# Column Study for Zn, Fe and Cu Removal from Single Metal Aqueous Solutions Using Microwave Incinerated Rice Husk Ash (MIRHA)

by

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**DISSERTATION REPORT**

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CERTIFICATION OF APPROVAL

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Muhammad Ezree bin Esmadee

A project dissertation submitted to the
Civil Engineering Programme
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Approved by,

____________________________

(AP. Dr. Mohamed Hasnain Isa)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
Sept 2012

# 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.

MUHAMMAD EZREE BIN ESMADEE

# ABSTARCT

Industrial wastewaters frequently contain high levels of heavy metals and treatment is needed before disposal, in order to avoid water pollution. Numerous sources of industrial effluents leading to heavy metal discharges apart from the mining and metal related industries. Research in recent years has focused on the use of rice husk and rice husk ash including numerous by product of agriculture for heavy metals removal. Since rice is the main food source in Malaysia with more than 2.4 million tonnes of paddy produced in Malaysia each year, it is possible rice husk ash can be utilized as an alternative to existing activated carbon. This project is study on column for Zn, Fe and Cu removal from single metal aqueous solutions using Microwave Incinerated Rice Husk Ash (MIRHA). The experiment proposed to assess the effect of using MIRHA as an adsorbent to remove heavy metals in single metal aqueous solution. The objectives of this proposed project are to obtain the breakthrough curves for zinc iron and copper aqueous solutions. The methodology of the study is experimental research. The variable in the proposed experiments are column depth which are 20 cm, 30 cm and 40 cm. Mass of MIRHA used for each column depth were also different which are 3.33 g, 5.00 g and 7.00 g. All experiment were conducted with a flow rate of 5 mL per minute and all results were analyzed using atomic adsorption spectrophotometer (AAS) machine. The results show that the breakthrough time obtained for 40 cm bed depth for zinc, iron and copper were 54 minutes, 70 minutes and 106 minutes. In conclusion, MIRHA has the ability to absorb heavy metal in a single aqueous solution.

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Water is a vital element for every living life form. Water covers 90 percent of the Earth, or approximately 510 million kilometres square of the Earth surface is covered by water. From the vast volume of water on Earth, only 0.5 percent is considered fresh water. Because of that, it is the responsibility of human being to take care of water and avoid it from being polluted. In the old days, the importance of water was only focused on the quantity. Our ancestors depended on the physical properties of water which are odour, colour and taste to determine the quality.

Nowadays, water quality can be determined through physical, chemical and biological properties from laboratory or in situ test (Hurley et al, 2012). Ecosystem will be harmed if polluted water is left untreated. An example of water pollution killing the ecosystem was the minamata disease where industrial wastewater containing methyl mercury was dumped into the river and killed thousands of people as well as aquatic life and other animals. Hence, in order to avoid water pollution, it is necessary to treat the wastewater according to the discharge standard before it is discharged.

Industrial wastewaters frequently contain high levels of heavy metals and treatment is needed before disposal, in order to avoid water pollution. There are numerous sources of industrial effluents leading to heavy metal discharges apart from the mining and metal related industries. Heavy metal pollution frequently results from the industrial use of organic compounds containing metal additives in the petroleum and organic chemical industries, e.g. textile mill products (Cr), organic chemicals

(Cr, Pb), petroleum refining, pulp industries and fertilizers (Cr, Cu, Pb), iron and steel manufacturing plants (Fe) (Inglezakis et al, 2004).

## 1.2 Problem Statements

The rapid economic growth causes the development of various industrial sectors. Total population increase also led to the increment of municipal solid waste. Due to this factor and rapid economic growth, it leads in increasing production and usage of toxic chemicals which direct or indirectly leads to the increment of heavy metals level in the river or sea which had been dump into the ecosystem without proper treatment (Singh et al, 2010). Apart from that, it is crucial to maintain the marine environment at pristine levels since extensive industrialization and urbanization have led to a strong risk of heavy metal contamination in many coastal environments around the world including Peninsular Malaysia (Yap et al, 2010).

Department of Environment Malaysia reported consistently much higher concentrations of heavy metals in rivers of the littoral states of the Straits of Malacca than in other parts of the country (United Nations, 2004). Admittedly, this is due to extensive land use and industrialization, especially in Penang, Perak, Selangor and Malacca. Hence, this research was aimed to study the feasibility of using microwave incinerated rice husk ash as a low cost material for heavy metal removal. Rice milling industry in Malaysia generates huge amount of rice husk during milling process. Most of the rice husk is served as fuel in the boiler for paddy processing. The rice husk ash created from the process is approximately 25 percent by weight when burnt. This huge amount of rice husk ash becomes a great environmental hazard and may cause harm to the surrounding area where it is dumped. Since rice is the main food source in Malaysia and yearly more than 2400 000 tonnes of paddy is produced in Malaysia, rice husk ash can be utilized as an alternative to commercial activated carbon (Johan, 2011).

