

REDUCTION OF Fe(II), Zn(II) AND Cd(II) USING  
RICE HUSK

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REDUCTION OF Fe(II), Zn(II), AND Cd(II) USING RICE HUSK

NURUL NADIA BINTI SAMIN

A report submitted in partial fulfillment of the  
requirements for the award of the degree of  
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*Special Dedication to My Beloved Family*

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## ABSTRACT

Adsorption method is simple and relatively cost-effective, thus has been widely used. Several materials are derived from natural resources, plant wastes or industrial byproducts. Rice husk is a byproduct of the rice milling industry. It was chosen because of its granular structure, chemical stability and its local availability at very low cost and there is no need to regenerate them due to their low production costs. The purpose of this research is reduction of Fe(II), Zn(II) and Cd(II) using Rice Husk. Besides that, this research is also to investigate the potential of rice husk for reduction of Fe(II), Zn(II) and Cd(II) heavy metal ions by the effect variation of contact time, adsorbent dosage and initial concentration. The adsorbent was prepared by collecting the rice husk, dried and ground the rice husk using grinder and mechanical sieve to specify the adsorbent to certain size. 0.5g of rice husk as adsorbent with 50 ml adsorbate was kept constant for this experiment using 100 mg/L for initial concentration then there were placed on orbital shaker, operated at 150 rpm by varied time in 10 minutes to 120 minutes. For variation adsorbent dosage, the solution was set for different of dosage from 0.3 until 1.8 grams then placed on orbital shaker at constant 150 rpm and 25°C. Initial concentration from 10 until 125 mg/L was used with adsorbent samples in optimum weight and time then placed on orbital shaker at constant 150 rpm and 25°C. Adsorption process using rice husk was found to be suitable at initial concentration on 100 mg/L. Kinetic studies were conducted where both pseudo-first order and pseudo-second order yield value of  $R^2$  from 0.885 to 0.961 and from 0.955 to 0.999 for each order. Adsorption isotherms were described by both Langmuir and Freundlich isotherms. Langmuir equation was found to represent the equilibrium data for adsorption of Fe(II), Zn(II) and Cd(II) using rice husk ( $0.942 < R^2 < 0.981$ ), and the maximum adsorption capacity was highest for Cd(II) with 1.4793 mg/g.



## ABSTRAK

Kaedah penjerapan mudah dan agak berkesan dari segi kos, dengan itu telah digunakan secara meluas. Beberapa bahan yang diperolehi daripada sumber-sumber asli, sisa tumbuhan atau hasil sampingan industri. Sekam padi adalah hasil sampingan industri pengilangan beras. Ia dipilih kerana struktur berbutir, kestabilan kimia dan ketersediaan tempatan pada kos yang sangat rendah dan tidak perlu untuk menjana semula mereka kerana kos pengeluaran yang rendah. Tujuan kajian ini adalah pengurangan Fe(II), Zn(II) dan Cd(II) menggunakan sekam padi. Selain itu, kajian ini juga untuk menyiasat potensi sekam padi untuk mengurangkan Fe(II), Zn(II) dan Cd(II) ion-ion logam berat oleh kesan perubahan masa, dos adsorben dan kepekatan awal. Adsorben telah disediakan dengan mengambil sekam padi, dikeringkan dan dikisar kepada saiz tertentu. 0.5g sekam padi sebagai adsorben dengan adsorbate 50 ml adalah malar bagi eksperimen ini menggunakan 100 mg / L untuk kepekatan awal maka diletakkan di atas penggongcang orbit, yang beroperasi pada 150 rpm oleh pelbagai masa dari 10 minit hingga 120 minit. Untuk dos perubahan adsorben, larutan telah ditetapkan untuk berbeza dos daripada 0.3 sehingga 1.8 gram kemudian diletakkan di atas penggongcang orbit pada 150 rpm yang berterusan dan 25°C. Kepekatan awal dari 10 hingga 125 mg / L telah digunakan dalam sampel adsorben dengan berat dan masa yang terhad kemudian diletakkan pada penggongcang pada 150 rpm dan 25°C. Proses penjerapan menggunakan sekam padi didapati sesuai pada kepekatan awal pada 100 mg / L. Kajian kinetik telah dijalankan di mana kedua-dua pseudo-tertib pertama dan pseudo-tertib kedua di mana hasil nilai  $R^2$  dalam lingkungan (0.885-0.961) dan (0.955-0.999) bagi setiap tertib. Penjerapan bagi Fe(II), Zn(II) dan Cd(II) akan diterangkan oleh kedua-dua *Langmuir* dan *Freundlich* model.

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**LIST OF ABBREVIATIONS**

AAS	Atomic Adsorption Spectrometer
Cu(II)	Copper(II)
Zn(II)	Zinc(II)
Fe(II)	Iron(II), Ferum(II), Ferrous

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## CHAPTER 1

### INTRODUCTION

#### 1.1 BACKGROUND

Nowadays, the contaminations of water resources not only cause from pollution but also from failing to treat the water successfully. Due to the discharge of large amounts of metal-contaminated wastewater, industries bearing heavy metals, such as Cd, Cr, Cu, Ni, As, Pb, Fe and Zn, are the most hazardous among the chemical-intensive industries. Because of their high solubility in the aquatic environments, heavy metals can be absorbed by living organisms. Once they enter the food chain, large concentrations of heavy metals may accumulate in the human body. If the metals are ingested beyond the permitted concentration, they can cause serious health disorders (Babel and Kurniawan, 2004). For example, Cadmium causes serious renal damage, anemia, hypertension and itai-itai. Usually, this resource comes from industry liquid waste. This condition can bring bad effect because the untreated liquid waste may contain heavy metals ions that are not good to environment but also to human. Heavy metal contamination exists in aqueous wastes of many industries, such as metal plating, mining operations, refining ores, paint and pigments, tanneries, chloralkali, sludge disposal, radiator manufacturing, smelting, alloy industries and storage batteries industries, etc. (Kadirvelu et al., 2001). The presence of heavy metal ions is a major concern due to their toxicity to many life forms.



Conventional methods for removing heavy metals from aqueous solutions include chemical precipitation, ion exchange, adsorption (Gode and Pehlivan, 2006) and membrane filtration technologies. Among them, adsorption method is simple and relatively cost-effective, thus has been widely used. Several materials are derived from natural resources, plant wastes or industrial byproducts.

Biosorption is a promising technique for the removal of heavy metals from aqueous environments especially when adsorbents are derived from lignocellulosic materials (Coelho et al., 2007). The search for new technologies to remove toxic metals from wastewaters has directed attention to biosorption, which is based on metal binding to various biological materials. Biosorption is a fast and reversible reaction of the heavy metals with biomass. Laszlo and Dintzis (1994) have shown that lignocellulosics have ion-exchange capacity and general sorptive characteristics, which are derived from their constituent polymers and structure. The polymers include extractives, cellulose, hemicelluloses, pectin, lignin and protein. Agricultural by-products vary greatly in their ability to remove metals from solution. The ability of biological materials to adsorb metal ions has received considerable attention for the development of an efficient, clean and cheap technology for wastewater treatment at metal concentrations as low as 1 mg/L (Chong and Volesky, 1995).

In recent years, special attention has been focused on the use of natural sorbents as an alternative to replace the conventional adsorbents, based on both the environmental and the economical points of view (Babel and Kurniawan, 2003) and (Bailey et al., 1999). Natural materials that are available in large quantities, or certain waste products from industrial or agricultural operations, may have potential as inexpensive sorbents. Due to their low cost, when these materials the end of their lifetime, they can be disposed of without expensive regeneration. The abundance and availability of agricultural by-products make them good sources of raw materials for natural sorbents.

The removal of heavy metal ions using low-cost abundantly available adsorbents: agricultural wastes such as tea waste and coffee (Orhan and Buyukgungor, 1993), hazelnut straw

(Cimino et al., 2000), peanut hull (Johnson et al., 2002), sawdusts, pinus bark (Vazquez et al., 1994) and different bark samples (Seki et al., 1997), coconut husk (Babarinde, 2002), Ponkan mandarin peels (Pavan et al., 2006), modified cellulosic materials (Acemioğlu and Alma, 2001), corn cobs (Hawthorne-Costa et al., 1995), apple wastes (Maranon and Sastre, 1991), wool fibers (Balkose and Baltacıoğlu, 1992), tea leaves (Tee and Khan, 1988), banana and orange peels (Annadurai et al., 2002), sugarcane bagasse (Khan et al., 2001), papaya wood (Saeed et al., 2005), maize leaf (Babarinde et al., 2006), leaf powder (Hanafiah et al., 2007), grape stalk wastes (Villaescusa et al., 2004) and different agricultural by-products were used and investigated. (Marshall and Champagne, 1995 and Pehlivan et al., 2006)

Rice husk is a byproduct of the rice milling industry. It is one of the most important agricultural residues in quantity. It represents about 20% of the whole rice produced, on weight basis of the whole rice (Daifullah *et al.* 2003). The estimated annual rice production of 500 million tones in developing countries, approximately 100 million tones of rice husk is available annually for utilization in these countries alone. Traditionally, rice husks have been used in manufacturing block employed in civil construction as panels and was used by the rice industry itself as a source of energy for boilers (Della *et al.* 2001). However, the amounts of rice husk available are so far in excess of any local uses and have posed disposal problems. It was chosen because of its granular structure, chemical stability and its local availability at very low cost and there is no need to regenerate them due to their low production costs.

