

**Removal of Heavy Metal from Aqueous Solutions using Ground Water Treatment  
Plant Sludge**

by

Muhammad Afifi Che Rawi

Dissertation submitted in partial fulfilment of  
the requirements for the  
Bachelor of Engineering (Hons)  
(Civil Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the  
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Approved by,



(AP. Dr. Shamsul Rahman Kutty)

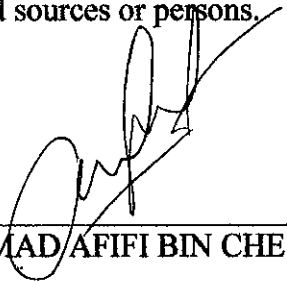
UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2008

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



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MUHAMMAD AFIFI BIN CHE RAWI

## ABSTRACT

Sludge waste produced from Ground Water Treatment Plant at Chicha, Kelantan was used as an adsorbent for copper ions in water. The adsorption process was carried out in a batch process and the effects of contact time (1–24 h), initial pH (4.3), initial metal ion concentration (1–100 mg/L) and temperature (20–30 °C) on the adsorption were investigated. Experimental results showed that the maximum adsorption capacity was achieved at pH 4.3 and adsorbed Cu (II) ion concentration was increased with increasing initial metal concentration and contact time. The isothermal data could be described well by the Langmuir equations and Freundlich equations. The correlation coefficient of the Langmuir isotherm ( $R^2$ ) were 0.457 for 1 mg/L, 0.678 for 10 mg/L, and 0.675 for 100 mg/L copper concentration. While, the correlation of Freundlich isotherms ( $R^2$ ) were 0.609, 0.638 and 0.735 for 1, 10 and 100 mg/L copper concentration.

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# **CHAPTER 1: INTRODUCTION**

## **1.1 Background Study**

Water, the universal solvent, is required to sustain biological and botanical life. It is broadly used in virtually every aspect of human existence and every single industrial process. Household and commercial water usage produces effluents containing a variety of organic, inorganic, metallic, hydrocarbon, and biological species (McKay, 1995). Because of the widespread pollution of water and soil by industrial and household activities, water treatments have been the subject of numerous investigations.

Heavy metals are commonly found in various industrial wastewaters. They can cause serious water pollution and threaten the environment. It is therefore, essential to control and prevent their unsystematic discharge into the environment. For this reason, increased attention is being focused on the development of technical know-how for their removal from metal bearing effluents before being discharged into water bodies and natural streams (M. H. Isa et al., 2007).

The conventional methods of metal removal from wastewater, such as, precipitation, adsorption, ion exchange, etc., are expensive and may not work well at low concentrations of metals (Ahluwalia and Goyal, 2006). While traditional technologies, such as ion exchange, chemical precipitation or reverse osmosis are often ineffective or expensive at low concentrations. Efficient and environmental friendly technologies are, therefore, needed to be developed to reduce heavy metal contents in wastewaters with cheaper and efficient alternatives.

## **1.2 Problem Statement**

Due to increased in industrial activities, chemical substance such as metals, organic and synthetic compounds generate pollution in environment and result in the deterioration of ecosystems. Unlike the other pollutant, metal are non-biodegradable pollution in the environment and contamination posses a serious threat to organisms due to the accumulation in living tissues (Volesky, 1990). The main sources of metal pollution include textile, electroplating, painting, and dyeing industries.

In present time, the development of adsorption mechanism which includes the application of adsorption column and adsorbent selection combination is still under progress. Adsorption is one of the most utilized wastewater treatment processes. Treatment methods used to remove heavy metal ions from waste effluents include precipitation, ion exchange, evaporation, freeze purification, reverse osmosis, electrolysis, cementation, flotation, and electro dialysis. These methods are often expensive and cannot consistently accommodate the strict water quality standards.

Natural or waste adsorbent may provide effective and economical water treatment alternative. Elsewhere control and reduction of waste volume in landfills are taken into account. As matter of fact, the sludge is considered as a waste. Sludge is generally the left over from any treatment facilities. It is disposed at landfills in the form of de-watered sludge or incinerator ash. Nevertheless, so far sludge waste has now foresee the huge potential in utilizing sludge as an alternative resource.

### **1.3 Objective and Scope of Study**

The main objective of this research is to study efficiency of waste in removing the heavy metal from the industrial waste by applying batch absorption method. The wastes used are sludge waste from Chicha Groundwater Treatment Plant in Kelantan. The adsorption studies are followed up with the supporting evidence that can be achieved from the laboratory works.

From the experiment result is to observe the adsorption occurs between the adsorbent and the heavy metal. The percentage of the adsorbed heavy metal by the adsorbent will be recorded. As the result of the experiment achieved, further analyses were made to confirm the effectiveness of the material used as an adsorbent. The final result of the project achieved when both material and the system can works in the peak condition where the adsorption of the heavy metal occurs in the system.

The further progress of this research is more towards identifying the potential of sludge waste to adsorb more types of heavy metal solution. In present study, the sludge waste show effective percentage of adsorption only for copper solution. Variations in copper concentration have been used in the proceeding of this research to test capacity of adsorption by the sludge waste. The methods of batch adsorption are to mixed adsorbent and the heavy metal in the BOD bottle and shake for specific hours. Physical and chemical reactions are belief to support the adsorption of heavy metal by the sludge waste using the batch adsorption method.

The aim of this study is to investigate the possible use of sludge waste (containing ferric oxide) to adsorb heavy metal, copper. The dynamic behavior of adsorption was studied on the effects of pH, concentration of sludge, temperature and capacity.

## **CHAPTER 2: LITERATURE REVIEW**

### **2.1 Sludge Waste**

Sludge is a generic term for solids separated from suspension in a liquid by a variety of processes. Sludge usually refers to solid waste extracted in the process of treatment.

Sludge resulting from treatment plants creates problems of disposal. Generally, dewatered sludge is disposed of by spreading on the land or by land filling. However, for highly urbanized cities, sludge disposal by land filling might not be appropriate due to land limitation. Incineration might be an alternative solution. The products from the process are dried sludge and sludge ash. The substantial amount of ash that produced after the burning process must be disposed of by other means.

### **2.2 Effects of Copper**

Copper is a very common substance that occurs naturally in the environment and spreads through the environment through natural phenomena. Humans widely use copper. For instance it is applied in the industries and in agriculture. The production of copper has lifted over the last decades and due to this copper quantities in the environment have expanded.

The world's copper production is still rising. This basically means that more and more copper ends up in the environment.

### **2.2.1 Health effects of Copper**

Copper in daily diet is necessary for good health. Normal person eat and drink about 1,000 micrograms (1,000 ug) of copper per day. Drinking water normally contributes approximately, 150 ug/day.

However, immediate effects from drinking water which contains elevated levels of copper include; vomiting, diarrhea, stomach cramps, and nausea. The seriousness of these effects can be expected to increase with increased copper levels or length of exposure. Long-term exposure to copper can cause irritation of the nose, mouth and eyes and it causes headaches, stomachaches, dizziness, vomiting and diarrhea. Intentionally high uptakes of copper may cause liver and kidney damage and even death.

Industrial exposure to copper fumes, dusts, or mists may result in metal fume fever with atrophic changes in nasal mucous membranes. Chronic copper poisoning are results in Wilson's disease, characterized by a hepatic cirrhosis, brain damage, demyelization, renal disease, and copper deposition in the cornea.

Children under one year of age are more sensitive to copper than adults. Long-term exposure (more than 14 days) to copper in drinking water which is much higher than 1,000 ug/l has been found to cause kidney and liver damage in infants. Other persons who are highly susceptible to copper toxicity include people with liver damage or Wilson's disease (Lenntech, 2008).

### **2.2.2 Environmental effects of Copper**

When copper ends up in soil it strongly attaches to organic matter and minerals. As a result it does not travel very far after release and it hardly ever enters groundwater. In surface water copper can travel great distances, either suspended on sludge particles or as free ions.

Copper does not break down in the environment and because of that it can accumulate in plants and animals when it is found in soils. On copper-rich soils only a limited number of plants has a chance of survival. That is why there is not much plant diversity near copper-disposing factories. Due to the effects upon plants copper is a serious threat to the productions of farmlands. Copper can seriously influence the proceedings of certain farmlands, depending upon the acidity of the soil and the presence of organic matter. Despite of this, copper-containing manures are still applied.

Copper can interrupt the activity in soils, as it negatively influences the activity of microorganisms and earthworms. The decomposition of organic matter may seriously slow down because of this.

When the soils of farmland are polluted with copper, animals will adsorb concentrations that are damaging to their health. Mainly sheep suffer a great deal from copper poisoning, because the effects of copper are manifesting at fairly low concentrations (Lenntech, 2008).



### **2.3 Adsorption theory**

The simplest definition of adsorption is the attraction of molecules to a surface. Physical adsorption takes place when weak intermolecular forces provide the attraction, whereas chemisorptions utilize chemical bonding forces to bind a species to a surface (Ebbing, 1996). A soluble chemical species, the adsorbate, is removed from a fluid by contacting a solid surface, the adsorbent. The force of interaction between adsorbent surface atoms and adsorbate molecules are comparable to Van der Waals forces between adjacent molecules (LaGrega, Buckingham, & Evans, 1994). Both attractive forces and repulsive forces act between molecules. The net force of attraction and repulsion depends on the distance between the surface of the adsorbent and the adsorbate molecule (Cooney, 1999).

Physical adsorption involves weak force, takes place at low temperatures and is reversible. Physical adsorption is exothermic (Conney, 1999). Physical adsorption is primarily affected by the surface area of the adsorbent, adsorbent pore structure, adsorbent surface chemistry, nature of the adsorbate, pH of the adsorbate, pH of the solution, and the presence of competing adsorbates. These major factors make physical adsorption a complex phenomenon (LaGrega et al., 1994; Conney, 1999).

The surface area of the adsorbent, like the surface area of a catalyst, is comprised of an external surface area and the internal surface area of the pore walls (Folger, 1999). The external surface of the adsorbent contributes to the external superficial surface area, while the pores of the adsorbent contribute mostly to the internal surface area. Because surface area so strongly affects adsorption capacity, the greater the surface area of the adsorbent becomes, the greater the capacity for physical adsorption (Conney, 1999). Adsorbent surfaces are typically made up of various surface functional groups. Adsorption of organic adsorbate is particularly dependent on the quality and nature of surface oxide groups. Carbon/oxygen surface functional groups may occur naturally or may be created by oxidation that occurs if an adsorbent undergoes an activation process.

In the case of activated carbon, the activation process involves heating in absence of air followed by steam or chemical treatments to increase surface area develop porosity.

#### **2.4 Adsorption of copper and zinc from aqueous solutions by using natural clay.**

In this study, removal of copper ( $\text{Cu}^{2+}$ ) and zinc ( $\text{Zn}^{2+}$ ) from aqueous solutions is investigated using Cankırı bentonite, a natural clay. During the removal process, batch technique is used, and the effects of pH, clay amount, heavy metal concentration and agitation time on adsorption efficiency are studied. Langmuir, Freundlich and Dubinin–Radushkevich (D–R) isotherms are applied in order to determine the efficiency of natural clay used as an adsorbent. Results show that all isotherms are linear. It is determined that adsorption of  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  is well-fitted by the second order reaction kinetic. In addition, calculated and experimental heavy metal amounts adsorbed by the unit clay mass are too close to each other. It is concluded that natural clay can be used as an effective adsorbent for removing  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  from aqueous solutions. (Sevil Veli et al., 2007)

#### **2.5 Copper removal from aqueous solutions using *Rhizopus Oligosporus***

Dried biomass of *Rhizopus oligosporus* produced from food processing wastewater was used as an adsorbent for copper ions in water. The adsorption process was carried out in a batch process and the effects of contact time (1–48 h), initial pH (2.0–6.0), initial metal ion concentration (20–100 mg L<sup>-1</sup>) and temperature (20–38 °C) on the adsorption were investigated. Experimental results showed that the maximum adsorption capacity was achieved at pH 5.0 and adsorbed Cu (II) ion concentration was increased with increasing initial metal concentration and contact time. The isothermal data could be described well by the Langmuir equations and monolayer capacity had a mean value of 79.37 mg/g. A pseudo-second order reaction model provided the best description of the data with a correlation coefficient 0.99 for different initial metal concentrations. (H. Duygu Ozsoya et al., 2007)

## **2.6 Copper removal from aqueous solution using Sugar Beet Pulp**

Sugar beet pulp was converted into effective copper sorption material by treating subsequently with NaOH and citric acid. Compared with the untreated sugar beet pulp, the cat ion exchange capacity of the modified sugar beet pulp increased from 0.86 to 3.21 mequiv/g. Swelling capacity and COD values of modified sugar beet pulp were found to be decreased in the ratio of 38% and 61%, respectively, compared to the corresponding values of native sugar beet pulp, meaning that modification causes stabilization. Sorption characteristics of the modified sugar beet pulp towards copper ions were studied with batch experiments. Pseudo-first, pseudo-second-order and intra-particle kinetic models were applied to the kinetic data and it was found that the sorption processes followed the pseudo-second-order rate kinetics with activation energy of 16.34 kJ/mol. The equilibration data fit best with the Langmuir isotherm the maximum copper sorption capacity of which is 119.43 mg/g. The mean free energy of copper sorption process calculated from Dubinin–Radushkevich model and the Polanyi potential concept was found to be in the range of 10.91–11.95 kJ/mol showing that the main mechanism governing the sorption process is ion exchange. The negative values found for enthalpy change ( $-14.797$  kJ/mol over the range of 25–55 °C) and free energy change ( $-19.361$  kJ/mol for 25 °C) indicate that the sorption process is exothermic and spontaneous in nature.