## 1.3 Objectives

The aim of this project is column study with varying the bed height to remove different ions from wastewater using MIRHA. As the aim mentioned above, the objectives of study are the following below:

1. To study the feasibility of using MIRHA as adsorbent for removal of iron, copper and zinc.

2. To obtain breakthrough curves of different heavy metals by varying the height of the column bed.

## 1.4 Project Scope

This project is focused on the column studies for Zn, Fe and Cu removal from synthetic wastewater using Microwave Incinerated Rice Husk Ash (MIRHA). The experimental research had been conducted at environmental lab, Block 14 at Civil Engineering Department, Universiti Teknologi PETRONAS, PERAK, MALAYSIA.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Definition of Heavy Metal

A heavy metal is a member of an ill defined subset of elements that exhibit metallic properties. Heavy metals include the transition metals, some metalloids, lanthanides, and actinides. Many different definitions have been proposed which some based on density, some on atomic number or atomic weight, and some on chemical properties or toxicity (Duffus, 2002). The term heavy metal has been called a "misinterpretation" due to the contradictory definitions and its lack of a "coherent scientific basis". There is an alternative term toxic metal, for which no consensus of exact definition exists either. As discussed below, depending on context, heavy metal can include elements lighter than carbon and can exclude some of the heaviest metals. Heavy metals occur naturally in the ecosystem with large variations in concentration. In this day and age, anthropogenic sources of heavy metals, i.e. pollution, have been introduced to the ecosystem. Waste-derived fuels are especially prone to contain heavy metals, so heavy metals are a concern in consideration of waste as fuel (Roy, 2010).

## 2.2 Relationship with Living Organisms

Living organisms require varying amounts of “heavy metals”. Iron, cobalt, copper, manganese, molybdenum, and zinc are required by humans. Excessive levels can be damaging to the organisms, other heavy metals such as mercury, plutonium and lead are toxic metals that have no known vital or beneficial effect on organisms, their accumulation over time in the bodies of animals can cause serious illness, certain elements that are normally toxic for certain organisms under certain conditions, beneficial. Examples include vanadium, tungsten, and even cadmium. Metal pollution of the world’s waters continues to pose a serious threat to the health of man. Some workable systems for the extraction of these metals from the environment must be devised.

Pollution of heavy metals in aquatic ecosystem is growing at an alarming rate and has become an important problem worldwide (Idriss and Ahmad, 2012). Heavy metals including both essential and non-essential elements have a particular significance in ecotoxicology, since they are highly persistent and all have the potential to be toxic to living organisms (Storelli et al., 2005). Heavy metals do not exist in soluble forms for a long time in waters, they are present mainly as suspended colloids or are fixed by organic and mineral substances (Idriss and Ahmad, 2012). Water pollution by heavy metals is one of the main types of pollution in aquatic ecosystems and may pressure the biotic community. The rapid economic growth has resulted in increasing production and usage of toxic chemicals such as trace elements in Malaysia (Tetsuro et al., 2005).

## 2.3 Categorization of Heavy Metals

Heavy metals can be divided into two different categories which are:

### 2.3.1 Essential Heavy Metals

Essential heavy metals are the ones needed by the body, though only small amount are needed, it is vital for the proper function on various biological systems. A heavy metal is term essential if its deficiency results in the impairment of a function (Harper, 2007). Example of essential trace elements includes iron, zinc, copper, calcium and manganese.

### 2.3.2 Non Essential Heavy Metals

There are some heavy metals which exist in human body which are identified as non essential to the body. These metals are harmless if they does not exceed their threshold levels. These metals include chromium, silicon, nickel etc. The non essential heavy metals are of two types:

#### 2.3.2.1 Non-Toxic non Essential Heavy Metals

These heavy metals have no function in the human body. Nevertheless, it will become a burden to the body when it reaches the biological systems (Roy, 2010). Nickel and Silicon are examples of non toxic non essential heavy metals.

#### 2.3.2.2 Toxic non Essential Heavy Metals

Examples of toxic metals are mercury, lead, arsenic, cadmium and even in small quantities, these metals are toxic in the human body. These types of metals are added to the aquatic systems through industrial wastes from steel factories alongside fungicides and herbicides.