The main constituents of rice husk are: 64-74% volatile matter and 12-16% fixed carbon and 15-20% ash (Armesto *et al.* 2002; Daifullah *et al.* 2003). The rice husk composition are: 32.24% cellulose, 21.34% hemicellulose, 21.44% lignin, 1.82% extractives, 8.11% water and 15.05% mineral ash (Govindarao 1980; Rhman *et al.* 1997; Nakbanpote *et al.* 2000).

## **1.2 PROBLEM STATEMENT**

Heavy metal pollution has become one of the most important environmental problems. The heavy metals such as ferum, cadmium, zinc, and copper are widely used in industries such as metal cleaning and plating, mining operations, printed circuit board, refining ores, sludge disposal, fertilizer, paints and pigments. The effluent from these industries spreads into the environment through soil and water streams and accumulates along the food chain, resulting in a high risk to human health. Removing this heavy metal need advance technologies that consume a lot of money. Hence, another alternative of low cost adsorption system is tested using rice husk, unused residues that could hopefully help to reduce pollution.

Rice husk is an agricultural residue produces from rice milling industry being thrown away or burned and some used for power production from combustion. However, this inexpensive waste can also be a solution to reduce water pollution. This alternative could save not only money but increases the usage of rice husk. Thus, this research will determine whether rice husk could be an effective adsorbent for the removal of heavy metal ions contained in wastewater discharged by the industries.

## **1.3 RESEARCH OBJECTIVE**

To investigate the potential of rice husk as natural adsorbent to adsorb Fe(II), Zn(II) and Cd(II) ions by the variation of contact time, adsorbent dosage and initial concentration.

## 1.4 SCOPE OF RESEARCH

- 1.4.1 Analyzation of the potential of rice husk as a low cost natural adsorbent to adsorb heavy metal ions from solution.
- 1.4.2 Investigation and observation of the process condition effect for Fe(II), Zn(II) and Cd(II) that can be removed by using rice husk.
- 1.4.3 Determination of the effect of process parameters to the percentage removal efficiency of heavy metal ions by analyzing the result of initial and final concentration for each variable using Atomic Adsorption Spectrophotometer :
  - 1.4.3.1 Contact Time
  - 1.4.3.2 Adsorbent Dosage
  - 1.4.3.3 Initial Concentration
- 1.4.4 Determination the kinetics study of different of heavy metal using rice husk as Adsorbent by various kinetic models.

## 1.5 RATIONALE AND SIGNIFICANCES

In this experiment, heavy metal ions, Fe(II), Zn(II) and Cd(II) acts as the atoms and molecules attached, called adsorbate. This solid or liquid surface, or adsorbent such as activated carbon is one of the material used in adsorption process, but it does not remove metal completely. Therefore, researches had studied to find other natural resources that could be an alternative to activated carbon. The cost of this research is lower because the material is easily to find and equipment also the common one is used. Furthermore, the aqueous solution that is ferum, zinc and cadmium solution are easily to prepare. Hence, this research would give benefit to chemical industries to manage their waste disposal.

“The processing and transformation of agricultural residues into heavy metal with good adsorption properties would alleviate problems of disposal and management of these waste by-products, while providing a high quality end product for water and wastewater treatment that could potentially expand the carbon market (Kalderis and Bethanis, 2008).” Rice husk is chosen in this research because it is selective towards metal ion adsorption, effective, economically feasible because it can be easily found as waste at rice factories, and also benefit for the environment.

## CHAPTER 2

### LITERATURE REVIEW

Kaneco et al. (2000) had studied adsorption method such as ion exchange and membrane separation is simple one for the removal of heavy metals. However, there is a limit in the generality in developing countries because chelating and ion-exchange resins are expensive. Therefore, it is worthwhile to develop the economical adsorbents of heavy metals which can be generally utilized in developing countries. Previously, we reported the removal method of heavy metals (Ag, Au, Co and Ni) with economical materials. In the present study, removal of cadmium(II), chromium(VI), copper(II) and lead(II) in wastewater was further carried out by adsorption with silicate rocks and mineral oxides. These materials can be obtained commercially because those are easily synthesized and fairly uniform in their chemical and physical properties. Therefore, the adsorbents are costly-adaptable treatment medium in developing countries. Since the heavy metals adsorbed onto the adsorbents can be readily stripped with acid solutions, the adsorbents can be reused several times. Finally, when the materials are released into the environment, there is little potential of secondary pollution owing to inherently nature minerals. Among these materials, montmorillonite and silica gel showed very high adsorption capacities and were successfully applied in the removal of heavy metals in the rinsing wastewater from plating factory. Since this proposed water treatment system is expected to be useful, rapid,

simple and cheap for the adsorption of heavy metals in the wastewater, it can be practically suitable for developing countries.

Cay et al. (2004) had studied the heavy metal contamination exist in aqueous waste streams from diverse industries such as metal plating, manufacturing, batteries, as well as agricultural sources where fertilizers and fungicidal sprays are intensively used. Cu, Zn, Hg, and Cd are harmful wastes produced by industry that pose a risk of contamination groundwater and other water resources. Heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders. For example, cadmium causes serious renal damage, anemia, hypertension and itai-itai.

Bhattacharya et al. (2006) had studied the reduction of the pollutant to an acceptable level is necessary when toxic metals are present in aquatic system. Adsorption and ion exchange processes are the most useful methods to remove them. These methods explore the availability of different kinds of adsorbents associated with convenient procedures for obtaining high efficiency. A large number of different adsorbent materials containing a variety of attached chemical functional groups have been reported for this purpose. For instance, activated carbon is the most popular material; however, its high cost restricts its large-scale use.

Yang et al. (2002) has done research the activated carbons can be commonly produced from coal, wood or agricultural wastes such as coconut and palm shell, corncob, rich husk, etc., activated by physical or chemical process. Because of their special pore structure, they have super adsorption capacity and are generally used in variety industrial and domestic fields, such water treatment, solvent decolourization, catalyst supports of fuel cell and surpercapacitors. In recent years, there is growing interest in the production of activated carbons from agricultural by-products and residual wastes.

Orhan et al. (1993) stated the agricultural products and by-products has been widely investigated as a replacement for current costly methods of removing heavy metals from water and wastewater. Some of the agricultural materials can be effectively used as a low-cost sorbent. Modification of agricultural by-product could enhance their natural capacity and add value to the by-product. In this review, an extensive list of sorbent literature has been compiled to provide a summary of available information on a wide range of low-cost agricultural product and by-product sorbent and their modification for removing heavy metals from water and wastewater.

Friedman et al. (1972) stated the idea of using various agricultural products and byproducts for the removal of heavy metal from solution has been investigated by number of authors and the efficiency of number of different organic waste materials as sorbents for heavy metals. The obvious advantages of this method compared to other are lower cost involved when organic waste materials are used. Activated carbon adsorption appears to be a particularly competitive and effective process for the removal of heavy metals at trace quantities. However, the use of activated carbon is not suitable for developing countries because of its high cost. For that reason, the uses of low cost materials as possible media for metal removal from wastewater have been highlighted.

Ahmad et al. (2009) investigated the removal of Cu (II) and Pb (II) ions from aqueous solutions by adsorption on agricultural waste. Batch kinetics and isotherm studies were carried out under varying the solution of pH, contact time and adsorbent dosage. Adsorption isotherms of Cu (II) and Pb (II) ions on adsorbents were determined and correlated with common isotherm equations such as Langmuir and Freundlich models. The thermodynamic parameters like free energy, enthalpy, and entropy changes for the adsorption of Cu (II) and Pb (II) ions have also been computed and discussed. The heat of adsorption [ $\Delta H = 31.47$  kJ/mol for Cu (II) and  $\Delta H = 20.07$  kJ/mol for Pb (II)] implied that the adsorption was endothermic in nature.