## 2.7 Equilibrium isotherms and kinetics of adsorption

The Langmuir isotherm was used to describe observed sorption phenomena. The Langmuir isotherm applies to adsorption on completely homogenous surface with negligible interaction between adsorbed molecules ([Ho, 2004] and [Yu et al., 2001]). The linear form of the equation can be written as

$$\left( \frac{1}{q_{eq}} = \frac{1}{bq_{max}} \cdot \frac{1}{C_{eq}} + \frac{1}{q_{max}} \right) \quad (1)$$

where  $C_{eq}$  is the equilibrium concentration of copper,  $q_{eq}$  is the amount of adsorption at equilibrium,  $q_{max}$  is the maximum mono layer capacity, and  $b$  is an equilibrium constant of Langmuir.

The Freundlich isotherm (empirical model adsorption in aqueous systems) was also tested with our experimental data. The linear form of the equation can be written as

$$\left( \ln q_{eq} = \ln K_f + \frac{1}{n} \ln C_{eq} \right) \quad (2)$$

where  $K_f$  is the measure of sorption capacity,  $1/n$  is sorption intensity.

In order to analyze the sorption kinetics of Cu (II) ions, pseudo-first order and pseudo-second order kinetic models were applied to data. A simple pseudo first-order equation due to Lagergren was used by different researchers ([Ho and McKay, 1998] and [Bhattacharyya and Sharma, 2005]):

$$\left( \log(q_{eq} - q_t) = \log q_{eq} - \frac{k_{ad}}{2.303t} \right) \quad (3)$$

where  $q_e$  and  $q_t$  are the amount of adsorption at equilibrium and at time  $t$ , respectively, and  $k_{ad}$  is the rate constant of the pseudo first-order adsorption process. A plot of  $\log(q_{eq} - q_t)$  versus  $t$  would provide a straight line for first order adsorption kinetics, which allows computation of the adsorption rate constant,  $k_{ad}$ .

Ho's second-order rate equation, which has been called a pseudo-second order kinetic expression, has also been applied widely ([Ho, 2005] and [Ho and McKay, 2000]). The linear form of the kinetic rate equations can be written as follows:

$$\left( \frac{t}{q_t} = \frac{1}{kq_{eq}^2} + \frac{1}{q_{eq}} t \right) \quad (4)$$

Where,

$k$  is the rate constant of sorption ( $\text{dm}^3 \text{mg}^{-1} \text{min}^{-1}$ ),

$q_e$  is the amount of metal ion adsorbed at equilibrium (mg/g),

$q_t$  is the amount of metal ion sorbed at time  $t$  (mg/g).

The constants can be determined experimentally by plotting of  $t/q_t$  against  $t$ .

## **CHAPTER 3: MATERIALS AND METHODS**

### **3.1 Preparation of Copper Solution**

Aqueous solutions were prepared with deionized water. A 10 mg/L Cu stock solution was prepared using copper nitrate,  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ . Copper nitrate was dilute subsequently to obtain the desired 10L working solutions for batch experiments.

Amount of copper nitrate to dilute with the deionized water are;

For 100 mg/L solution,

=0.30 g of copper nitrate

For 10 mg/L solution,

=0.038 g

For 1 mg/L solution,

=  $3.8 \times 10^{-3}$  g

Refer Appendix 1-1 for Copper Nitrate Calculation.

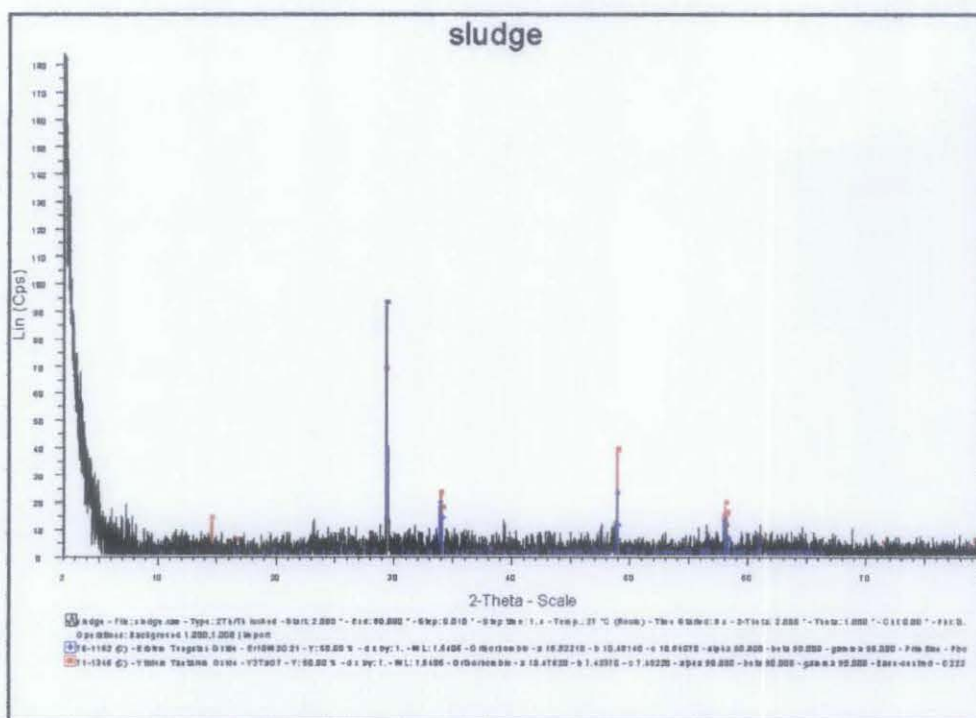
### **3.2 Preparation of Groundwater Treatment Plant Sludge Waste**

Sludge waste supplied by Chicha Underground Wastewater Treatment Plant in Kelantan. This sludge waste sample was collected and dried in the oven at  $105^{\circ}$  T for a day to ensure entire sample humidity and moisture is reduced so that it can be easily grind and also reducing the effect of humidity during experiment. The sample adsorbent material was grinded till it becomes powdered form before it was diluted in the copper solutions. Sludge waste then was sieve through  $600\mu$  of sieving pan. This process is to make sure that sludge have the same particle size.

The sludge waste was undergone XRD test to analyze the component in the sludge waste. Below are test results from SEM and XRD on iron sludge.

**Table 3.1: Composition Content in Sludge Waste**

<b>Material</b>	<b>Quantities</b>	<b>Percentage (%)</b>
Al <sub>2</sub> O <sub>3</sub>	7.8KCps	6.15
SiO <sub>2</sub>	33.6 KCps	16.4
P <sub>2</sub> O <sub>5</sub>	8.2 KCps	2.53
K <sub>2</sub> O	1.1 KCps	0.0555
CaO	300.2 KCps	16.3
MnO	13.8 KCps	0.310
Fe <sub>2</sub> O <sub>3</sub>	3021.5 KCps	56.4
SrO	3.8 KCps	0.0186
BaO	1.4 KCps	0.193
Tb <sub>4</sub> O <sub>7</sub>	8.6 KCps	0.0445
Re	5.5 KCps	0.211
Compton	-	0.74
Rayleigh	-	1.47
Norm	-	100.00



**Figure 3.1:** XRD Result on Sludge Waste

### 3.3 Experimental Batch Study

Batch tests were conducted at room temperature ( $28 \pm 1$  °C) to obtain the adsorption rates and equilibrium data. Unless otherwise stated, BOD bottles, each filled with 100mL of Cu solutions and a certain dosages of sludge waste were used in all experiments. The desired dosages of sludge waste were adjusted according to the purposed of the test. The BOD bottles contents were agitated using an orbital shaker for the desired period (1, 3, 6 and 24 hours) at a speed of 150 rpm. Whatman No. 1 filter paper was used to filter the contents of the BOD bottles, followed by the analysis of Cu in the filtrate using atomic absorption spectrometry (AAS).

Initial Cu solutions of concentration 1, 10, and 100 mg/L were used to determine the effects of adsorbent dosage. The amount of sludge waste in the BOD bottle was varied from 25 mg to 400 mg and the BOD bottles were agitated for 3h, 6h and 24h.



## **CHAPTER 4: RESULTS AND DISCUSSION**

### **4.1 Experiment 1**

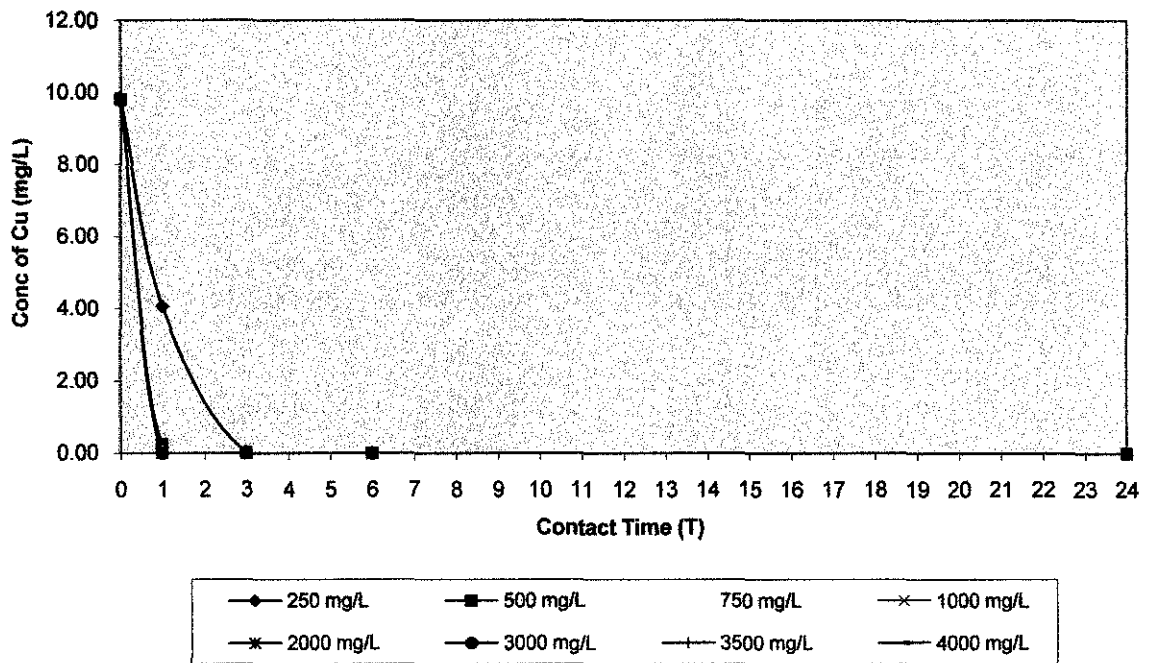
In experiment 1, batch test were conducted at room temperature ( $28\pm 1$  °C). Copper concentration is 10 mg/L. The pH of the copper solution was set to 4.3 to get the optimum absorption.

The BOD bottles were filled with 100mL of 10mg/L Cu solutions. The desired dosages of sludge waste were adjusted according to the purposed of the test. The dosages were: 25, 50, 75, 100, 200, 300, 350, and 400 mg.

The BOD bottles contents were agitated using an orbital shaker for the desired period (1, 3, 6 and 24 hours) at a speed of 150 rpm. Whatman No. 1 filter paper was used to filter the contents of the BOD bottles, followed by the analysis of Cu in the filtrate using atomic absorption spectrometry (AAS).