## 2.4 Heavy Metal Treatment Techniques

Faced with more and more strict regulations, nowadays heavy metals are the environmental priority pollutants and are becoming one of the most serious environmental problems. So these toxic heavy metals should be removed from the wastewater to protect the people and the environment. Wang (2011) stated that, among types of heavy metal treatment techniques are chemical precipitation, ion exchange, adsorption, membrane filtration, coagulation and flocculation, floatation and electrochemical treatment. However, though many techniques are available for heavy metal treatment, selection for most suitable treatment depends on the initial metal concentration, the component of the wastewater, capital investment and operational cost, plant flexibility and reliability and environmental impact (Kurniawan, 2006).

## 2.5 Adsorption

Adsorption is now recognized as an effective and economic method for heavy metal wastewater treatment. The adsorption process offers flexibility in design and operation and in many cases will produce high-quality treated effluent (Wang, 2011). In addition, because adsorption is sometimes reversible, adsorbents can be regenerated by suitable desorption process

### 2.5.1 Bioadsorbents

Modern studies have confirmed a very promising process in the removal of heavy metal from aqueous solutions by biosorption. High effectiveness and more economical comparing with the existing activated carbon are the main advantages of bioadsorbents. Main types of biosorbents are from three sources non living biomass, algal biomass and microbial biomass (Apiratikul and Pavasant, 2008). Other forms of cheap plant material which had been use in heavy metal adsorbent studies are potato peels, sawdust, coffee husk, citrus peel and many more.

## 2.6 Microwave Incinerated Rice Husk Ash

Research in recent years has focused on the use of some natural biomaterials including agricultural products and by-products for heavy metals removal (Isa et al., 2012). Since rice is the main food source in Malaysia with more than 2400 000 tonnes of paddy is produced in Malaysia each year, it is possible rice husk ash can be utilized as an alternative to commercial activated carbon (Johan, 2011). Rice milling industry in Malaysia generates a lot of rice husk during milling of paddy which comes from the field. This rice husk is mostly used as a fuel in the boilers for processing paddy. Rice husk ash is about 25 percent by weight of rice husk when burnt in the Universiti Teknologi Petronas Microwave Inccinerator (UTPMI) (Nuruddin et al., 2008). It is estimated that approximately 70 million tonnes of rice husk ash is produced annually worldwide and rice husk ash is a great environment threat causing damage to the land and surrounding area in which it is dumped (Singhania, 2008). Rice husk ash, which is developed from the burning of rice husk at a certain temperature, has been used for cementing material and also has good adsorptive properties (Johan, 2011). Table 2.1 below shows chemical composition of MIRHA.

**Table 2.1:** Chemical composition of MIRHA (Source: Nuruddin et al., 2008)

| Chemical Composition of MIRHA | |
|---|---|
| **Oxide** | **Percentage** |
| **$Na_2O$** | 0.12 |
| **MgO** | 0.49 |
| **$Al_2O_3$** | 0.45 |
| **$SiO_2$** | 89.34 |
| **$P_2O_5$** | 2.58 |
| **$K_2O$** | 0.76 |
| **CaO** | 0.76 |
| **$TiO_2$** | 0.02 |
| **$Fe_2O_3$** | 0.40 |
| **$SO_3$** | 0.90 |
| **MnO** | 0.08 |

## 2.7 Fluidized-Bed Reactor

In fluidized-bed reactor, the wastewater flows upward to a bed of 0.4 mm to 0.5 mm sand or activated carbon. Bed depths are usually in the range of 3m to 4m. The specific surface area is about 1000 $m^2/m^3$ of reactor volume, which is greater than any of the other fixed film packing (Metcalf and Eddy, 2004). Based on adsorption isotherm, up flow velocities will be determined based on the adsorbent used, effluent recirculation is necessary to provide the fluid velocity within the necessary treatment detention time. Aerobic fluidized bed reactors are frequently used to treat groundwater contaminated with hazardous substances such as heavy metals. In these applications activated carbon is used for the packing to provide both carbon adsorption and biological degradation (Ghosh and Ullhyan, 2012).