Mohammad R. H. et.al. (2011) explained in their study by the experiment of the removal of Cr (VI) from aqueous solution was performed using pine needles powder using batch adsorption technique. Parameters studied including adsorbent dose, particle size, agitation speed, pH of solution, contact time and initial Cr (VI) concentration, where the adsorption process was found to be highly pH dependent and the optimum pH range for adsorption of Cr (VI) was found to be between 2 and 3. Adsorption isotherms were modelled with the Langmuir, Freundlich, Dubinin–Radushkevich and Tempkin isotherms, resulting with Langmuir equation that is found to be the best representing the equilibrium data for Cr (VI) - pine needles powder system than other isotherms with  $R^2 = 0.9946$  and the maximum monolayer adsorption capacity was found to be  $40.0 \text{ mg g}^{-1}$  at  $298\text{K}$ .

Rahman et al. (2005) stated the rice husk is an agricultural waste, obtained from the rice mills after the separation of rice from paddy. It is mostly used as a fuel in the boiler furnaces of industries like sugar, paper, etc. to produce steam. The rice husk ash (RHA) is collected from the particulate collection equipment attached upstream to the stack of the rice husk-fired boilers. Rice husk ash is available in plenty and almost free of cost. Rice husk ash has good adsorptive properties and has been used for the removal of various dyes, adsorption characteristics of malachite green on activated carbon derived from rice husks produced by chemical–thermal process, heavy metals, and other compounds like chlorinated hydrocarbons. Average particle size of RHA was  $150.47 \mu\text{m}$ . Bulk density and heating value of RHA were found to be  $104.9 \text{ kg/m}^3$  and  $9.68 \text{ MJ/kg}$ , respectively. Proximate analysis showed the presence of 0.73% moisture, 5.37% volatile matter, 88.0% ash and 5.90% fixed carbon in RHA. High amount of ash indicates that RHA is basically inorganic in nature. Elemental analyses showed 7.424% carbon, 0.061% hydrogen, 0.846% nitrogen, and rest others. The heating of rice husk at different temperatures produces RHA containing different contents of carbon and silicon dioxide. Nakbanpote et al. (2000) reported that the RHA heated at higher temperatures had reduced percentages of carbon but an increased proportion of silicon dioxide. Almost all of the carbon was lost when heated at temperatures  $\geq 400 \text{ }^\circ\text{C}$ . In the paper mills, rice husk is heated at temperatures  $\geq 700 \text{ }^\circ\text{C}$  to generate steam. Therefore, it is expected that the amount of carbon will be very small in rice husk ash.

## CHAPTER 3

### RESEARCH METHODOLOGY

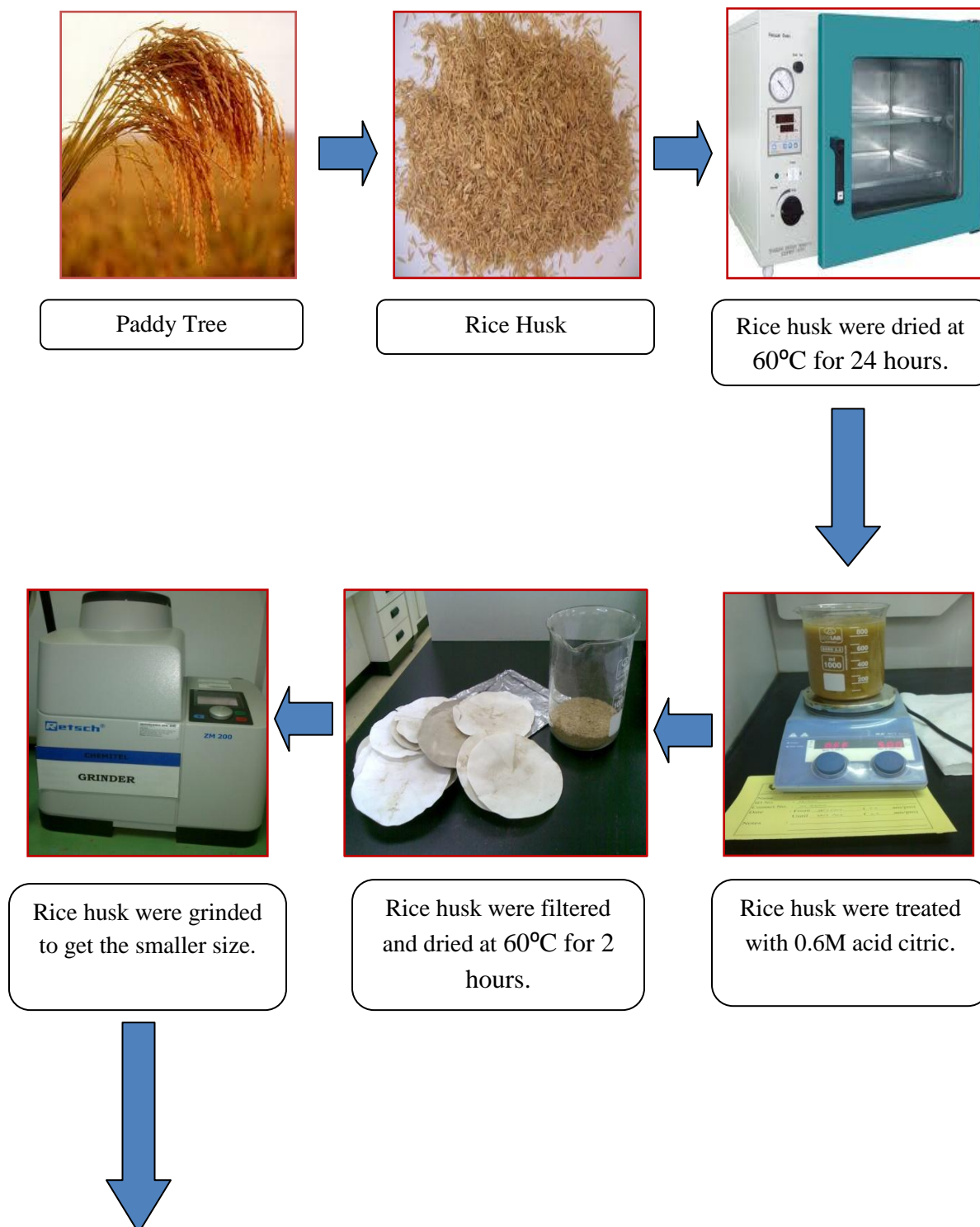
#### 3.1 MATERIAL

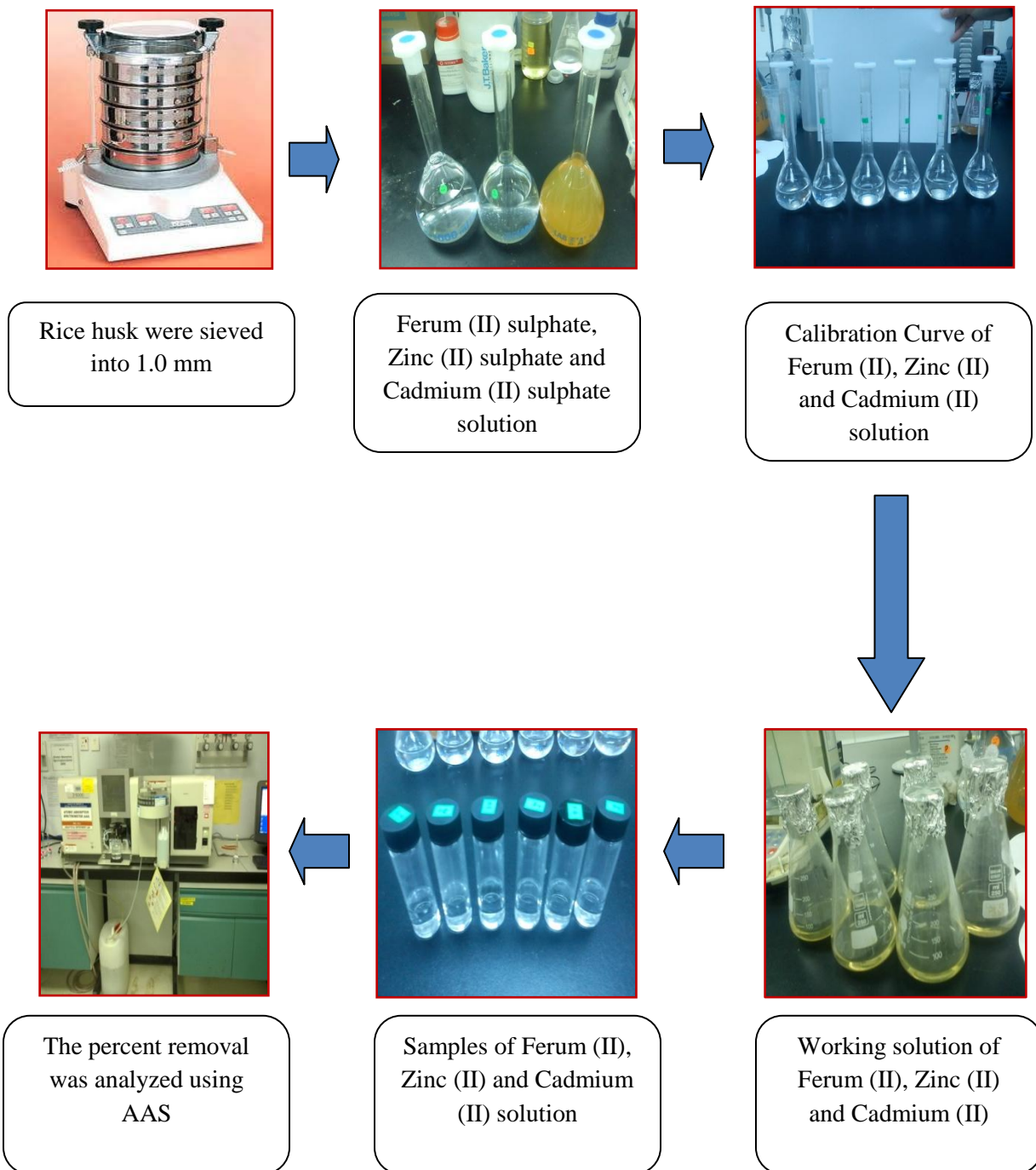
- i. Rice Husk
- ii. Ferrous (II) Sulphate  $\text{FeSO}_4$
- iii. Zinc (II) Sulphate  $\text{ZnSO}_4$
- iv. Cadmium (II) Sulphate  $\text{CdSO}_4$
- v. 0.1 M NaOH
- vi. 0.1 M HCl
- vii. Distilled Water