#### 4.1.1 Experiment 1: Effect of Iron Sludge Dosage

In the experiment studies carried on in order to determine the optimum sludge dosage, solutions with an initial metal concentration of 10 mg/L were used at pH 4.3. During the contact time of 1 hr, 3 hr, 6 hr, and 24 hr, the amount of dosage of iron sludge added to the solutions varied between 250, 500, 750, 1000, 2000, 3000, 3500, and 4000 mg/L.

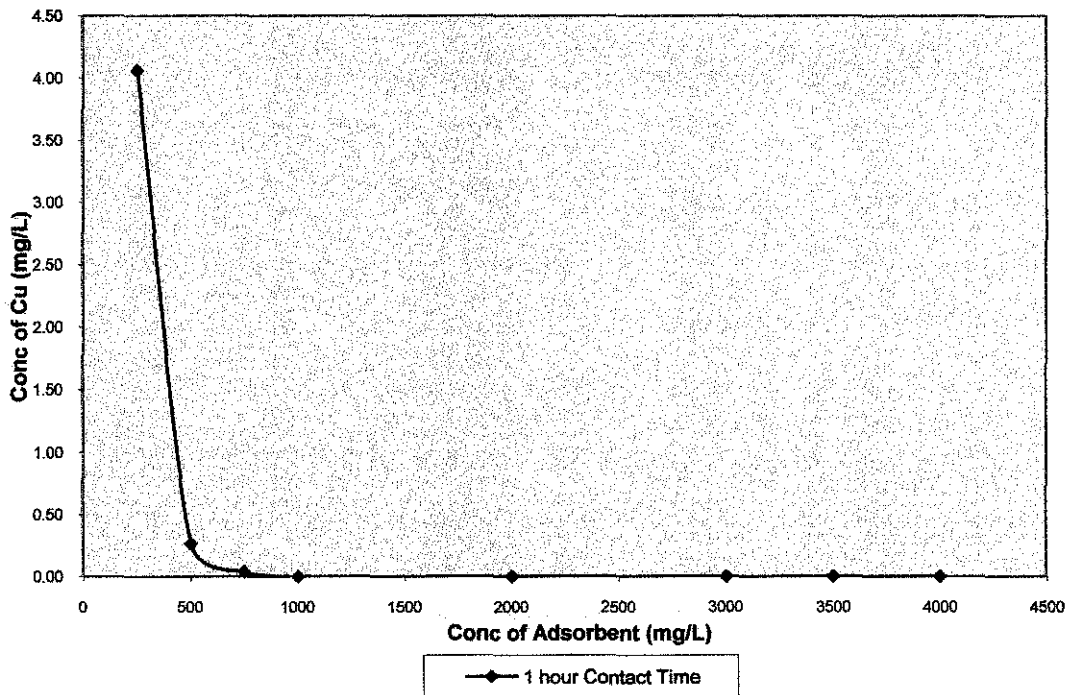


**Figure 4.1:** Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_o = 9.80$  mg/L.

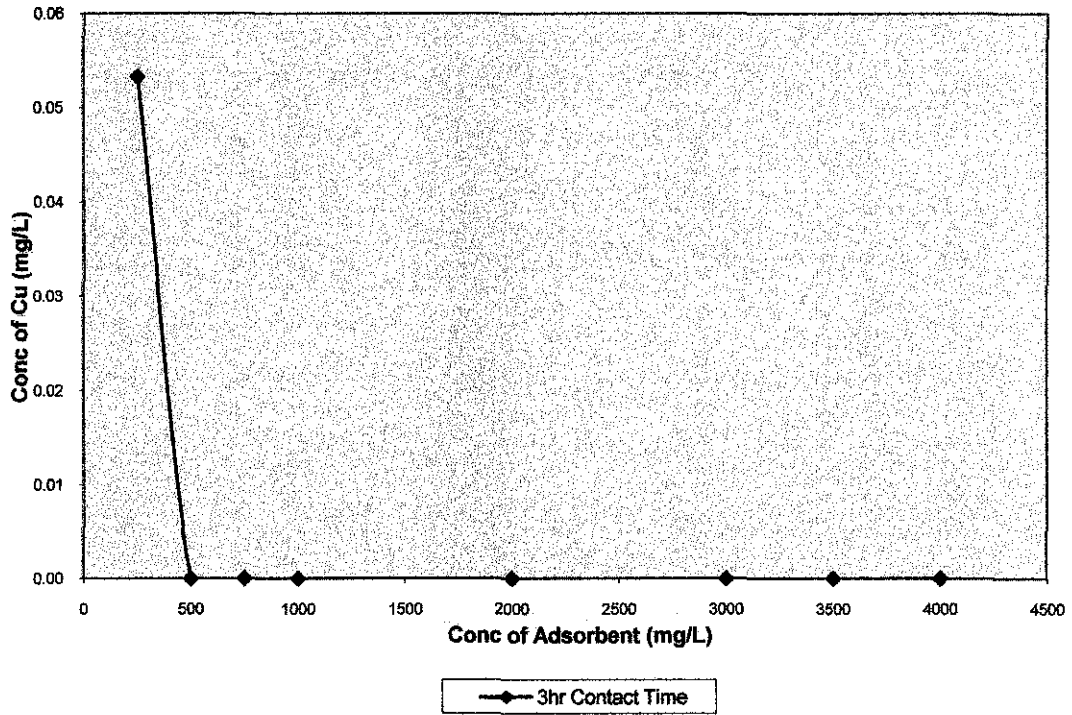
### 4.1.2 Experiment 1: Effect of Agitation Time

The agitation time was also evaluated as one of the most important factors affecting the adsorption efficiency (Sevil Veli and Bilge Alyuz, 2007). By using pH of 4.3, sludge waste and copper concentration, the effect of time on removal efficiency was analyzed. At the study carried out in a batch system, samples were taken at different time periods; 1, 3, 6, and 24 hr.

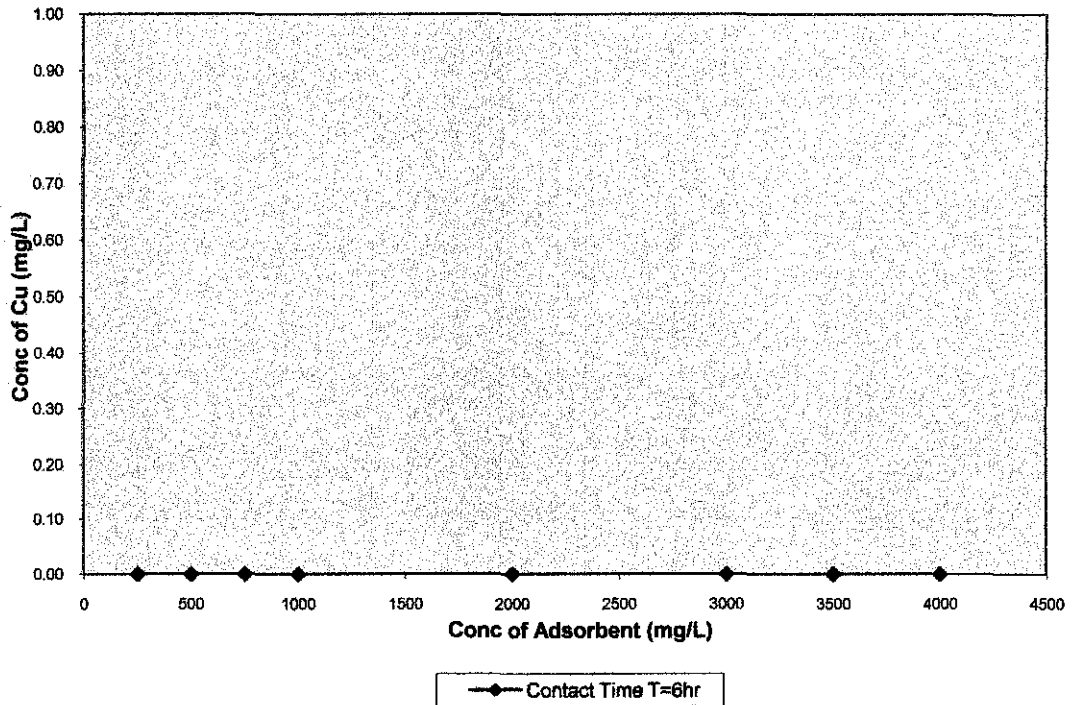
Figures show the removal efficiencies for 250, 500, 750, 1000, 2000, 3000, 3500, and 4000 mg/L of sludge waste. It is evident that an increase in the contact time resulted in an increase in the removal efficiency of Cu from solution.



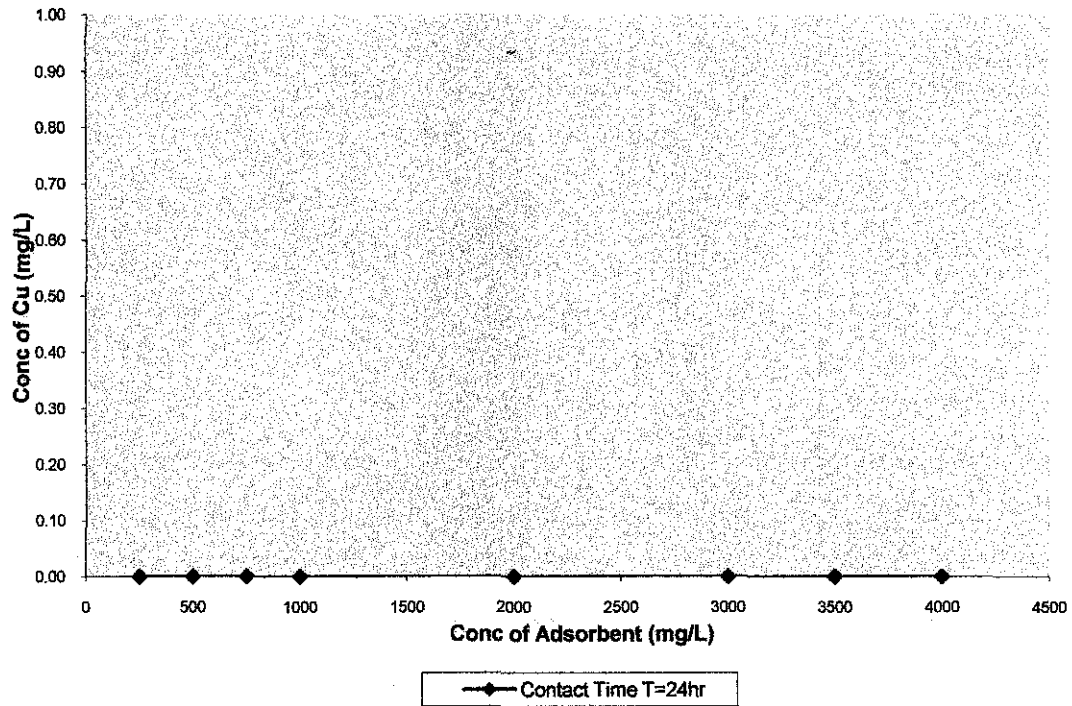
**Figure 4.2:** Final Concentration of Copper vs. Sludge dosage for 1 hour Contact time. ( $C_0=9.80$  mg/L)



**Figure 4.3:** Final Concentration of Copper vs. Sludge dosage for 3 hours Contact time. ( $C_0=9.80$  mg/L)



**Figure 4.4:** Final Concentration of Copper vs. Sludge dosage for 6 hours Contact time. ( $C_0=9.80$  mg/L)



**Figure 4.5:** Final Concentration of Copper vs. Sludge dosage for 24 hours Contact time. ( $C_0=9.80$  mg/L)

#### 4.1.3 Experiment 1: Effect of Copper Concentration

Optimum concentration will be determined after experimental studies done under various metal concentrations ranging between 1 mg/L and 100 mg/L. For these experimental studies, only 10 mg/L concentration of Copper was used.

#### 4.1.4 Experiment 1: Discussion

The results show that the entire copper ion of 9.8 mg/L copper concentration was adsorb by sludge waste at dosage 750, 500, 250, 250 mg/L at duration 1, 3, 6, 24 hours, respectively. So, it was recommended to decrease the dosage of the sludge to find the optimum dosage. The recommended ranges of dosage to use for the next experiment were 100 to 1250 mg/L. The concentrations of copper also need to vary from 1 to 100 mg/L.

## **4.2 Experiment 2**

In experiment 2, batch test were conducted at room temperature ( $28\pm 1$  °C). The pH of the copper solution was set to 4.3 to get the optimum absorption.

The BOD bottles were filled with 100 mL of copper solutions. The concentration of copper solutions used are; 2.03, 15.08, 103.2 mg/L. The desired dosages of sludge waste were varied and decreased up to 125 mg. The dosages were: 10, 25, 50, 75, 100, and 125 mg.