## 2.8 Column Studies

Many previous researchers had conducted column studies in determining the adsorption of newly use material to act as adsorbents such as rice husk, coffee husk, fly ash and etc. Mohan and Sreelakshmi (2008) had study on removal of lead, copper, zinc and manganese based on column studies. They manage to obtained breakthrough curves for the metals and adopted modified Adam Bohart isotherm in determining bed depth service time for a full scale fixed bed column reactor. Other than Mohan and Sreelakshmi (2008), Eisazadeh also conducted column studies on heavy metal removal using nanometer size polypyrrole coated on rice husk ash in 2012 and the results of her research are shown in Table 2.2 below:

**Table 2.2:** Eisazadeh's breakthrough time on her column studies

| Metal | Bed depth | Breakthrough time (min) | Treated volume (mL) | Total mass of nanocomposite |
|---|---|---|---|---|
| Mn | 10 | 60 | 60 | 1.65 |
| | 20 | 180 | 180 | 2.95 |
| | 30 | 270 | 270 | 4.54 |
| Cu | 10 | 90 | 90 | 1.65 |
| | 20 | 120 | 120 | 2.95 |
| | 30 | 180 | 180 | 4.54 |
| Fe | 10 | 30 | 30 | 1.65 |
| | 20 | 60 | 60 | 2.95 |
| | 30 | 120 | 120 | 4.54 |
| Zn | 10 | 30 | 30 | 1.65 |
| | 20 | 60 | 60 | 2.95 |
| | 30 | 90 | 90 | 4.54 |

Many previous researcher's experimental theory and set up were similar in their method of conducting research, hence, the author has use almost similar experimental set up used by Eisazadeh (2012) to conduct this project but with different type of adsorbent to run the experiment which was microwave incinerated rice husk ash.

# CHAPTER 3

# METHODOLOGY

## 3.1 Preparation of MIRHA

Rice husk samples were obtained from BERNAS Rice Mill in Seberang Perak, Malaysia. The samples were burned using The UTP Microwave Incinerator (UTPMI) at temperature of 800°C. The UTPMI used in the research adopted the Air Cooled Magnetron system with an overall dimension of 2.3(H)x4.0(W)x4.0(L) with a chamber capacity of 1 m³ (Nuruddin, 2008). The MIRHA was soaked with 10% of HCl for 24 hours before it was rinsed with distilled water and oven dried at 105°C for 24 hours.

**Figure 3.1:** UTPMI (Source: Nuruddinet al., 2008)

### 3.2 Preparation of Reagents and Standard Solutions

All chemicals used were reagent grade. Stock solutions of 10 mg/L were prepared by dissolving 10 mL of the standard solutions for each metal into 1 litre volumetric flask and dilute with deionized water. Distilled deionized water was used throughout this work.

### 3.3 Experimental Methodology

The column studies were conducted using 1cm inner diameter column with three different length which were 20 cm, 30 cm and 40 cm. Cotton plug was inserted at the bottom of each column to support the MIRHA as well as at the top of the column in order to prevent floating up of adsorbent. In this experiment, the influent was fed upward into the column by peristaltic pump at a flow rate of 5mL/min. After running through the column, the effluent was transported to a storage tank where samples were collected with a time interval of 10 minutes for the first six samples and 15 minutes time interval for the next eight samples and 30 minutes for the rest of the samples afterwards. Before the experiment was started using heavy metal solution, the experimental set up was run with deionized distilled water to remove bubbles if existed along the experimental set up. The schematic diagram of the experimental set up is shown in Figure 3.2.

**Figure 3.2:** Schematic diagram of experimental set up

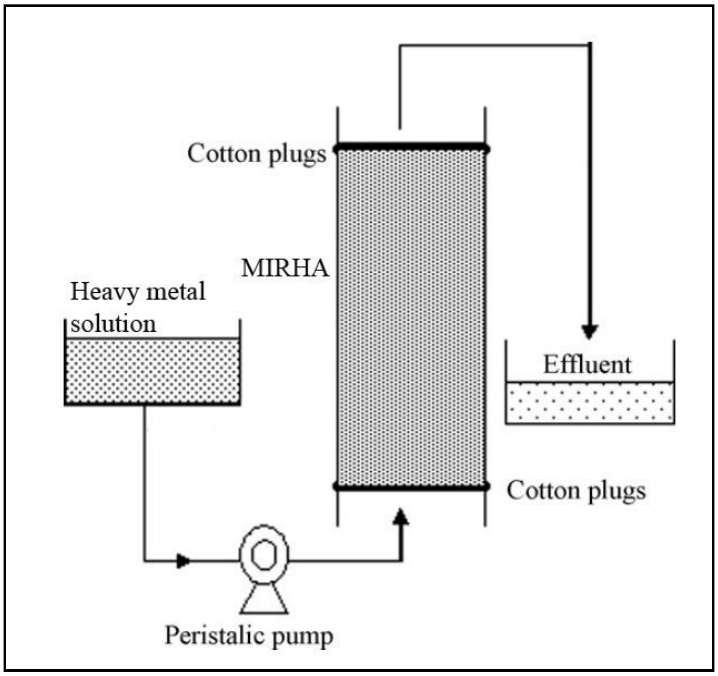


Figure 3.3 shows the experimental set up which the author had used in this project.

