorption isotherm models

Langmuir model

The plots of specific sorption (C_e/q_e) against the equilibrium concentration (C_e) for Cu(II) ions are shown in Figure 6 and their linear isotherm parameters, q_m, K_L and coefficient value(R^2) are presented in Table 1.

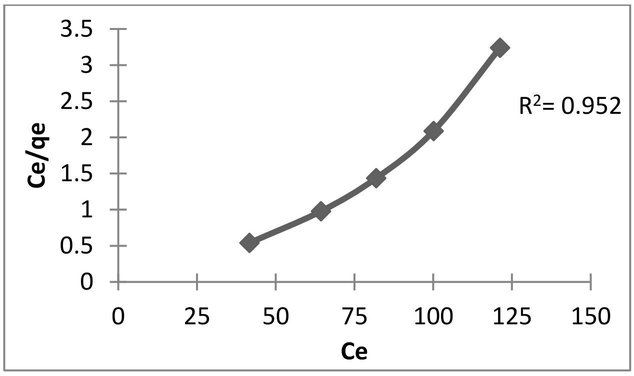

Figure 5: Langmuir equilibrium isotherm model for the bio-sorption of Cu(II) onto Pseudo stem.

Table 1: Parameters of Langmuir isotherm.

q_{max} (mg/g)	K_L (l/mg)	R^2
29.94	0.031	0.952

If the values of K_L are less than unity (K_L<1) for all metals adsorption means that the higher affinity of heavy metal adsorption (Anwar *et al.* 2010). Hence, the K_L value of Cu(II) ions is 0.031 l/g which is less than 1 shows that the strong affinity of Cu(II) ions adsorption.

The maximum predicted adsorption capacity of Cu(II) ions using chemically modified Pseudo stem is 29.94 mg/g.

Freundlich model

The Freundlich isotherm for the Cu(II) ions are presented by plotted of $\ln q_e$ against the $\ln C_e$ was shown in Figure 6 and their linear isotherm parameters, K_F, 1/n and coefficient value(R^2) are presented in Table 2.

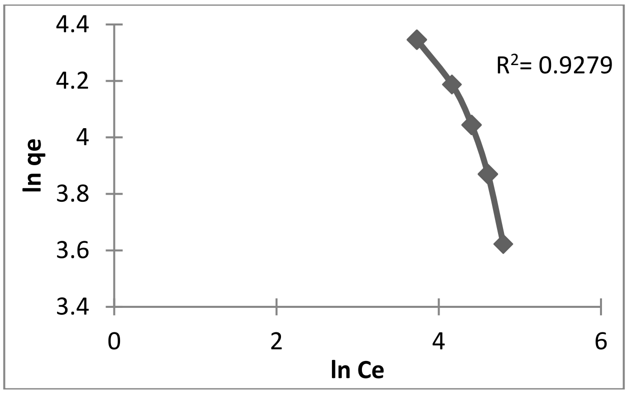

Figure 6: Freundlich equilibrium isotherm model for the bio-sorption of Cu(II)ions onto Pseudo stem.

Table 2: Parameters of Freundlich isotherm.

K_F(l/mg)	1/n	R^2
0.939	0.6525	0.9279

The Freundlich parameter n is a measure of intensity of metal biosorption by the biosorbents. When the value of n >1 or 0.1<1/n<1.0 it means that the bio-sorption of heavy metal ions on the bio-sorbent is practically favourable. However, the bio-sorption capacity was good adsorption if the value of n in the range (1-10) or greater than unity (T.Fatima *et al.* 2013). In this study, the n value is 1/n is 0.6525 which is less than 1 thus, the Cu(II)ions is favourable on the chemically modified Pseudo stem.

The adsorption capacity for Freundlich constant, K_F values are higher than the magnitudes of experimental and predicted capacity of the Langmuir isotherm, K_L and K_R value of Redlich-Peterson. The value of K_F is 0.939 l/mg was higher than (K_L=0.031 l/mg) and (K_R = 0.001 l/mg). Hence, Freundlich model has greater tendency towards the chemically modified Pseudo stem of Cu(II) ions and easier in biosorption.

Redlich-Peterson model

The Redlich-Peterson isotherm plot for the Cu(II) ions are presented in Figure 9 and the parameters of isotherm have been given in Table 3.