The BOD bottles contents were agitated using an orbital shaker for the desired period (1, 3, 6 and 24 hours) at a speed of 150 rpm. Whatman No. 1 filter paper was used to filter the contents of the BOD bottles, followed by the analysis of Cu in the filtrate using atomic absorption spectrometry (AAS).

### **4.2.1 Experiment 2: Effect of Sludge Dosage**

In the experiment studies carried on in order to determine the optimum sludge dosage, solutions with an initial copper concentration of 1, 10, and 100 mg/L were used at pH 4.3. During the contact time of 1, 3, 6, and 24 hr, the amount of dosage of iron sludge added to the solutions varied between 100, 250, 500, 750, 1000, and 1250 mg/L.

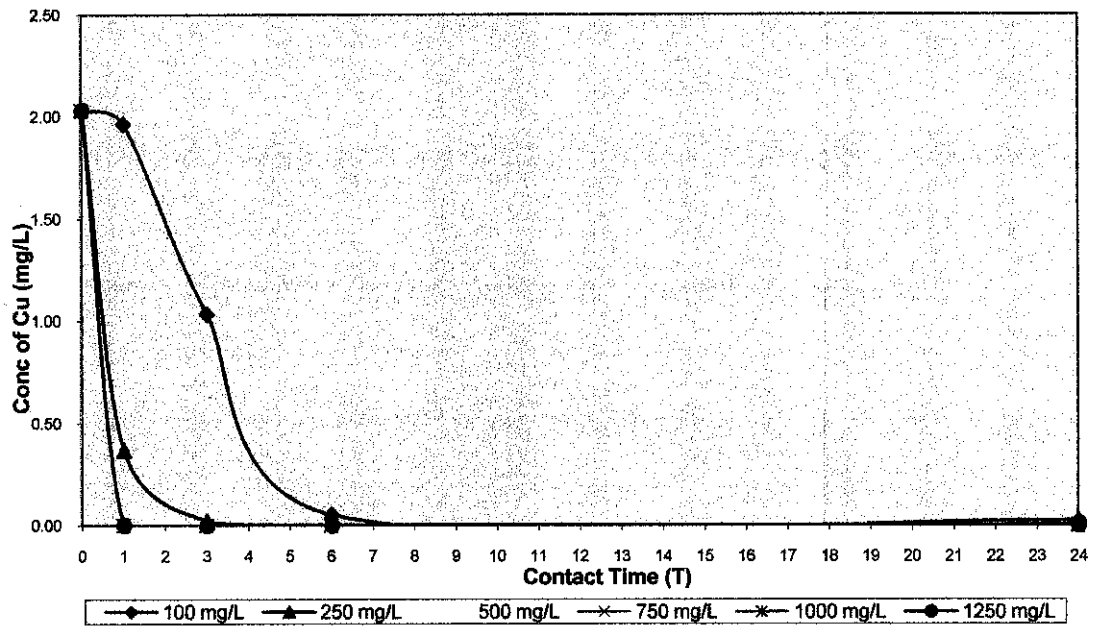


Figure 4.6: Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_o = 2.03$  mg/L.

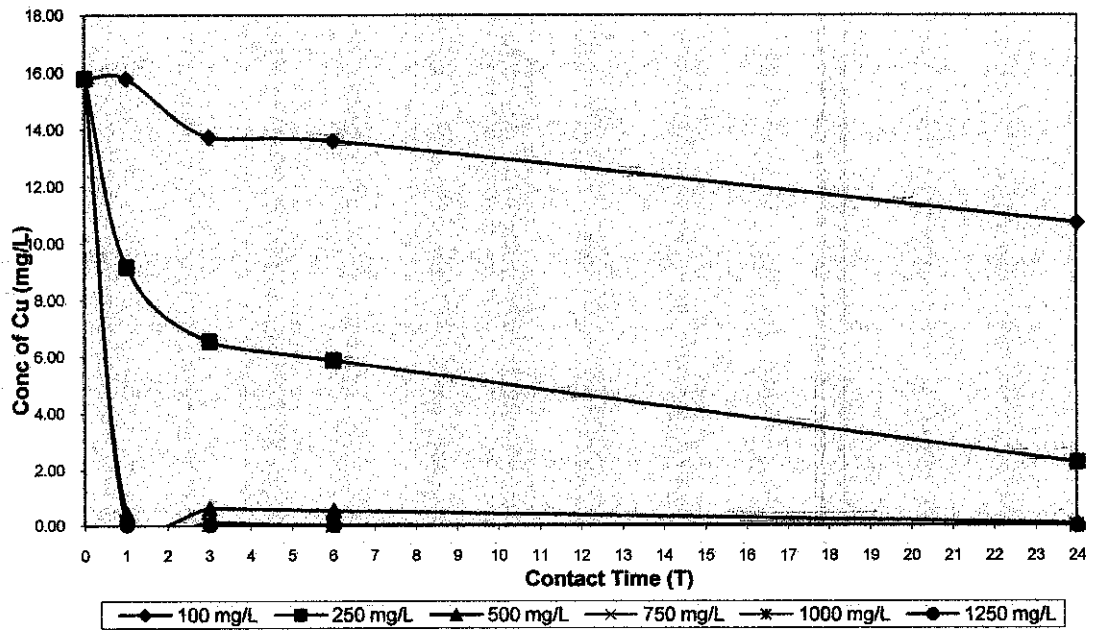
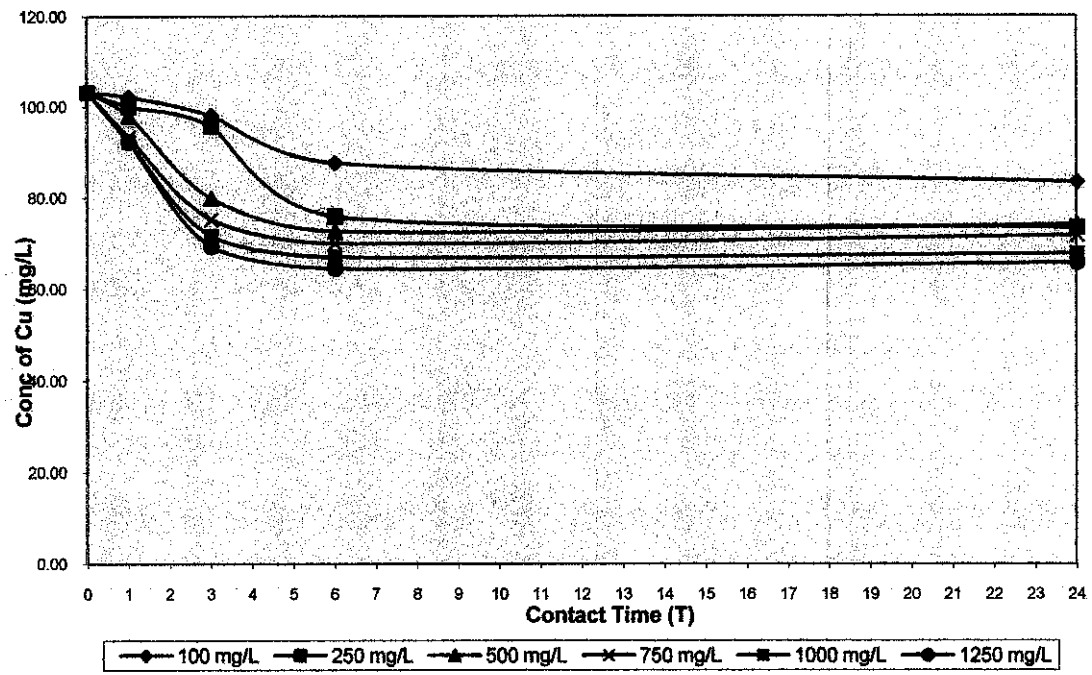


Figure 4.7: Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_o = 15.08$  mg/L.



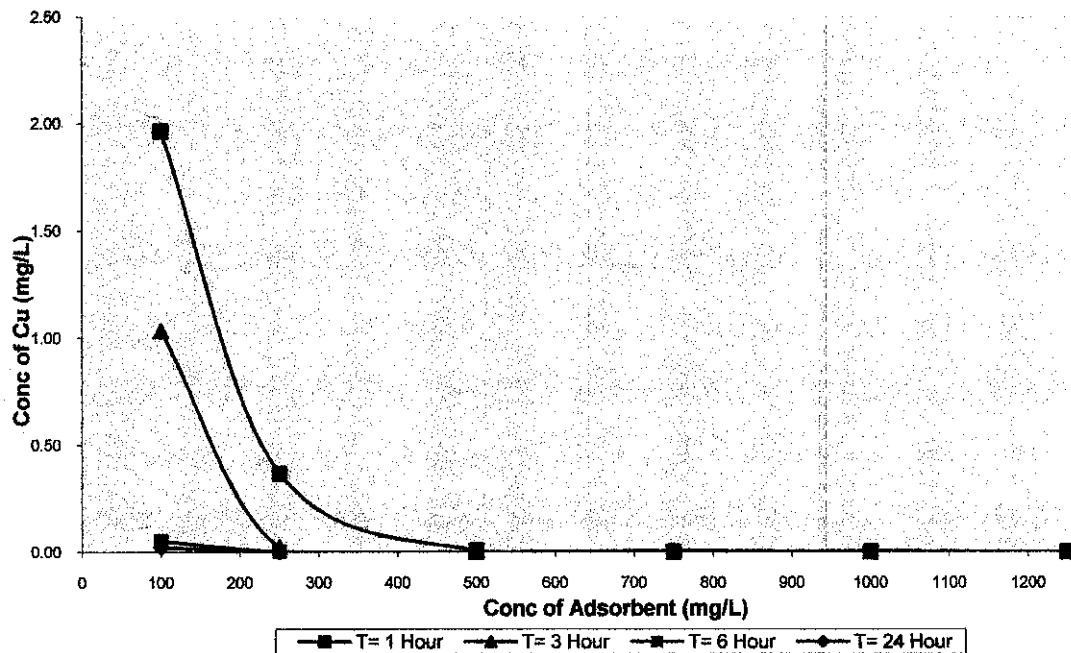
**Figure 4.8:** Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_0 = 103.02$  mg/L.



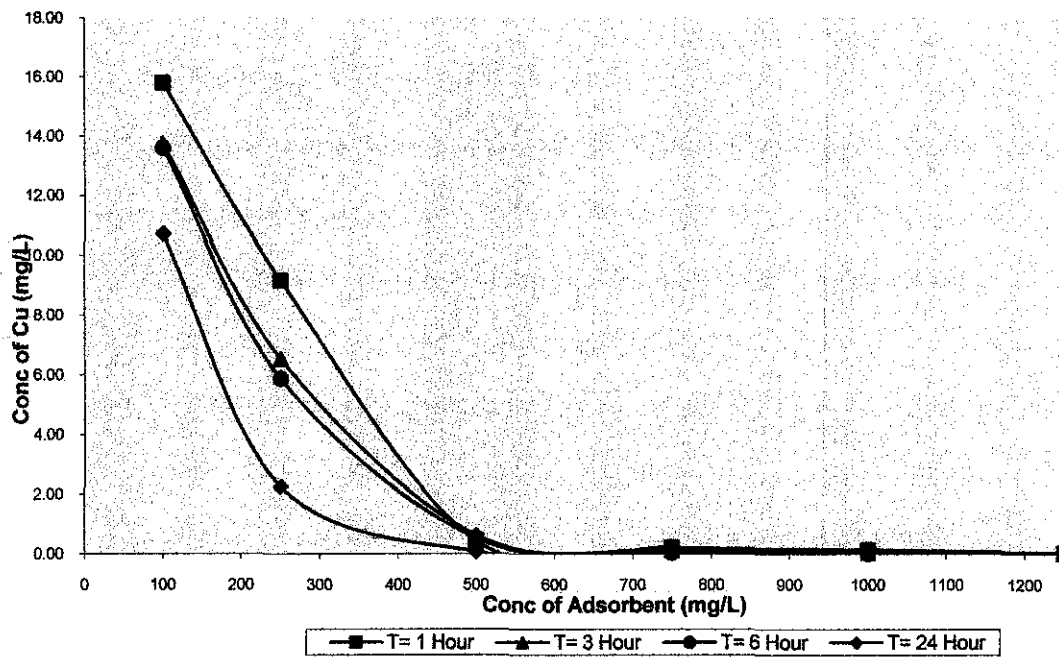
#### 4.2.2 Experiment 2: Effect of Agitation Time

The agitation time was also evaluated as one of the most important factors affecting the adsorption efficiency (Sevil Veli and Bilge Alyuz, 2007). By using pH of 4.3, sludge waste and copper concentration, the effect of time on removal efficiency was analyzed. At the study carried out in a batch system, samples were taken at different time periods; 1, 3, 6, and 24 hours.