### 3.2 APPARATUS

- i. Volumetric flask 5ml, 10ml, 25ml, 50ml, 100ml and 1000ml
- ii. Beaker 50ml and 100ml
- iii. Conical Flask 100ml and 250ml
- iv. Sieve shaker
- v. Magnetic Stirrer
- vi. Glass rod
- vii. Hot plate
- viii. Whatman filter paper
- ix. Micropipette 10 to 1000 $\mu$ L
- x. Atomic Adsorption Spectrophotometer (AAS)
- xi. Orbital shaker
- xii. Oven
- xiii. Grinder
- xiv. pH Meter
- xv. Syringe filter

### 3.3 FLOWCHART





### 3.4 EXPERIMENTAL PROCEDURE

#### 3.4.1 Preparation of Adsorbent

The rice husk were bought from BERNAS rice mill Simpang Empat, 2700 Perlis. The rice husk was washed with distilled water to remove any impurities and dried at 60°C for 24 hours. 45 grams of rice husk were treated with 900 ml of 0.6 M citric acid and stirred with magnetic stirrer for 2 hours. The pretreatments of rice husk can remove lignin, hemicelluloses, reduce cellulose crystallinity and increased the porosity of surface area. Many researchers found that the modified rice husk exhibited higher adsorption capacities than unmodified rice husk ( Wan Ngah and Hanafiah, 2008). The mixture solution was filtered and dried at 60°C for 1 hours. The dry rice husk treatment then crushed by using grinder and sieve to the size of 1.0 mm.

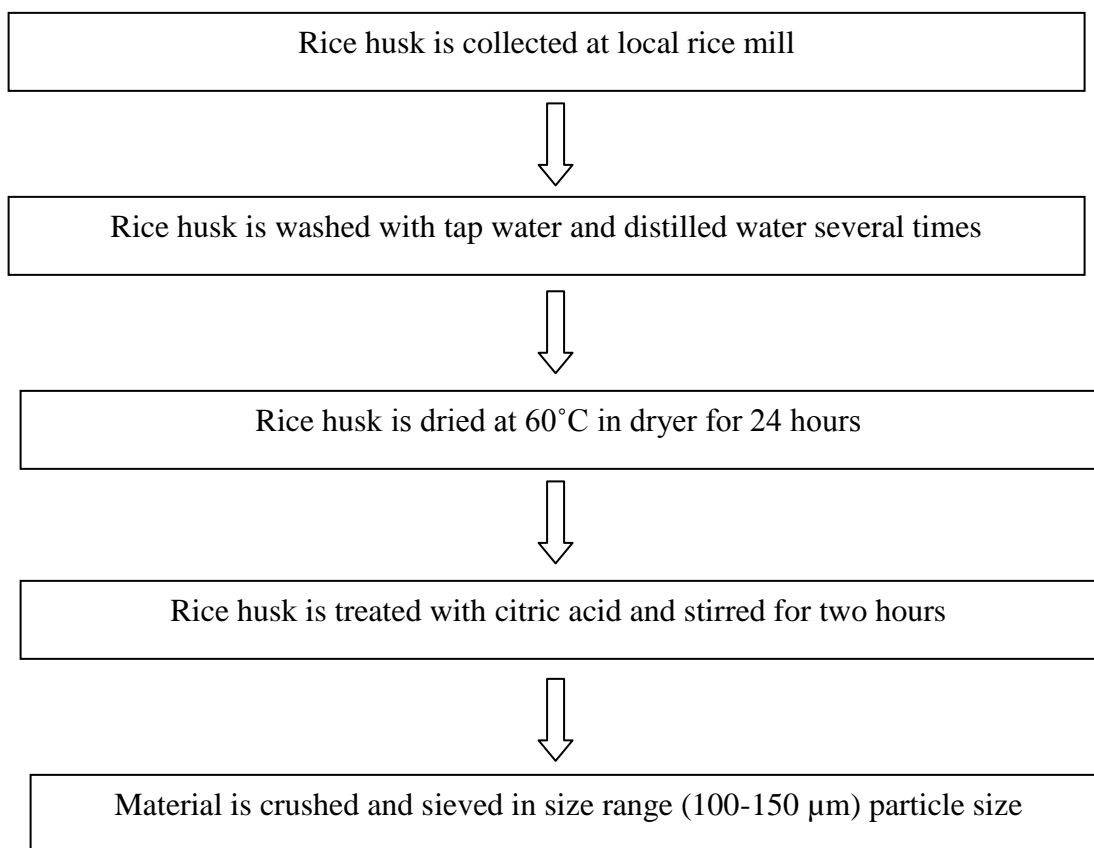


Figure 3.4.1 Flow Diagram for Preparation of Adsorbent

### **3.4.2 Preparation of Adsorbate**

Adsorbate solution having concentrations of 1000 mg/l of Fe (II), Zinc (II) and Cd (II) were prepared by dissolving exact amount of  $\text{FeSO}_4 \cdot x\text{H}_2\text{O}$ ,  $\text{ZnSO}_4 \cdot \text{H}_2\text{O}$  and  $\text{CdSO}_4 \cdot x\text{H}_2\text{O}$  in distilled water respectively. Adsorbate solution of heavy metal is placed in different volumetric flask for usage of working solution.

### **3.4.3 Preparation of Calibration Curve**

Standard curve was prepared by dilution of Ferum (II), Zinc (II) and Cadmium (II) solution into 0 until 10 mg/L. Standard or calibration curve is a method used to determine the unknown concentration of Ferum (II), Zinc (II) and Cadmium (II) solution by comparing the unknown concentration with a standard sample of known concentration. The method can convert the value of absorption (ABS) from Atomic Adsorption Spectrophotometer (AAS) reading into the value of concentration. Hence final concentration of ferum (II), Zinc (II) and Cadmium (II) solution can be known.

## **3.5 PREPARATION OF SAMPLES**

### **3.5.1 Effect of Contact Time**

5ml of Fe (II) from adsorbate solution was poured into 100 ml conical flask and added distilled water until 50 ml. The pH of Fe (II) solution is kept constant by adjusting using 0.1 M of NaOH and 0.1 M HCl until all six solutions analyzed using pH meter reaches pH 6. The adsorbent dosage is kept constant at 0.5 grams, where the rice husk is weighed using weighing board and placed into the conical flask containing Fe (II) solution. The solution and rice husk was shaken using orbital shaker at constant speed of 150 rpm at 25°C for different variation of

time ranging from 10 minutes to 120 minutes. For variation time was reached, the solution is collected from the shaker and filtered using syringe filter 0.45  $\mu\text{m}$  where the final concentration is analyzed using Atomic Adsorption Spectrophotometer (AAS). Above steps is repeated for Zn (II) and Cd (II) heavy metal solution.