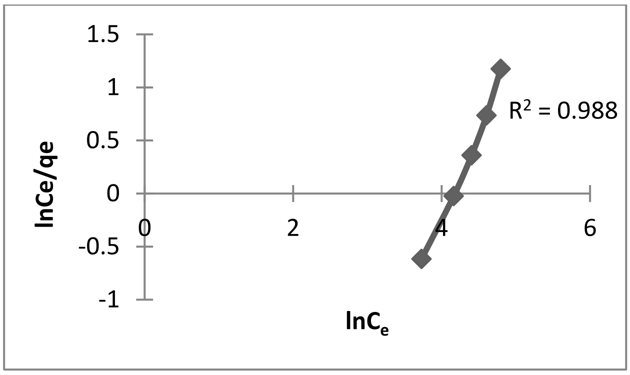

Figure 9: Redlich-Peterson equilibrium isotherm model for the bio-sorption of Cu(II)ions onto Pseudo stem.

Table 3: Parameters of Redlich-Peterson isotherm.

K_R(l/mg)	*g*	R^2
0.001	1.6525	0.988

The value of *g* was between 0 and 1. If the adsorption process follow the Langmuir isotherm, the value of *g* is 1. When the value of *g* is 0, the process follows the Henry' law. In this study, the calculated values of *g* did not lies between 0 and 1. It shows that the *g* value of the adsorption process follows neither Langmuir nor Henry's law. It can be presumed that chemically modified Pseudo stem has heterogeneous surfaces to biosorption.

The fitness of the data was established using R^2 value which is called the coefficient of determination. The R^2 value of Redlich-Peterson was 0.988 which is higher than R^2 value of Langmuir and Freudlich isotherms. It was shows that the Redlich-Peterson was the best isotherm and fitted to the equilibrium data for chemically modified Pseudo stem. This is because a degree of heterogeneity (β) is included and the equation can be applied successfully at high concentration of solutes (Shahmohammadi-Kalalagh *et al.* 2011).

IV. CONCLUSION

The studies presented here shows that chemical modification of Pseudo stem of *Musa Paradisiaca l.* with sodium hydroxide (NaOH), calcium chloride ($CaCl_2$) and ethanol (EtOH) became an effective biosorbent to remove Copper(II) ions. The maximum bio-sorption capacity of Cu(II) ions was 79% at 75 mg/l Cu(II) ions concentrations. The optimum pH for biosorption of Cu(II) ions was found to be at pH 5.90. Batch experiments were conducted to study the impacts of initial concentration of Cu(II) ions, dosage of adsorbent and contact time on the bio-sorption efficiency. All the experiment was conducted in room temperature of 27˚C. Varying other factors can also be investigated in the future experiments such as temperature and pH value which are affected on the biosorption capacity. The biosorption capacity of Cu(II) ions were analysed by Atomic Absorption Spectrometer (AAS). The result revealed that increase in chemical modification Pseudo stem (CMPS) dosage leads to increase in Cu(II) adsorption due to the large number of bio-sorbent sites. The biosorption equilibrium time was reached at 60 minutes. When the equilibrium was reached, it is important to know that no adsorption of heavy metals occurred in filter paper when the supernatant of sample was filtered by a filter paper. Moreover, as the initial concentration of Cu(II) ions increase, the biosorption capacity and adsorption efficiency decreases because of the saturation of binding site. Pseudo stem powders should sieved into smaller size such as less than 75μm to ensure higher amount of metals absorbed on the biosorbent. Three isotherm models can be described the adsorption of Cu(II) ions on the CMPS which are Langmuir, Freundlich and Redlich-Peterson models. The behaviour of equilibrium adsorption of Cu(II) ions by the CMPS obeyed the Redlich-Peterson model due to the higher and better correlation, R^2 with the experimental data compared to another two isotherm models. The R^2 value for Redlich-Peterson was 0.988 which is bigger than Langmuir (0.952) and Freundlich (0.9279). From the results obtained in the experimental study, the Pseudo stem of *Musa Paradisiaca l.* was a promising biosorbent for removal Cu(II) ions from electroplating wastewater industries. Additionally, it was very cheap besides easily available and renewable. Thus, chemically modified of Pseudo stem was suitable and became effectiveness to adsorb Cu(II) ions. However, further studies are needed by combination of biosorbent with more than one biosorbent to evaluate the heavy metal ions from wastewater. Additionally, it should to investigate the applicability of the research outcomes on real industrial wastewater.

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