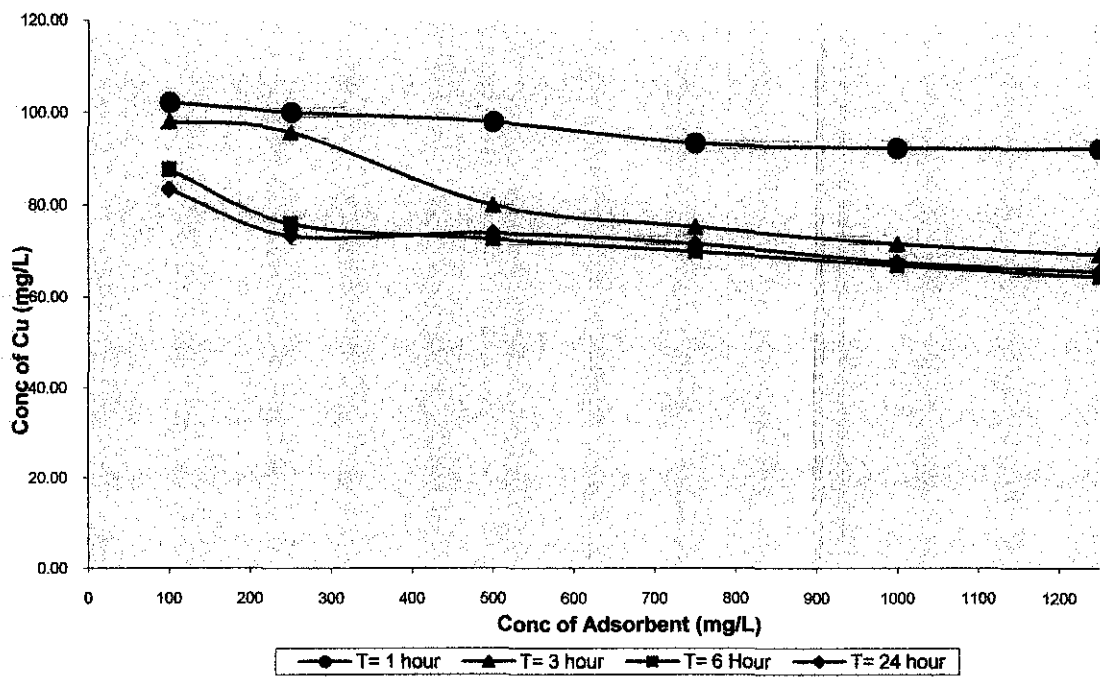
Figures show the removal efficiencies for 100, 250, 500, 750, 1000, and 1250 mg/L of sludge waste. It is evident that an increase in the contact time resulted in an increase in the removal efficiency of copper from solution.



**Figure 4.9:** Final Concentration of Copper vs. Sludge dosage for 2.03 mg/L copper solution.



**Figure 4.10:** Final Concentration of Copper vs. Sludge dosage for 15.08 mg/L copper solution.



**Figure 4.11:** Final Concentration of Copper vs. Sludge dosage for 103.2 mg/L copper solution.

### **4.2.3 Experiment 2: Discussion**

The results show that the entire copper ion of 2.03 mg/L copper concentration was adsorb by sludge waste at dosage 750, 500, 250,250 mg/L at duration 1, 3, 6, 24 hours, respectively. While for the 15.08 mg/L copper concentration, the copper were 100% adsorb by sludge waste dosage of 1250, 1250, 1000, 750 mg/L at duration 1, 3, 6, 24 hours, respectively. For 103.2 mg/L copper concentration, the maximum dosage of sludge, 1250 mg/L adsorb 13, 15.6, 16.64, and 22.8% at duration of 1, 3, 6, and 24 hours, respectively.

The study was then recommended to decrease the dosage of the sludge to find the optimum dosage of sludge. The recommended ranges of dosage to use for the next experiment were 100 to 600 mg/L.

### **4.3 Experiment 3**

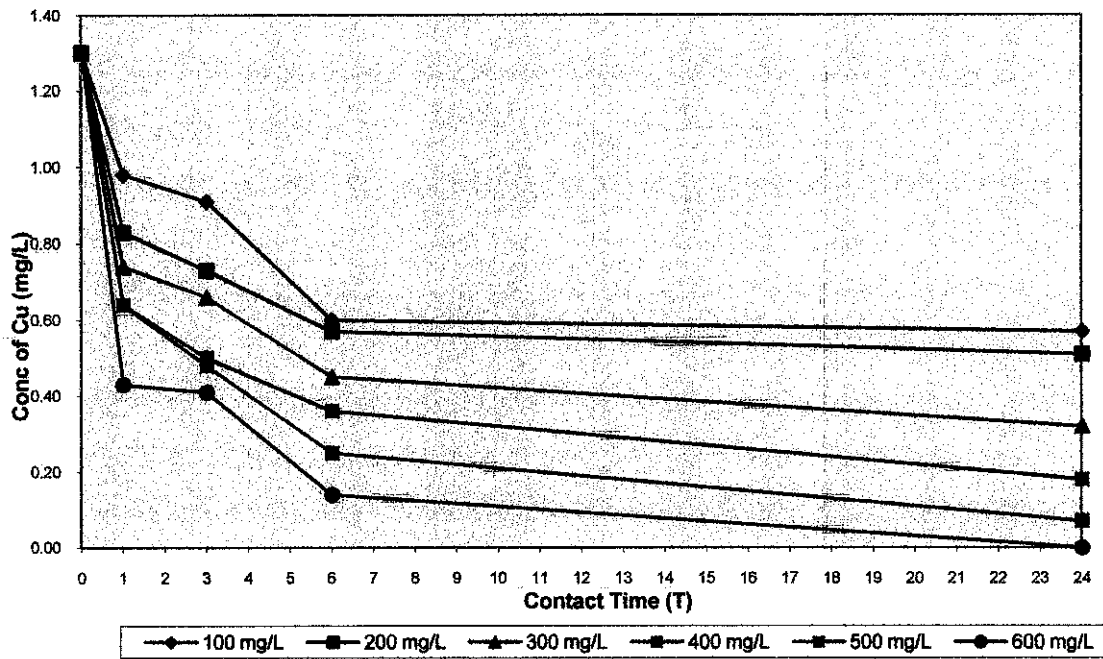
In experiment 3, batch test were conducted at room temperature ( $28 \pm 1$  °C). The pH of the copper solution was set to 4.3 to get the optimum absorption.

The BOD bottles were filled with 100 mL of copper solutions. The concentration of copper solutions are; 1.30, 10.2, 102.45 mg/L. The desired dosages of sludge waste were varied and decreased up to 125 mg. The dosages were: 10, 25, 50, 75, 100, and 125 mg. The experiments were repeated for 3 variation of copper concentration; 1, 10 and 100 mg/L.

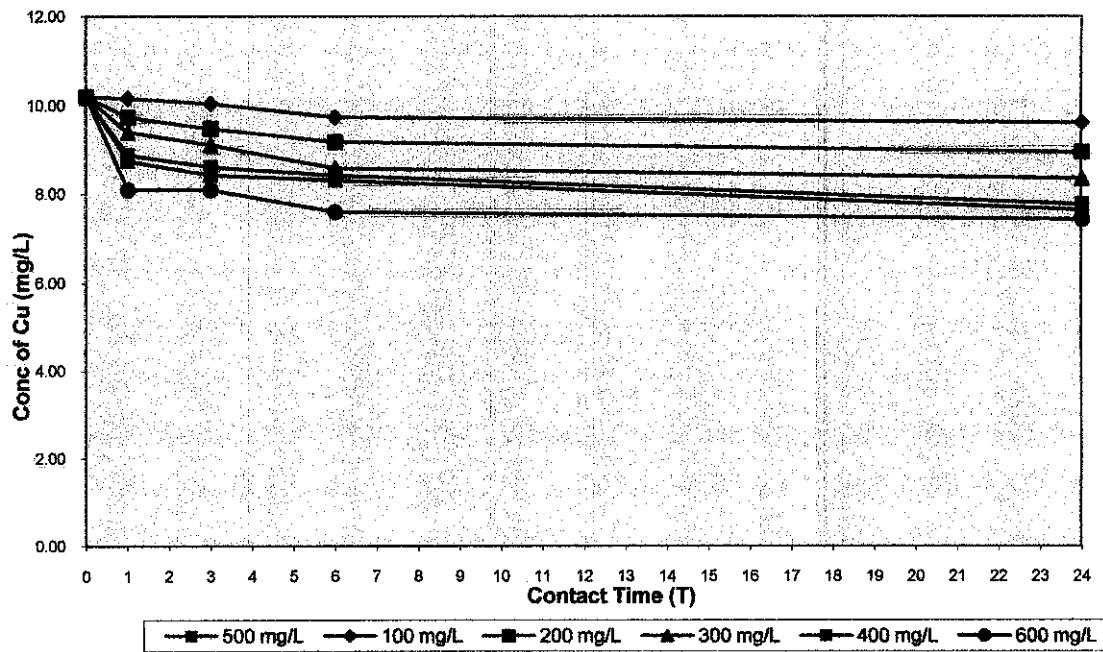
The BOD bottles contents were agitated using an orbital shaker for the desired period (1, 3, 6 and 24 hours) at a speed of 150 rpm. Whatman No. 1 filter paper was used to filter the contents of the BOD bottles, followed by the analysis of Cu in the filtrate using atomic absorption spectrometry (AAS).

#### **4.3.1 Experiment 3: Effect of Iron Sludge Dosage**

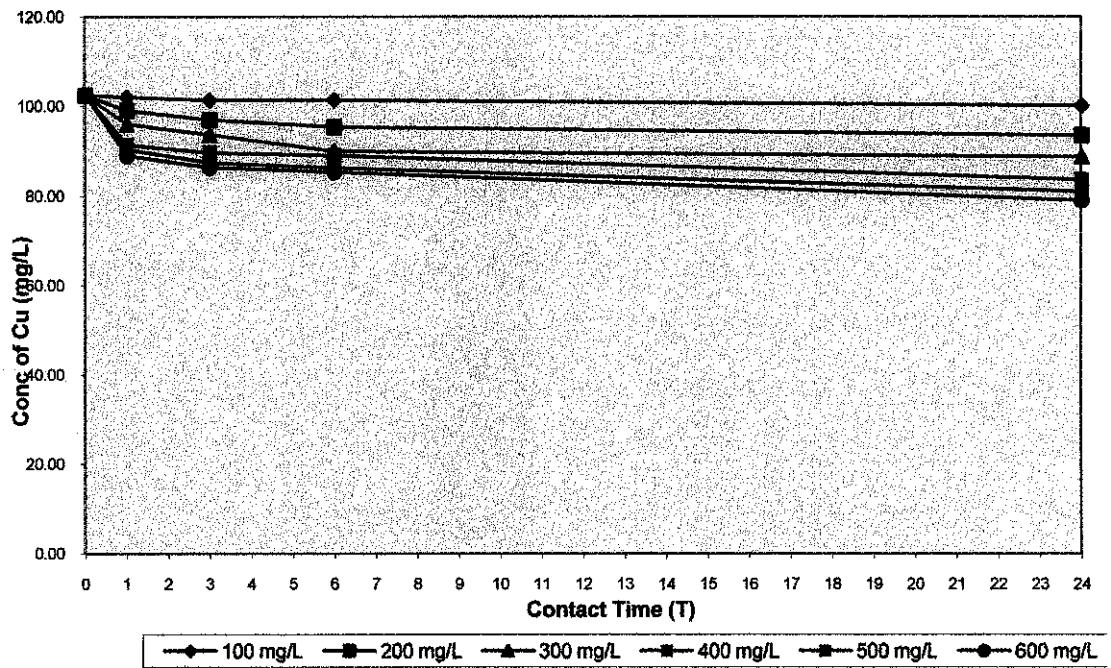
In the experiment studies carried on in order to determine the optimum sludge dosage, solutions with an initial copper concentration of 1, 10, and 100 mg/L were used at pH 4.3. During the contact time of 1, 3, 6, and 24 hours, the amount of dosage of iron sludge added to the solutions varied between 100, 200, 300, 400, 500, and 600 mg/L.