### **3.5.2 Effect of Adsorbent Dosage**

5ml of Fe (II) from adsorbate solution was poured into 100 ml conical flask and added distilled water until 50 ml. The pH of Fe (II) solution is kept constant by adjusting using 0.1 M of NaOH and 0.1 M HCl until all six solutions analyzed using pH meter reaches pH 6. Rice husk adsorbent is weighed using weighing board for different mass of dosage ranging from 0.3 to 1.8 grams and placed into the conical flask. The conical flasks are placed onto the orbital shaker and set to operate at constant 150 rpm, 25°C for optimum time of Fe (II) removal. Samples are collected and filtered using syringe filter 0.45 $\mu\text{m}$  then analyzed the final concentration using Atomic Adsorption Spectrophotometer (AAS). Above steps is repeated for Zn (II) and Cd (II) heavy metal solution.

### **3.5.3 Effect of Initial Concentration**

5ml of Fe (II) from adsorbate solution was poured into 100 ml conical flask and added distilled water until 50 ml. The pH of Fe (II) solution is kept constant by adjusting using 0.1 M of NaOH and 0.1 M HCl until all six solutions analyzed using pH meter reaches pH 6. The variation of initial concentration ranging from 10 mg/l to 125 mg/l. The adsorbent dosage is kept constant at optimum dosage, where the rice husk is weighed using weighing board and placed into the conical flask containing Fe (II) solution. The conical flasks are placed onto the orbital shaker and set to operate at constant 150 rpm, 25°C for optimum time of Fe (II) removal.



Samples are collected and filtered using syringe filter  $0.45\mu\text{m}$  then analyzed the final concentration using Atomic Adsorption Spectrophotometer (AAS). Above steps is repeated for Zn (II) and Cd (II) heavy metal solution.

Experiment on the three variables, effect of pH solution, contact time, adsorbent dosage and initial concentration is done for adsorbate, Copper(II) Sulfate, Zinc(II) Sulfate and Ferrous(II) Sulphate solution.

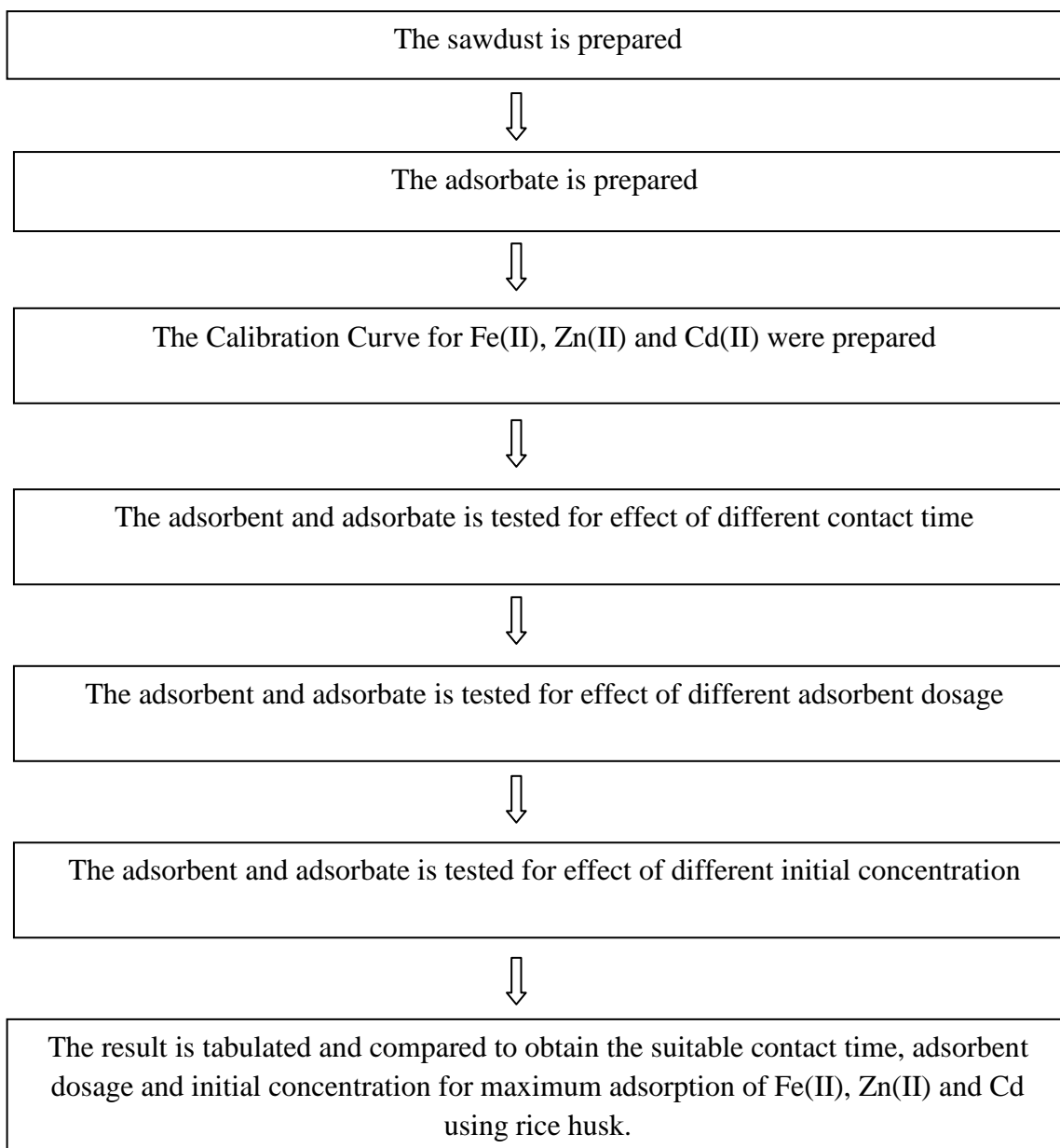


Figure 3.5 Flow Diagram of Adsorption of Fe(II), Zn(II) and Cd(II) Using Rice Husk Process

### 3.6 ANALYZING OF SAMPLES

Standard solutions are prepared before running the Atomic Adsorption Spectrometer. Solutions are prepared by diluting 1000mg/L of standard into a 50 ml volumetric flask for concentration 50mg/l with standard solution ranging from 0 to 10 mg/l by using the equation.

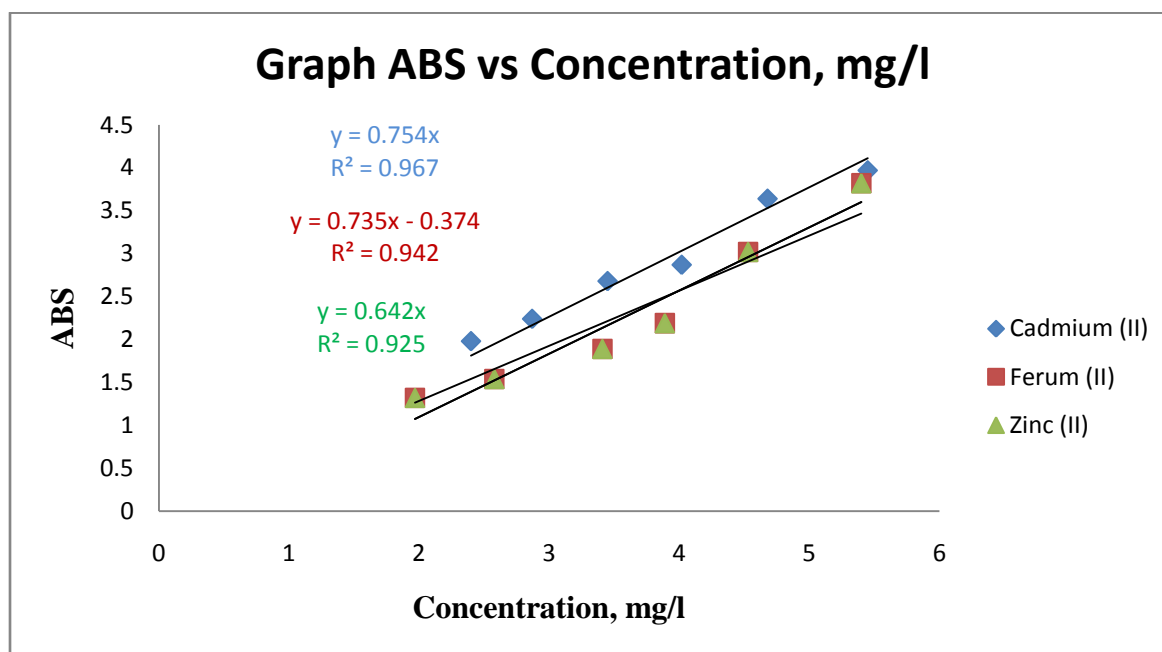
$$M_1 V_1 = M_2 V_2$$

Samples that are filtered will be diluted to 25mg/L in 100ml volumetric flask, having the dilution factor of 10 using the same equation. The standards are then titrated with 1 drop nitric acid and then the samples are analysed based on the AAS procedures, where final concentration of heavy metal solution is obtained.