**Figure 3.3:** Experimental set up

The effective column height, area, initial concentration of metal ion, diameter of the column, weight of adsorbent taken and flow rate are listed in Table 3.1.

**Table 3.1:** Specification of experimental studies

| Number | Parameters | Specification |
|---|---|---|
| **1** | Column bed height | 20 cm, 30 cm, 40 cm |
| **2** | Inner diameter of the column | 1 cm |
| **3** | Area of the column | 0.79 $cm^2$ |
| **4** | Weight of adsorbent used | 3.33 g, 5.00 g, 7.00 g |
| **5** | Volume of the column | 15.71 $cm^3$, 23.56 $cm^3$, 31.42 $cm^3$ |
| **6** | Volume of metal solution | 2 L |
| **7** | Initial concentration | 10 mg/l |
| **8** | Flow rate | 5 ml/min |

### 3.4 Laboratory Test

For analyzing results of the effluent, atomic adsorption spectrophotometer (AAS Shimadzu model AA 6800, Japan) was used in the experiment. All rules and regulations as well as standard procedure in handling the AAS were strictly followed. Figure 3.4 below shows AAS machine at environmental laboratory, Block 14, UTP.


**Figure 3.4:** AAS machine at environmental laboratory Block 14 UTP

# CHAPTER 4

# RESULTS AND DISCUSSION

This section discusses the results obtained from the experimental work. Characterization of the MIRHA and its surface morphology has an effect on the adsorbent rate. Physical and chemical properties of MIRHA will be discussed in this chapter as well as the results obtained. All effluent has been analyzed using atomic adsorption spectrophotometer (Shimadzu model AA 6800, Japan) and the results shows remaining heavy metal concentration which exists in the effluent with respect to time. Discussion on this experiment can be divided into several categories which are:

i. Discussion based on the physical and chemical properties of MIRHA.

ii. Discussion based on breakthrough curves obtained with varying the bed height for Zinc, iron and copper removal in synthetic wastewater solution.

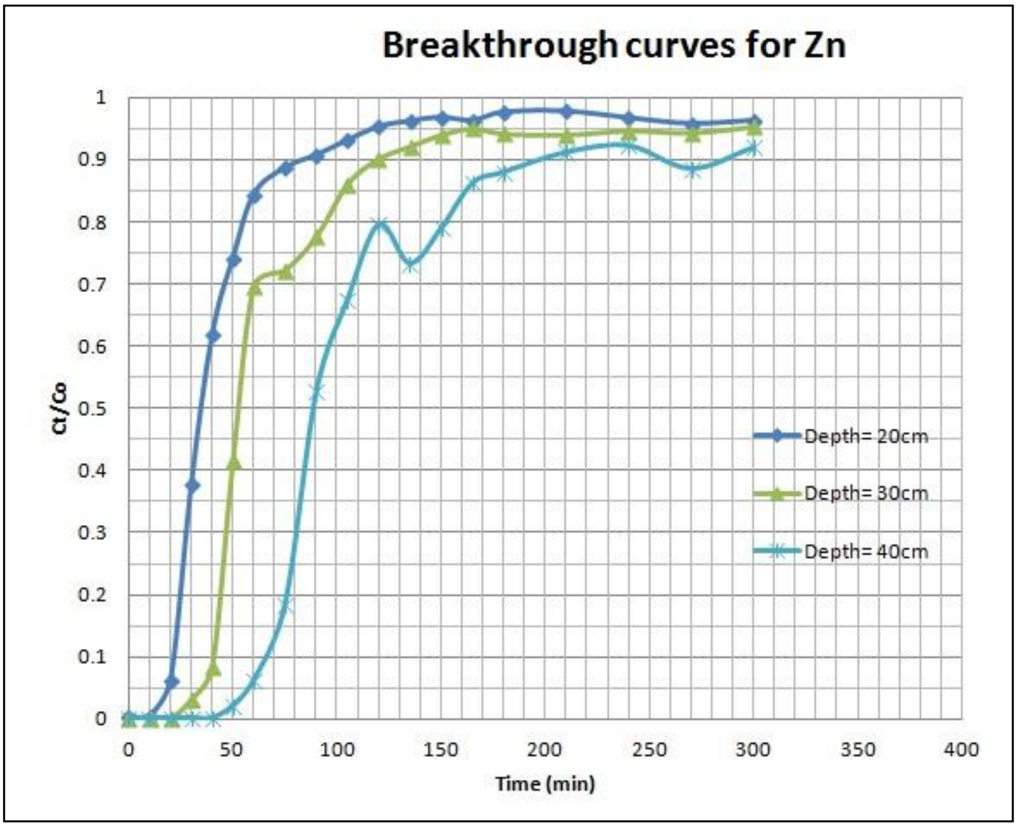
iii. Discussion on the effectiveness of using MIRHA as adsorbent of heavy metal for removal of Zinc, iron and copper.