**Figure 4.12:** Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_o = 1.30$  mg/L.



**Figure 4.13:** Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_o = 10.2$  mg/L.

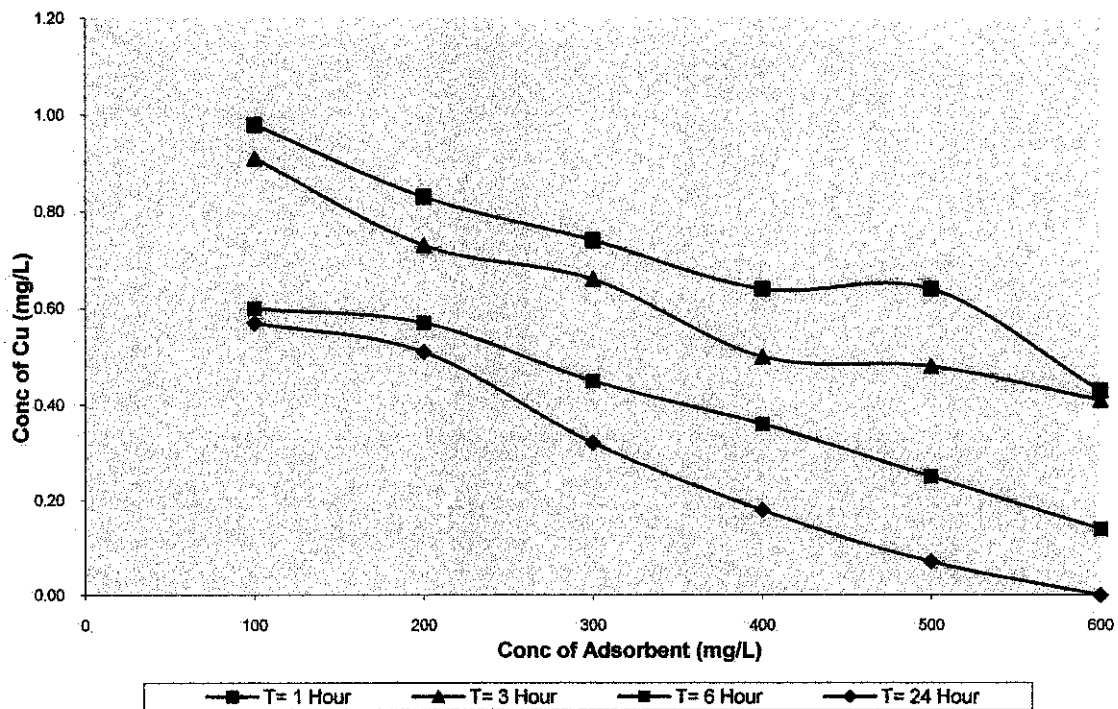


**Figure 4.14:** Influence of sludge dosage vs. contact time on sorption capacity of Copper at  $C_0 = 102.45$  mg/L.

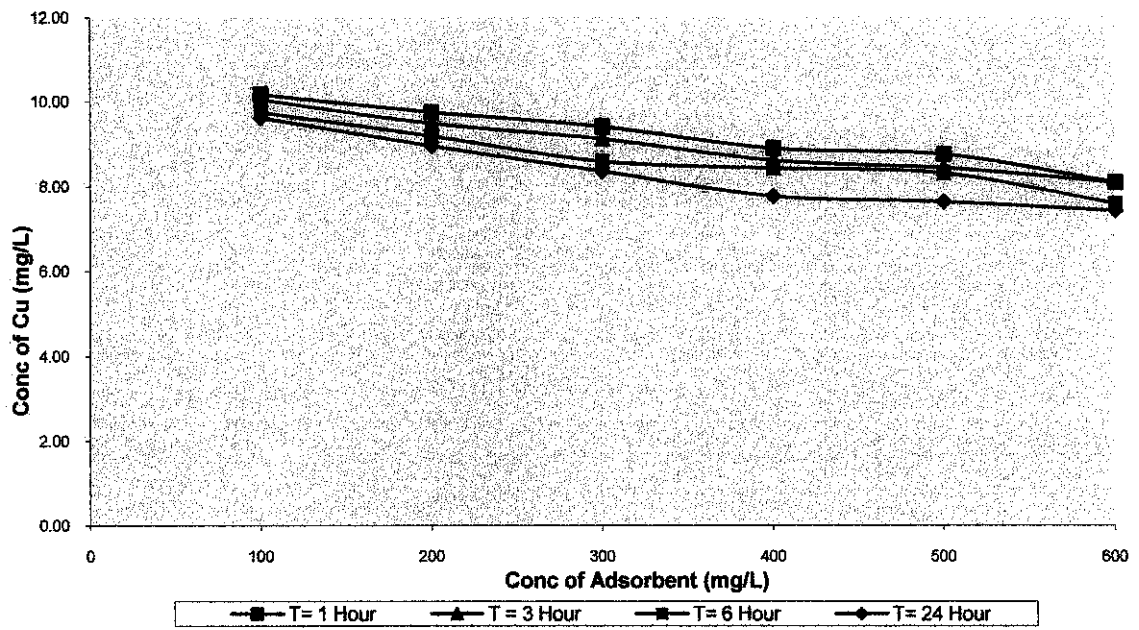
### 4.3.2 Experiment 3: Effect of Agitation Time

The agitation time was also evaluated as one of the most important factors affecting the adsorption efficiency (Sevil Veli and Bilge Alyuz, 2007). By using pH of 4.3, sludge waste and copper concentration, the effect of time on removal efficiency was analyzed. At the study carried out in a batch system, samples were taken at different time periods; 1, 3, 6, and 24 hours.

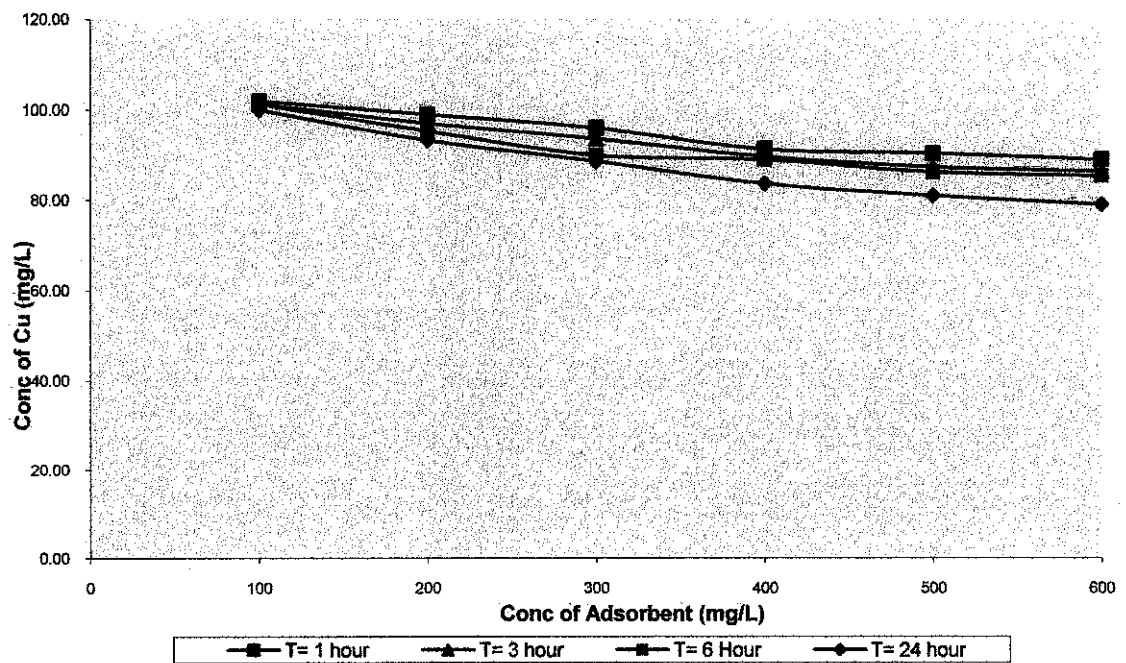
Figures show the removal efficiencies for 100, 200, 300, 400, 500, and 600 mg/L of sludge waste. It is evident that an increase in the contact time resulted in an increase in the removal efficiency of copper from solution.



**Figure 4.15:** Final Concentration of Copper vs. Sludge dosage for 1.3 mg/L copper solution.



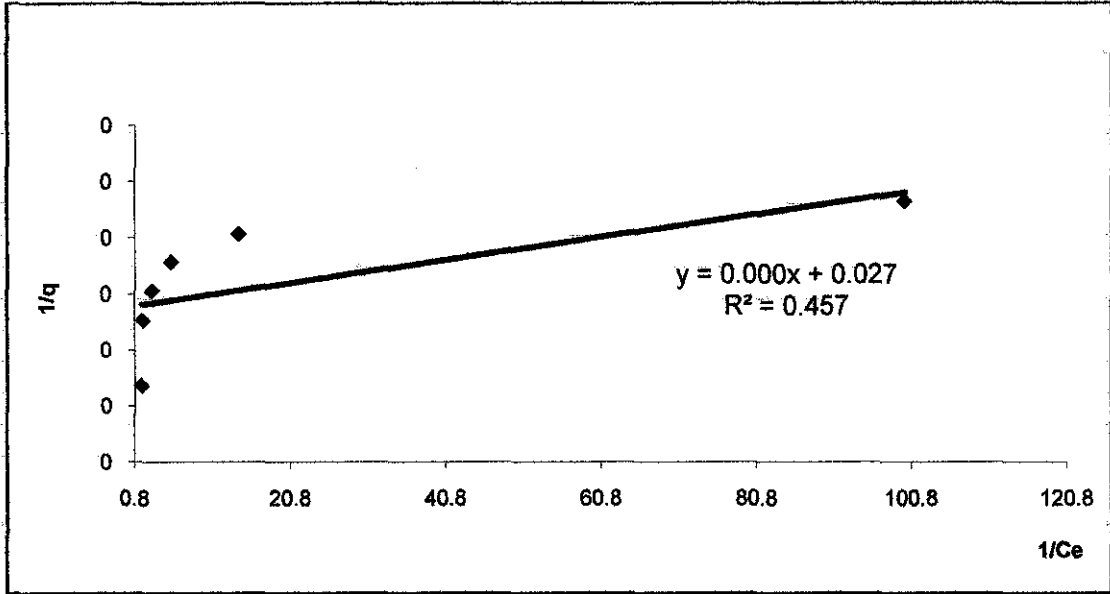
**Figure 4.16:** Final Concentration of Copper vs. Sludge dosage for 10.2 mg/L copper solution.



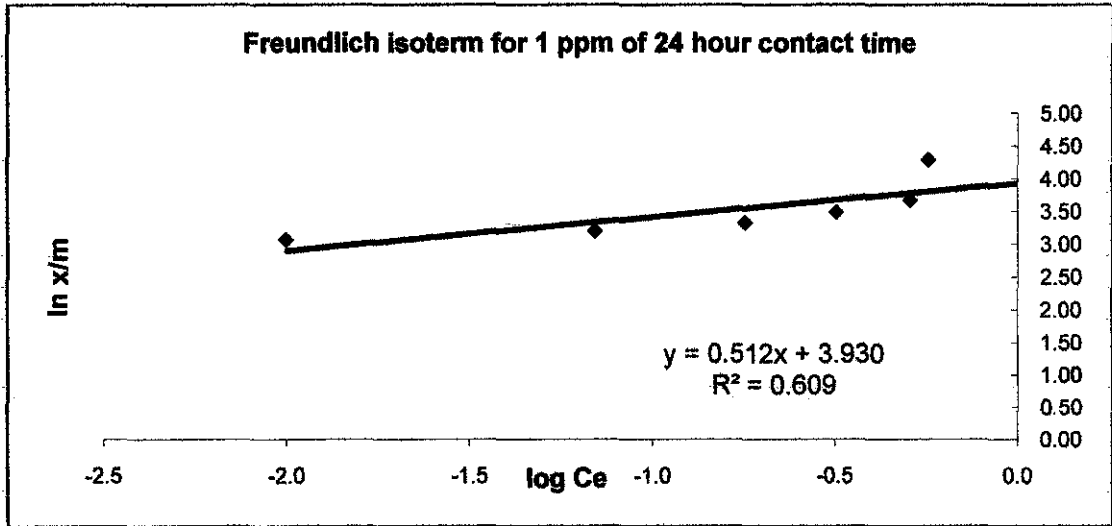
**Figure 4.17:** Final Concentration of Copper vs. Sludge dosage for 102.45 mg/L copper solution.