## CHAPTER 4

## RESULT AND DISCUSSIONS

## 4.1 Calibration Curve



Graph 4.1 : Standard Curve for Fe(II), Zn(II) and Cd(II) solution

## 4.2 Effect of Contact Time

Adsorption of heavy metals Fe(II), Zn(II), and Cd(II) was measured at different contact time starting from minute 10 to minute 120 at constant initial concentration of heavy metal solution of 100 mg/L, amount of 0.5g of rice husk, solution pH and placed on orbital shaker at 150 rpm and 25°C using mechanical shaker. The graph of heavy metal removal efficiency, % versus contact time, minute is plotted by using formula below:

$$\% \text{ Removal Efficiency} = \frac{(C_0 - C_e)}{C_0} \times 100$$

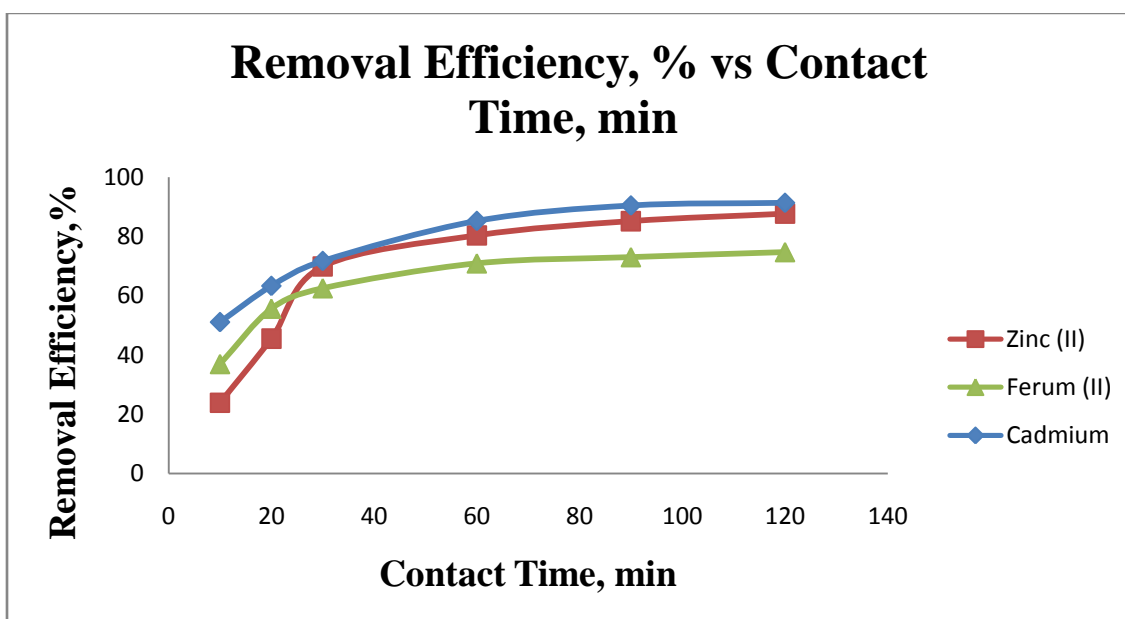


Figure 4.2 : Graph of Heavy Metal Efficiency, % vs Contact Time, min

Figure 4.2 shows that the removal efficiency for Cd(II) is the highest with the maximum removal efficiency of 91.30%, followed by Zn(II) with 87.60% and Fe(II) with 74.70%. The removal efficiency increases with the increasing of contact time. This is due to increasing of adsorption time helps more heavy metal ions adsorbed to the rice husk hence increasing the removal efficiency of heavy metal. Most of the maximum percent heavy metal removal was attained after about 70 min of contact time with the rice husk.

### 4.3 Effect of Adsorbent Dosage

The effect of the adsorbent dosage was studied at room temperature (25°C) by varying the adsorbent amount, ranging from 0.3g to 1.8g. For all these runs, initial concentration of heavy metal, solution pH and contact time was fixed as 100 mg/L, pH 6 and 70 minutes of contact time.

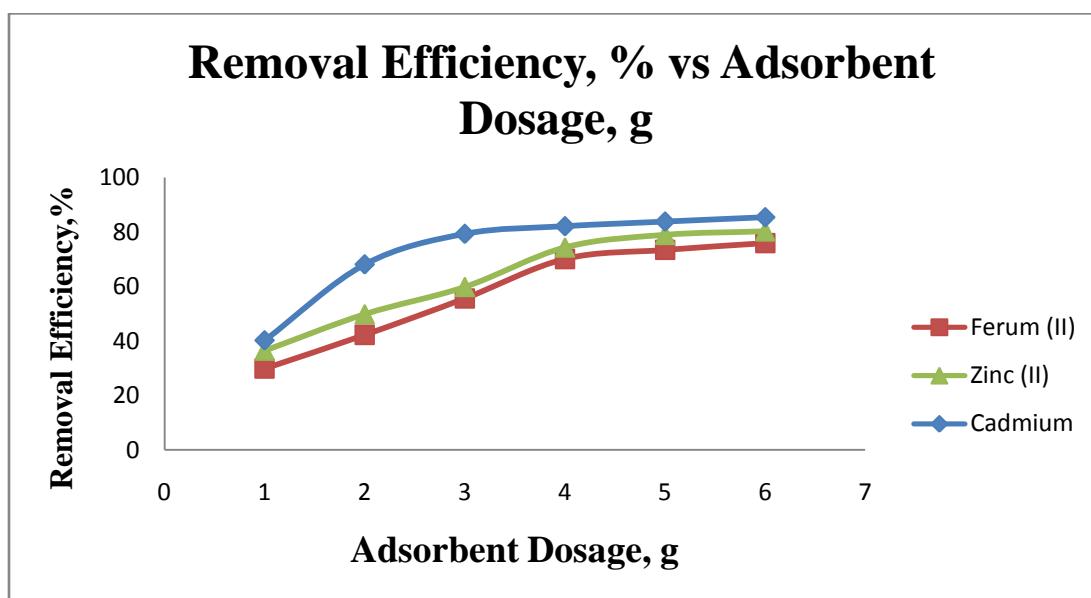


Figure 4.3: Graph of Removal Efficiency, % vs Adsorbent Dosage, g

Based on the graph above, removal efficiency for Cd(II) increase rapidly after 0.3g of adsorbent added, whereas slow increment of removal efficiency is attained heavy metal Zn(II) and Fe(II). The removal efficiency is directly proportional with the adsorbent dosage. This is

because the increasing of adsorbent dosage provides more adsorption site which increasing the total area for adsorption activity, hence resulting in increasing of heavy metal ions uptake.

#### 4.4 Effect of Initial Concentration

The effect of heavy metal concentration in the range of 10 to 125 mg/L on removal efficiency was observed with constant solution pH 6, contact time of 70 minutes and 0.5 g of adsorbent.

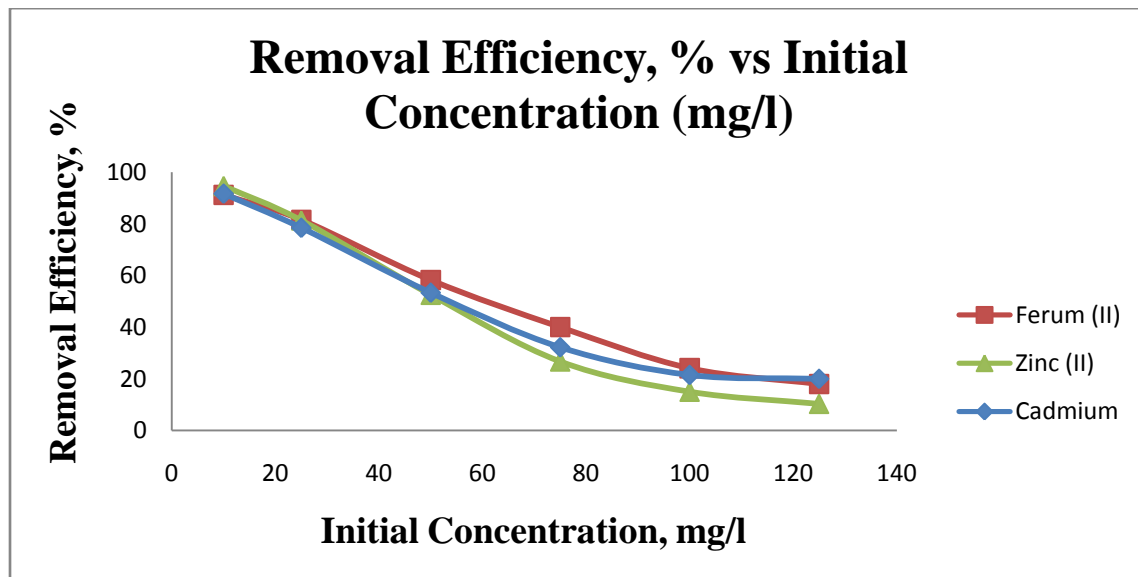


Figure 4.4 : Graph of Removal Efficiency, % vs Initial Concentration, mg/L

From the figure above, the removal efficiency decreases with the increasing amount of initial concentration. As the amount of adsorbate increases, the uptake of heavy metal ions decreases due to limiting of adsorption sites. The lowest removal efficiency was attained at 100 mg/L of adsorbate, with the removal efficiency of 20.00%, 18.00% and 10.00% for Cd(II), Fe(II) and Zn(II) heavy metal ions.