For the results and discussion in this chapter, all value for heavy metals removal were use in percentage as to give a better view and understanding of the results obtained.

### 4.1 Behaviour of Adsorption Column for Individual Metal Solutions

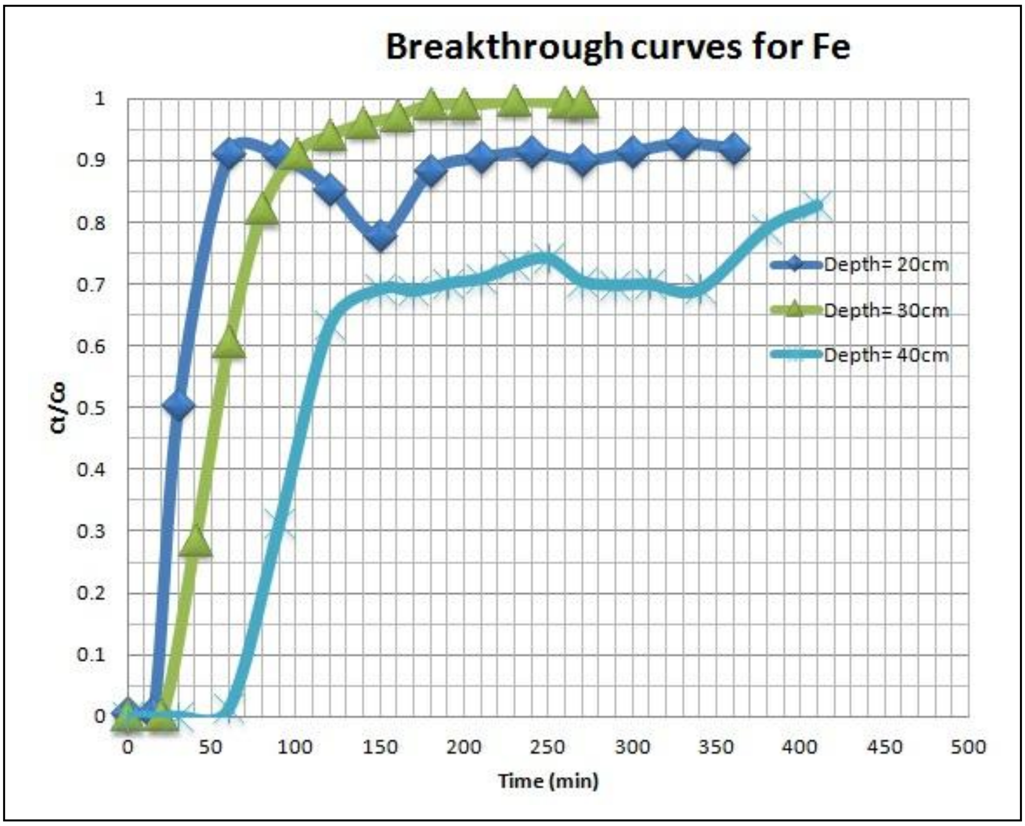
From the results obtained by the column experiments, the breakthrough curves are plotted for Zn, Fe and Cu solutions and are shown in Figures 4.1, 4.2, and 4.3 respectively. Breakthrough is said to have occurred when the effluent concentration reaches five percent of the influent value. From the breakthrough curves obtained, we can observe that the metal uptake capacity is increased with the increase of bed height. This is due to the increase metal solutions contact time with the adsorbent in the column. All graphs obtained were S-curve graph as the metal adsorption capacity by MIRHA is decreased when it reached the breakthrough time.