**4.3.3 Experiment 3: Langmuir Isotherm and Freundlich Isotherm.**



**Figure 4.18:** Langmuir isotherm for 1 ppm of 24 hours contact time.



**Figure 4.19:** Freundlich isotherm for 1 ppm of 24 hours contact time.

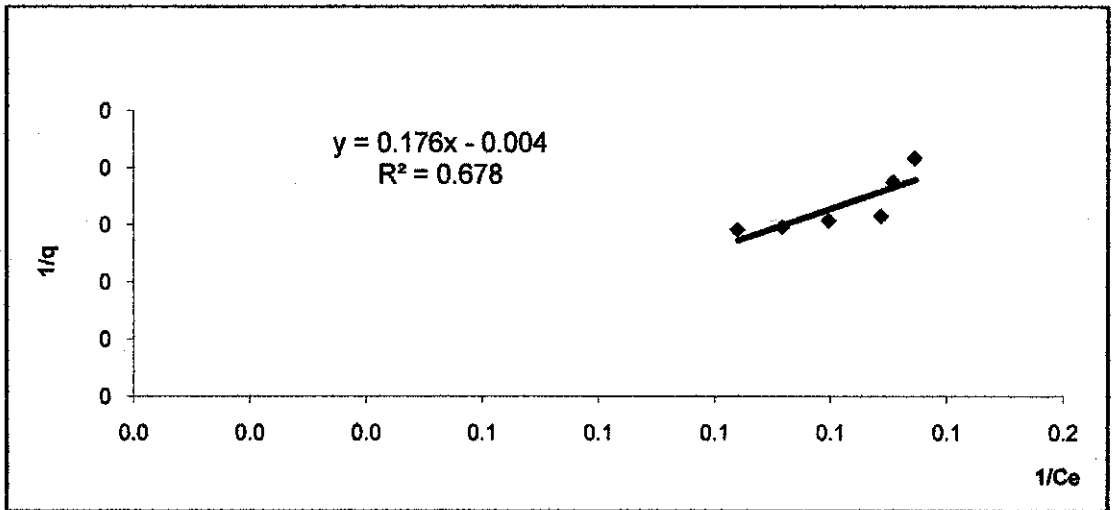


Figure 4.20: Langmuir isotherm for 10 ppm of 24 hours contact time.

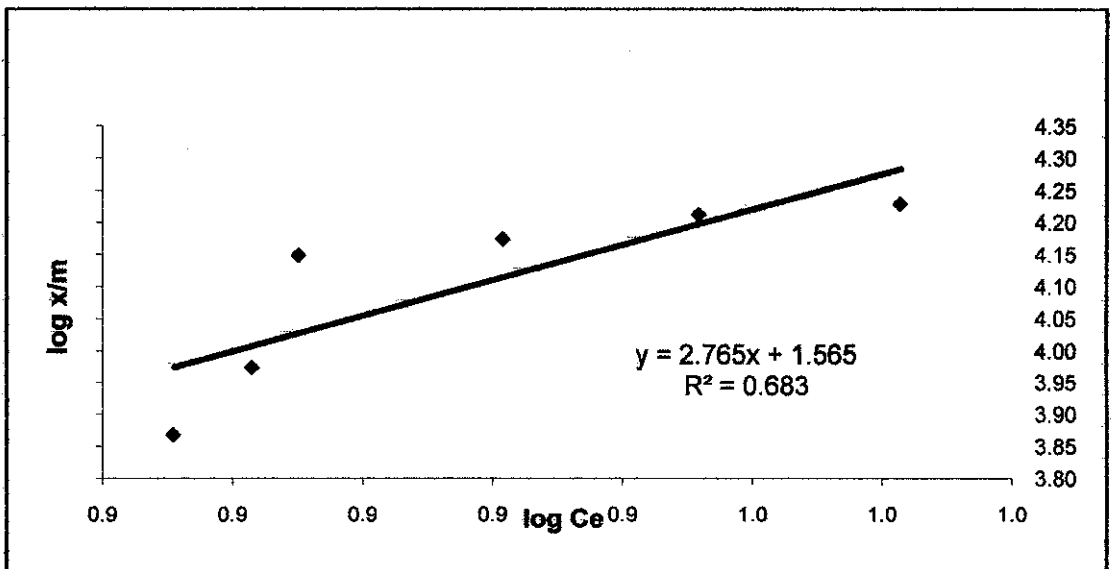


Figure 4.21: Freundlich isotherm for 10 ppm of 24 hours contact time.

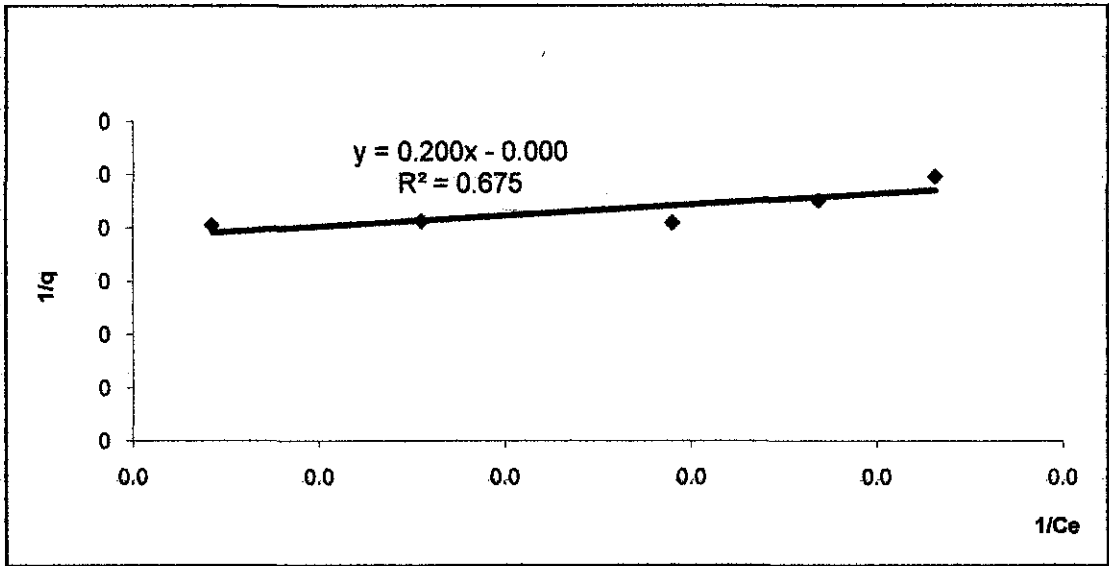


Figure 4.22: Langmuir isotherm for 100 ppm of 24 hours contact time.

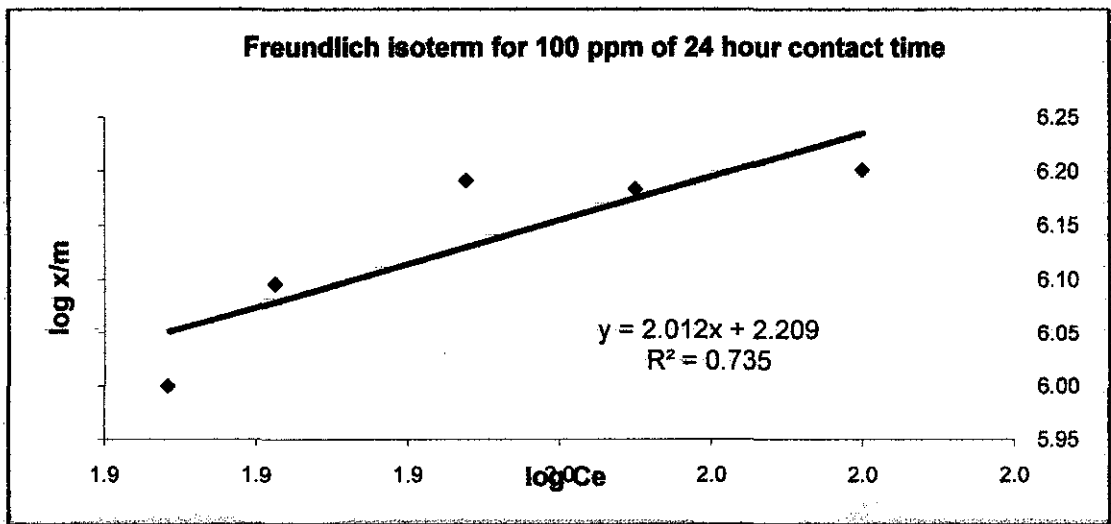


Figure 4.23: Freundlich isotherm for 100 ppm of 24 hours contact time

#### 4.3.4 Experiment 3: Discussion

In the third experiment which batch test were conducted with copper concentration of 1.30, 10.2, 102.45 mg/L. The desired dosages of sludge waste were: 10, 25, 50, 75, 100, and 125 mg.

The BOD bottles contents were agitated using an orbital shaker for the desired period (1, 3, 6 and 24 hours) at a speed of 150 rpm. Whatman No. 1 filter paper was used to filter the contents of the BOD bottles, followed by the analysis of Cu in the filtrate using atomic absorption spectrometry (AAS).

The maximum adsorption of 1.30 mg/L copper concentration is 100% in 24 hours agitation time with the 600 mg/L of sludge dosage. While the maximum adsorption of 10.2 mg/L copper concentration is 27.15% in 24 hours agitation time with 600 mg/L of sludge dosage. The 102.45 mg/L of copper concentration have the maximum adsorption of 22.88 % at 24 hours agitation and 600 mg/L sludge dosage.

The correlation coefficient of the Langmuir isotherm ( $R^2$ ) were 0.457 for 1 mg/L, 0.678 for 10 mg/L, and 0.675 for 100 mg/L copper concentration. While, the correlation of Freundlich isotherms ( $R^2$ ) were 0.609, 0.638 and 0.735 for 1, 10 and 100 mg/L copper concentration.

## CHAPTER 5: CONCLUSION AND RECOMMENDATION

### 5.1 Conclusions

Sludge waste, widely available in Malaysia, can be utilized as a low cost adsorbent for the removal of Cu from aqueous solutions. Sludge is generally the left over from any treatment facilities. It is disposed at landfills in the form of de-watered sludge or incinerator ash. Moreover, it is preserving the economic aspects of the treatment. In these studies, sludge waste produced from Ground Water Treatment Plant at Chicha, Kelantan was used as an adsorbent for copper ions in water.

The Cu adsorption capacity of sludge waste was favored at very low concentration and very short contact time. The amount of Cu adsorb increased with the increase in contact time and dosage of sludge. The Cu uptake is also attributes to different pores of grind sludge which being mixed in solution. During the adsorption process, metal ion passed through the pores of the sludge waste where the adsorption process occurred.

The adsorption process was carried out in a batch process and the effects of contact time (1–24 h), initial pH (4.0–6.0), initial metal ion concentration (1–100 mg L<sup>-1</sup>) and temperature (20–30 °C) on the adsorption were investigated. Experimental results showed that the maximum adsorption capacity was achieved at pH 4.3 and adsorbed Cu (II) ion concentration was increased with increasing initial metal concentration and contact time. The isothermal data could be described well by the Langmuir equations and Freundlich equations. The correlation coefficient of the Langmuir isotherm ( $R^2$ ) were 0.457 for 1 mg/L, 0.678 for 10 mg/L, and 0.675 for 100 mg/L copper concentration. While, the correlation of Freundlich isotherms ( $R^2$ ) were 0.609, 0.638 and 0.735 for 1, 10 and 100 mg/L copper concentration.

## **5.2 Recommendation**

After this study on copper ion complete, it is recommended to improve more on the methods. The initial and final pH of the copper solutions should be recorded to make sure the copper was removed by adsorption or by precipitation. Other recommendation is time of agitation, it should be varies frequently to have clearer view of copper removing activities. This is to get where is the exact point where the copper ions start to remove effectively. The studies also were recommended to use the effects of ground water treatment plant sludge on other metals such as, zinc, nickel, cadmium, and etc.

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W. W. Eckenfelder, Industrial Water Pollution Control, 3<sup>rd</sup> ed., McGrawHill Companies, 2000, pp. 417-460.