#### 4.5 Adsorption Kinetic Study

Kinetic models have been used to investigate the mechanism of adsorption process and determine the value of rate constant for pseudo- first and pseudo-second order kinetics, which is helpful for selecting optimum operating conditions for the full-scale batch process, using batch contact time study(Wahab, 2007). The Lagergen pseudo-first order rate expression is given by

$$\frac{dq}{dt} = k_1(q_e - q)$$

where,  $q_e$  is the amount of heavy metal ion adsorbed at equilibrium (mg/g),  $q$  is the amount adsorbed at time  $t$  (mg/g),  $k_1$  is the rate constant of first order adsorption (L/min). Integration with applying boundary conditions,  $t = 0$  to  $t$  and  $q = 0$  to  $q_e$  where the integrated form is

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

A linear graph of  $\log(q_e - q_t)$  plotted against  $t$  to retained the value of  $k_1$  from slope of the graph. All three Cu(II), Zn(II) and Fe(II) heavy metals ions are observed in the graph below

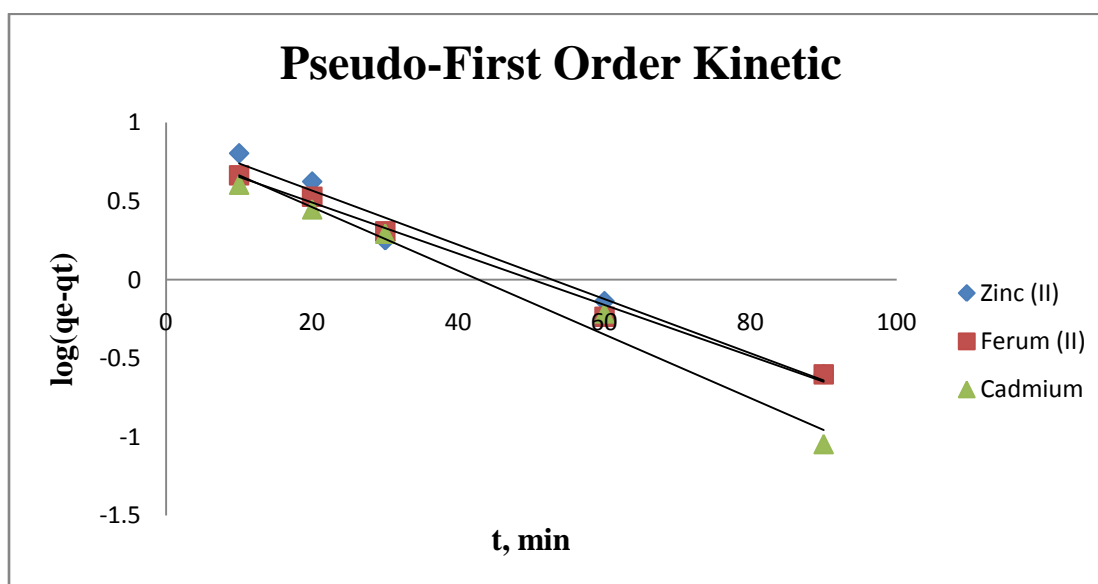


Figure 4.5.1 : Pseudo-First Order Kinetic for Fe(II), Zn(II) and Cd(II) Adsorption Using Rice Husk

Pseudo-second order model proposed by Ho and McKay is based on assumption that the adsorption follows second-order chemisorptions. Pseudo-second order kinetics can be expressed as

$$\frac{d_q}{d_t} = k_2(q_e - q)^2$$

Integrating equation above will give

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

where  $q_e$  and  $q_t$  are amount of heavy metal ions adsorbed at equilibrium and at time  $t$ , mg/g. A linear plot of  $\frac{t}{q_t}$  versus  $t$  will give the value of  $q_e$  and  $k_2$ , rate constant for second order adsorption.

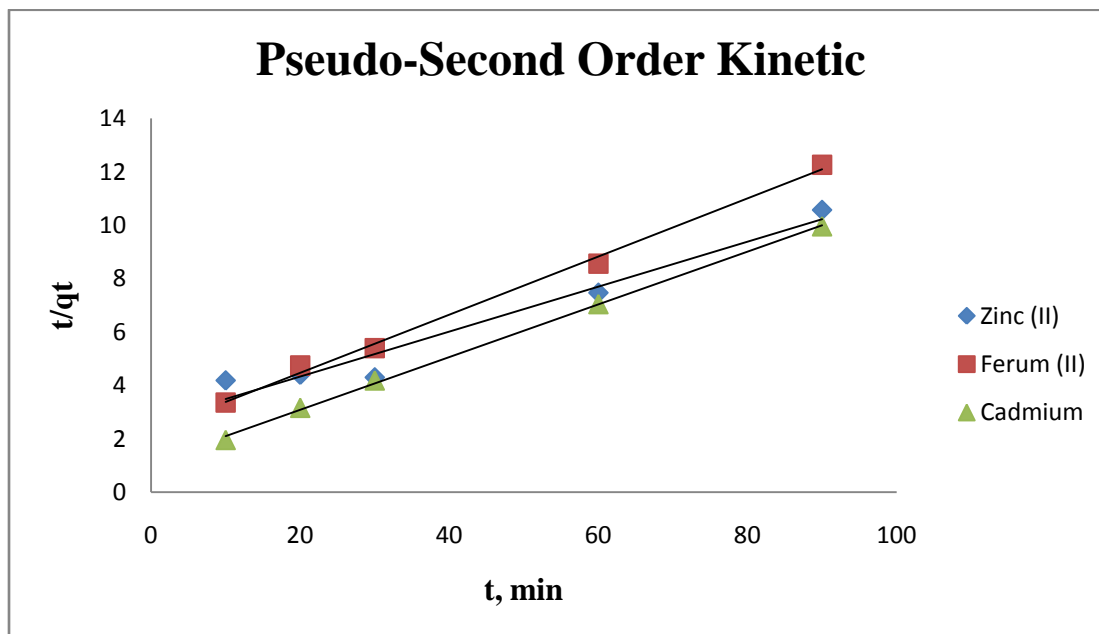


Figure 4.5.2 : Pseudo-Second Order Kinetic for Fe(II), Zn(II) and Cd(II) Adsorption Using Rice Husk.



Values from the graphs of pseudo-first and pseudo-second order kinetics are tabulated below.  
(M.M. El Jamal, 2011)

Table 4.5 Pseudo-First Order and Pseudo-Second Order Kinetic Model Constants

Heavy metal	Pseudo-First Order Kinetic		Pseudo-Second Order Kinetic	
	K <sub>1</sub> ads, L/min	R <sup>2</sup>	K <sub>2</sub> ads, g/mg.min	R <sup>2</sup>
Cd(II)	-0.0461	0.885	0.1224	0.999
Zn(II)	-0.0391	0.894	0.1551	0.955
Fe(II)	-0.0368	0.961	0.1607	0.996

From the calculated values above, the adsorption process of Cd(II), Zn(II) and Fe(II) heavy metal ions are more favourable towards pseudo-second order kinetics due to higher value of R<sub>2</sub> and in the range of 0 to 1.

#### 4.6 Adsorption Equilibrium Study

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate species among liquid and adsorbent, based on a set of assumptions that are mainly related to the heterogeneity/homogeneity of adsorbents. In this study, isotherm used to calculate maximum heavy metal ions uptake are Langmuir and Freundlich isotherms. Experimental isotherm data were conducted at an equilibrium time of 145 minute, constant adsorbent dosage of 0.5g, and adsorbate solution with pH 4. The Langmuir adsorption isotherm is based on the assumption the maximum adsorption occurs when a saturated monolayer of solute molecules is present on the adsorbent surface, the energy of adsorption is constant and there is no migration of adsorbate molecules in the surface plane. It may be represented in the linear form as follows (P. Senthil Kumar\*, 2009)

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_L q_m}$$

where  $q_m$  is the maximum heavy metal ions uptake, mg/g,  $K_L$  the Langmuir adsorption constant in L/mg. The graph of  $\frac{C_e}{q_e}$  plotted against  $C_e$  yielding the value of ranging from 19.19mg/g to 25.71mg/g.