**Figure 4.1:** Breakthrough curves for Zn



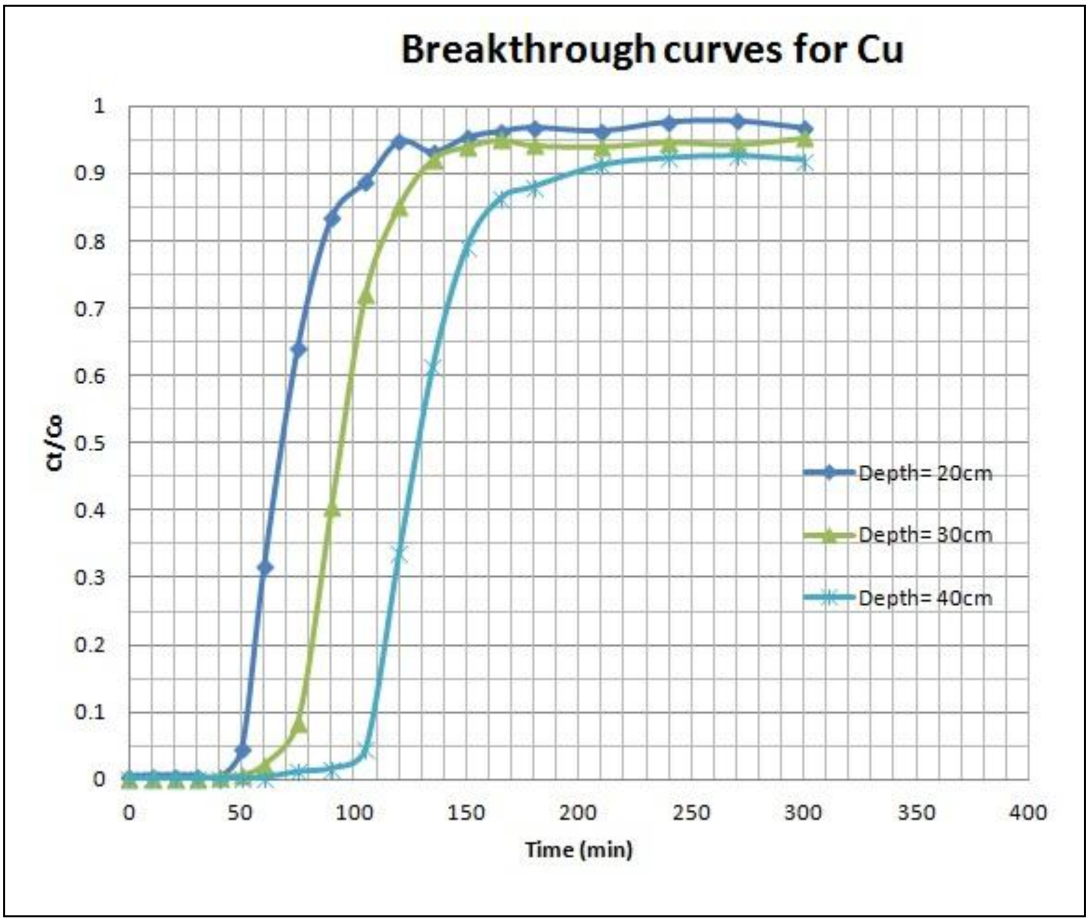

Based on Figure 4.1, we can see that the shape of breakthrough curves for 20 cm, 30 cm and 40 cm bed depth of Zn solution were S-shape graph. Breakthrough time for 20 cm bed depth was 18 minutes with 3.33 g of adsorbent used, breakthrough time for 30 cm bed was 35 minutes with 5.00 g of adsorbent and breakthrough time for 40 cm bed was 54 minutes with 7.00 g of adsorbent used.

**Figure 4.2:** Breakthrough curves for Fe




From the breakthrough curves graph of Fe (Figure 4.2), we can see that the shape of the lines were similar to the breakthrough curves graph of Zn. The breakthrough time for 20 cm, 30 cm and 40 cm bed of Fe aqueous solution were 18 minutes, 35 minutes and 70 minutes respectively. By comparison with breakthrough time of Zn, it shows that, for 20 cm and 30 cm bed depth, the breakthrough time obtained for each individual metal solution were the same which were 18 minutes and 35 minutes. However, for 40 cm bed depth column, the different of breakthrough time between Zn and Fe aqueous solution were 16 minutes where Fe was adsorb by MIRHA at a higher rate compare to Zn. As stated by Mohsen (2012), in his journal of fixed bed column study, it is due to the ionic radius and electro positive charges of the ions which extend the breakthrough time of Fe compare to Zn.