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**Calculation of copper nitrate**

FW Copper Nitrate = 241 g/mol

AW Copper = 63.54 g/mol

Copper/mol = 241/63.54

= 3.8 g/mol

**For 100 mg/L solution,**

=0.30 g of copper nitrate

**For 10 mg/L solution,**

=0.038 g

**For 1 mg/L solution,**

= 3.8 x 10<sup>-3</sup> g

APPENDIX 2-1

**Experiment 1: Absorption of 9.08 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
25	250	9.80	4.06	0.05	0.00	0.00
50	500	9.80	0.26	0.00	0.00	0.00
75	750	9.80	0.04	0.00	0.00	0.00
100	1000	9.80	0.00	0.00	0.00	0.00
200	2000	9.80	0.00	0.00	0.00	0.00
300	3000	9.80	0.00	0.00	0.00	0.00
350	3500	9.80	0.00	0.00	0.00	0.00
400	4000	9.80	0.00	0.00	0.00	0.00

**Table 1: Adsorption of 9.08 mg/L Copper Concentration**

**APPENDIX 2-2**

**Experiment 2: Absorption of 2.03, 15.08, 103.2 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
10	100	2.03	1.96	1.03	0.05	0.02
25	250	2.03	0.37	0.02	0.00	0.00
50	500	2.03	0.01	0.00	0.00	0.00
75	750	2.03	0.00	0.00	0.00	0.01
100	1000	2.03	0.00	0.00	0.00	0.00
125	1250	2.03	0.00	0.00	0.00	0.00

**Table 2.1: Adsorption of 2.03 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
10	100	15.08	15.80	13.74	13.60	10.73
25	250	15.08	9.18	6.55	5.88	2.26
50	500	15.08	0.43	0.61	0.53	0.08
75	750	15.08	0.21	0.13	0.05	0.02
100	1000	15.08	0.13	0.05	0.02	0.00
125	1250	15.08	0.03	0.03	0.02	0.00

**Table 2.2: Adsorption of 15.08 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
10	100	103.2	102.15	98.04	87.60	83.40
25	250	103.2	100.10	95.67	76.00	73.40
50	500	103.2	98.00	80.00	72.67	74.00
75	750	103.2	93.40	75.33	70.00	71.70
100	1000	103.2	92.30	71.67	67.00	67.67
125	1250	103.2	92.20	69.33	64.50	65.67

**Table 2.3: Adsorption of 103.2 mg/L Copper Concentration**

**APPENDIX 2-3**

**Experiment 3: Absorption of 1.30, 10.2, 102.45 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
10	100	1.30	0.98	0.91	0.60	0.57
20	200	1.30	0.83	0.73	0.57	0.51
30	300	1.30	0.74	0.66	0.45	0.32
40	400	1.30	0.64	0.50	0.36	0.18
50	500	1.30	0.64	0.48	0.25	0.07
60	600	1.30	0.43	0.41	0.14	0.00

**Table 3.1: Adsorption of 1.30 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
10	100	10.20	10.16	10.04	9.75	9.61
20	200	10.20	9.75	9.48	9.19	8.95
30	300	10.20	9.40	9.12	8.59	8.35
40	400	10.20	8.89	8.61	8.42	7.77
50	500	10.20	8.75	8.43	8.31	7.64
60	600	10.20	8.10	8.10	7.60	7.43

**Table 3.2: Adsorption of 10.2 mg/L Copper Concentration**

Weight of Absorbent (mg)	Conc. of Absorbent (mg/L)	Initial Concentration of Metal Ion Concentration	Average Final Concentration of Metal Ion Concentration			
			1 Hour	3 Hour	6 Hour	24 hour
10	100	102.45	102.00	101.33	101.33	100.00
20	200	102.45	99.00	97.00	95.33	93.33
30	300	102.45	96.00	93.67	90.00	88.67
40	400	102.45	91.33	89.67	89.00	83.67
50	500	102.45	90.40	87.43	86.26	81.00
60	600	102.45	89.00	86.40	85.40	79.00

**Table 3.3: Adsorption of 102.45 mg/L Copper Concentration**

Experiment 3: Langmuir Isotherms and Freundlich Isotherms.

Wt. of Adsorbent, m (mg)	Conc. Of Adsorbent, (mg/L)	Co, (mg/L)	Ce, (mg/L)	x = Co-Ce, mg	x/m (mg/g)	log x/m	log Ce	1/Ce	1/(x/m)
10	100	1.30	0.57	0.7300	0.07300	-1.1367	-0.2441	1.7544	13.6986
20	200		0.51	0.7900	0.03950	-1.4034	-0.2924	1.9608	25.3165
30	300		0.32	0.9800	0.03267	-1.4859	-0.4949	3.1250	30.6122
40	400		0.18	1.1200	0.02800	-1.5528	-0.7447	5.5556	35.7143
50	500		0.07	1.2300	0.02460	-1.6091	-1.1549	14.2857	40.6504
60	600		0.01	1.2900	0.02150	-1.6676	-2.0000	100.0000	46.5116

Table 4.1: Langmuir and Freundlich Isotherms of 1 ppm copper concentration at 24h

Wt. of Adsorbent, m (mg)	Conc. Of Adsorbent, (mg/L)	Co, (mg/L)	Ce, (mg/L)	X = Co-Ce, mg	x/m (mg/g)	log x/m	log Ce	1/Ce	1/(x/m)
10	100	10.30	9.61	0.6867	0.06867	-1.1633	0.9829	0.1040	14.5631
20	200		8.95	1.3500	0.06750	-1.1707	0.9518	0.1117	14.8148
30	300		8.35	1.9500	0.06500	-1.1871	0.9217	0.1198	15.3846
40	400		7.77	2.5333	0.06333	-1.1984	0.8902	0.1288	15.7895
50	500		7.64	2.6600	0.05320	-1.2741	0.8831	0.1309	18.7970
60	600		7.43	2.8700	0.04783	-1.3203	0.8710	0.1346	20.9059

Table 4.2: Langmuir and Freundlich Isotherms of 10 ppm copper concentration at 24h

Wt. of Adsorbent, m (mg)	Conc. Of Adsorbent, (mg/L)	Co, (mg/L)	Ce, (mg/L)	x = Co-Ce, mg	x/m (mg/g)	log x/m	log Ce	1/Ce	1/(x/m)
10	100	103.20	100.00	3.2000	0.32000	-0.4949	2.0000	0.0100	3.1250
20	200		93.33	9.8667	0.49333	-0.3069	1.9700	0.0107	2.0270
30	300		88.67	14.5333	0.48444	-0.3148	1.9478	0.0113	2.0642
40	400		83.67	19.5333	0.48833	-0.3113	1.9226	0.0120	2.0478
50	500		81.00	22.2000	0.44400	-0.3526	1.9085	0.0123	2.2523
60	600		79.00	24.2000	0.40333	-0.3943	1.8976	0.0127	2.4793

Table 4.3: Langmuir and Freundlich Isotherms of 100 ppm copper concentration at 24h

<b>JOB SAFETY ANALYSIS (JSA)</b>
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<b>Title of Job Operation</b>	Sieving	<b>Date</b>	7/1/2008	<b>Ref No</b>	FYP2/JSA/001
<b>Title of Person Who Does The Job</b>	Muhammad Afifi	<b>Employee</b>		None	
<b>Location</b>	Building 14	<b>Observed</b>			
<b>Department/Program</b>	Civil Engineering	<b>Prepared By</b>		Muhammad Afifi Che Rawi	
<b>Section/ Lab</b>	Highway Engineering Lab	<b>Approved by</b>		Ap. Dr. Shamsul Rahman Kutty	

<b>Sequence of Basic Job Step</b>	<b>Potential Accident or Hazard</b>	<b>Recommended Safe Job Procedure</b>
1. Sampling	1.1 Material Spill	1.1.1 Wear proper PPE (Lab Coat)
2. Shaking the sieving machine	2.1 High decibel sound.	2.1.1 Wear proper PPE (Ear Plug)
3. Collecting sample to beaker.	3.1 Irritation to respiratory	3.1.1 Wear proper PPE (Mask)

**JOB SAFETY ANALYSIS (JSA)**

<b>Title of Job Operation</b>	Prepare Copper Solution	<b>Date</b>	11/1/2008	<b>Ref No</b>	FYP2/JSA/002
<b>Title of Person Who Does The Job</b>	Muhammad Afifi	<b>Employee</b>	None		
<b>Location</b>	Building 14	<b>Observed</b>			
<b>Department/Program</b>	Civil Engineering	<b>Prepared By</b>	Muhammad Afifi Che Rawi		
<b>Section/ Lab</b>	Environmental Lab	<b>Approved by</b>	Ap. Dr. Shamsul Rahman Kutty		

<b>Sequence of Basic Job Step</b>	<b>Potential Accident or Hazard</b>	<b>Recommended Safe Job Procedure</b>
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Pouring distilled water to the basin.	2.1 Slip	2.1.1 Wear rubber shoes.
3. Diluting the Heavy Metal	3.1 Irritation	3.1.1 Wear gloves.

### JOB SAFETY ANALYSIS (JSA)

<b>Title of Job Operation</b>	Prepared BOD Bottle	<b>Date</b>	11/2/2008	<b>Ref No</b>	FYP2/JSA/003
<b>Title of Person Who Does The Job</b>	Muhammad Afifi	<b>Employee Observed</b>			none
<b>Location</b>	Building 14				
<b>Department/Program</b>	Civil Engineering	<b>Prepared By</b>	Muhammad Afifi Che Rawi		
<b>Section/ Lab</b>	Environmental Lab	<b>Approved by</b>	Ap. Dr. Shamsul Rahman Kutty		

<b>Sequence of Basic Job Step</b>	<b>Potential Accident or Hazard</b>	<b>Recommended Safe Job Procedure</b>
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper PPE (lab coat)
2. Pour Heavy Metals	2.1 Spill material to body	2.1.1 Wear proper PPE (lab coat)
		2.1.2 Wear Proper PPE (Gloves)



**JOB SAFETY ANALYSIS (JSA)**

<b>Title of Job Operation</b>	Shaking BOD Bottle	<b>Date</b>	16/1/2008	<b>Ref No</b>	FYP2/JSA/004
<b>Title of Person Who Does The Job</b>	Muhammad Afifi	<b>Employee</b>	none		
<b>Location</b>	Building 14	<b>Observed</b>			
<b>Department/Program</b>	Civil Engineering	<b>Prepared By</b>	Muhammad Afifi Che Rawi		
<b>Section/ Lab</b>	Environmental Lab	<b>Approved by</b>	Ap. Dr. Shamsul Rahman Kutty		

<b>Sequence of Basic Job Step</b>	<b>Potential Accident or Hazard</b>	<b>Recommended Safe Job Procedure</b>
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Inset BOD to shaker.	2.1 Spill material to body	2.1.1 Wear proper ppe (lab coat)
	2.2 Spill material to eye	2.2.1 Wear goggle if necessary
	2.3 Electric Shock	2.3.1 Keep hands dry before on the switch.
	2.4 Slip	2.4.1 Wear proper PPE (Rubber Shoes)

## JOB SAFETY ANALYSIS (JSA)

<b>Title of Job Operation</b>	Filter	<b>Date</b>	16/2/2008	<b>Ref No</b>	FYP2/JSA/005
<b>Title of Person Who Does The Job</b>	Muhammad Afifi	<b>Employee</b>	none		
<b>Location</b>	Building 14	<b>Observed</b>			
<b>Department/Program</b>	Civil Engineering	<b>Prepared By</b>	Muhammad Afifi Che Rawi		
<b>Section/ Lab</b>	Environmental Lab	<b>Approved by</b>	Ap. Dr. Shamsul Rahman Kutty		

<b>Sequence of Basic Job Step</b>	<b>Potential Accident or Hazard</b>	<b>Recommended Safe Job Procedure</b>
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2 Filtering process	2.1 Irritation	2.1.1 Wear proper glove
		2.1.2 Wear proper PPE (lab coat)
		2.1.3 Wear proper PPE (mask)

**JOB SAFETY ANALYSIS (JSA)**

<b>Title of Job Operation</b>	AAS	<b>Date</b>	19/2/2008	<b>Ref No</b>	FYP2/JSA/006
<b>Title of Person Who Does The Job</b>	Muhammad Afifi	<b>Employee Observed</b>		none	
<b>Location</b>	Building 14				
<b>Department/Program</b>	Civil Engineering	<b>Prepared By</b>	Muhammad Afifi Che Rawi		
<b>Section/ Lab</b>	Environmental Lab	<b>Approved by</b>	Ap. Dr. Shamsul Rahman Kutty		

<b>Sequence of Basic Job Step</b>	<b>Potential Accident or Hazard</b>	<b>Recommended Safe Job Procedure</b>
1. Sampling	1.1 Spill material to body	1.1.1 Wear proper ppe (lab coat)
2. Prepare Standards Solution	2.1 Irritation	2.1.1 Wear proper glove
		2.1.2 Wear proper PPE (lab coat)
		2.1.3 Wear proper PPE (mask)
3. AAS	3.1 Heat	3.1 Wear goggle
	3.2	