**Figure 4.3:** Breakthrough curves for Cu




From Figure 4.3, we can see that the shape of the curves obtained were S-curve which were similar to breakthrough curves of Zn and Fe. Comparing the breakthrough time obtained for 20 cm, 30 cm and 40 cm bed depth with breakthrough time of Zn and Fe, it shows that Cu have the longest breakthrough time with 52 minutes, 70 minutes and 106 minutes. From all three Figure (4.1 to 4.3), we can conclude that Cu is the easiest metal solution to be remove by MIRHA compare to Zn and Fe.

The volume of treated effluent up to the breakthrough time and the total mass of MIRHA used in the each column for each metal solution were shown in Table 6. Based on the results obtained, it was also observed that the breakthrough time was increased with the increment of column depth. Apart from that, the results also shown that Cu has the best breakthrough curve compare to Zn and Fe with 52 minutes for 20 cm column, 70 minutes for 30 cm column and 106 minutes for 40 cm column. It was also noted that the breakthrough time for various metal followed order Cu>Fe>Zn. This is due to the ionic radius and electro positive charges of the ions (Eisazadeh, 2012).

**Table 4.1:** Volume of effluent treated and the mass of MIRHA used

| Metal | Bed depth (cm) | Breakthrough time (min) | Treated volume (mL) | Total mass of MIRHA 800°C |
|---|---|---|---|---|
| Zn | 20 | 18 | 90 | 3.33 g |
| | 30 | 35 | 175 | 5.00 g |
| | 40 | 54 | 270 | 7.00 g |
| Fe | 20 | 18 | 90 | 3.33 g |
| | 30 | 35 | 175 | 5.00 g |
| | 40 | 70 | 350 | 7.00 g |
| Cu | 20 | 52 | 260 | 3.33 g |
| | 30 | 70 | 350 | 5.00 g |
| | 40 | 106 | 530 | 7.00 g |

# CHAPTER 5

# CONCLUSIONS

In this column study, MIRHA was investigated for its ability to absorb heavy metal in single metal solutions. Based on the results obtained, it was observed that MIRHA can be used as adsorbent for heavy metal removal in single metal solutions of Zn, Fe and Cu. It was also shown that the increase in metal uptake capacity with the increase of bed height was due to the increase of adsorbent and contact time with MIRHA in the column hence increasing in heavy metal adsorption.

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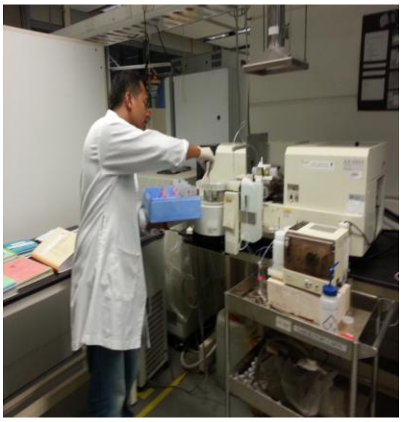
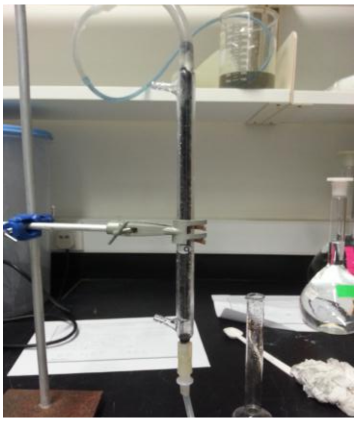
# APPENDIX 1

## PROGRESS PHOTO

Appendix 1.1: MIRHA sample


Appendix 1.2: Experimental set up


Appendix 1.3: Analyzing effluent


Appendix 1.4: Column experiment

# APPENDIX 2

### Gaant Chart for FYP II

| No. | Detail/ Week | 1 | 2 | 3 | 4 | 5 | 6 | 7 | MID SEM BREAK | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Project work | ■ | ■ | ■ | ■ | ■ | ■ | ■ | | | | | | | | | |
| 2 | Submission of progress report | | | | | | | | | | | ○ | | | | | |
| 3 | Poster Pre Sedex | | | | | | | | | | | ○ | | | | | |
| 4 | Project work continues | | | | | | | | | ■ | ■ | ■ | ■ | ■ | | | |
| 5 | Submission of Draft report | | | | | | | | | | | | | ○ | | | |
| 6 | Submission of dissertation(soft bound) | | | | | | | | | | | | | | ○ | | |
| 7 | Submission of Technical paper | | | | | | | | | | | | | | ○ | | |
| 8 | VIVA | | | | | | | | | | | | | | | ○ | |
| 9 | Submission of dissertation(hard bound) | | | | | | | | | | | | | | | | ○ |

Legends:

■ Project Activity

○ Key Milestone