### Langmuir Isotherm Model

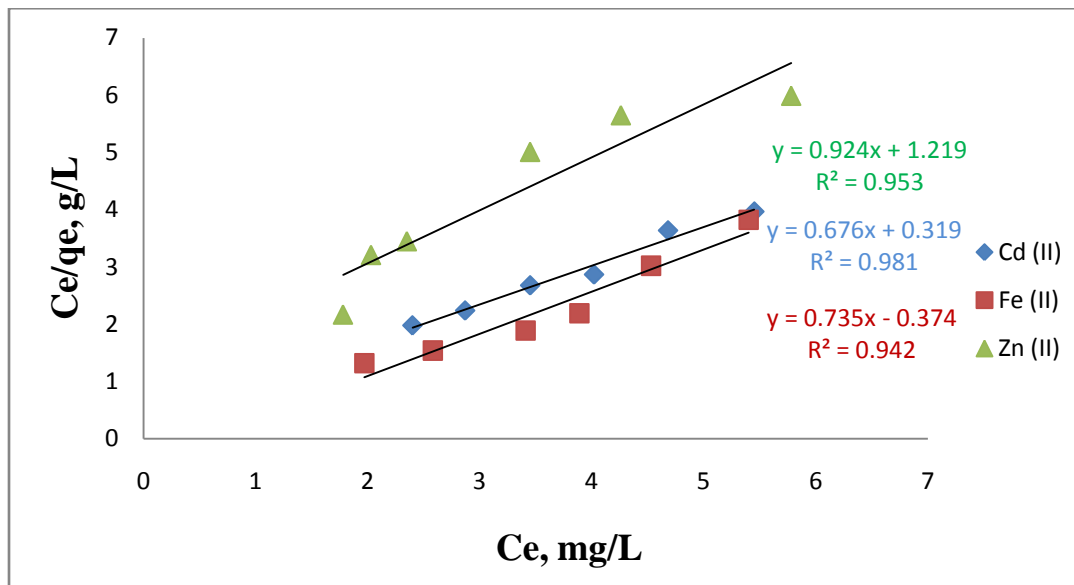


Figure 4.6.1 : Langmuir Isotherm for Cd(II), Zn(II) and Fe(II) Adsorption Using Rice Husk.

The Freundlich isotherm is expressed by

$$q_e = K_f C_e^{\frac{1}{n}}$$

The logarithmic form given as

$$\ln q_e = \log K_f + \frac{1}{n} \log C_e$$

where,  $K_f$  is the Freundlich constant, indicating the relative adsorption capacity of the adsorbent related to the bonding energy, and  $n$  is the heterogeneity factor representing the deviation from linearity of adsorption and is also known as Freundlich coefficient. The Freundlich constants,  $K_f$  and  $n$  are obtained by plotting the graph between of  $\ln q_e$  versus  $\ln C_e$ .

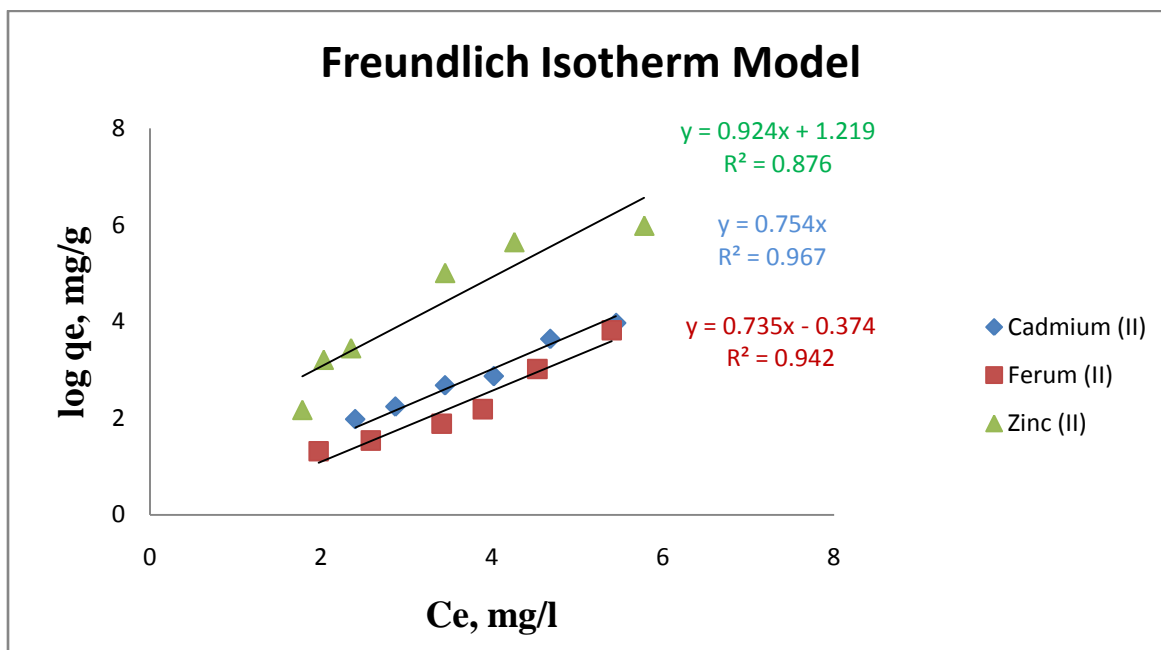


Figure 4.6.2 : Freundlich Isotherm for Cd(II), Zn(II) and Fe(II) Adsorption Using Rice Husk

Cd(II) yields higher value of  $R^2$  compared to Fe(II) and Zn(II) ions where it is adsorbed more onto the rice husk, with  $K_f$  values ranging from 2.4 to 3.0 and heterogeneity factor,  $n$  of 2.4 to 2.73. Figure below shows the summary of adsorption equilibrium study.

Table 4.6 Langmuir and Freundlich Adsorption Isotherm Model Constants

	Langmuir				Freundlich		
	$Q_m, \text{mg/g}$	$K_L, \text{l/mg}$	$R_L$	$R^2$	$K_f, (\text{mg/g})(\text{l/mg})^{(1/n)}$	$n$	$R^2$
<b>Cd(II)</b>	1.4793	2.1191	0.0037	0.981	16.557	1.3263	0.967
<b>Zn(II)</b>	1.0822	0.7580	0.0104	0.953	0	1.0822	0.876
<b>Fe(II)</b>	1.3605	1.9653	0.0041	0.942	2.3659	1.3605	0.942

From the data above, it is observed that Langmuir isotherm model fitted the experimental data better than Freundlich isotherm model due to higher regression coefficient ranging from 0.942 to 0.981.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATIONS

Rice husk has been successfully approved to act as an adsorbent to remove Fe(II), Zn(II) and Cd(II) ions from aqueous solution. The adsorption process can be affected by the variation of parameters such as contact time, adsorbent dosage and initial concentration. The adsorption process is optimum at 100 mg/L of initial concentration, 70 minutes with presence of 0.5 grams adsorbent dosage of rice husk. Kinetic studies were conducted using pseudo-first order and pseudo-second order kinetic models, where the kinetic approved all three heavy metal fitted the second-order kinetic yielding good  $R^2$  values from 0.955 to 0.99 and  $k$  values of 0.1224 to 0.1607. Isotherm studies using Langmuir and Freundlich model shows that this experiment is well fitted with Langmuir model with maximum heavy metal uptake between 1.0822 to 1.4793 mg/g and Langmuir constants ranging from 0.7580 to 2.1191 L/mg.

There will be a few suggestions for the improvement of this research in the future. There are a few suggestions in order to improve the Fe(II), Zn (II) and Cd(II) ions removal capacities:

1. This research uses other potential adsorbents such as sawdust, coconut husk, oil palm shell, and sugarcane bagasse.

2. The analysis of removal efficiency should be tried using another equipment such as UV/Visible Spectrophotometric and Fourier transform infrared spectroscopy (FTIR).
3. This research uses another heavy metal such as Aluminium, Copper, Chromium and Magnesium.
4. The rice husk must be placed in the desiccators for a certain period until it is used for analysis because the air would reduce the surface area of the rice husk as the particles will combine together into the bigger particles of sizes.

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## APPENDICES

### Appendix A : Material Safety Data Sheet of Zinc(II) Sulfate

#### 1. Chemical Product and Information

**MSDS Name:** Zinc Sulfate Heptahydrate

**Synonyms:** Sulfuric acid zinc salt (1:1) heptahydrate

CAS#	Chemical Name	Percent	EINECS/ELINCS
7446-20-0	Zinc sulfate, Heptahydrate	100	unlisted

#### 2. Hazards Identification

##### EMERGENCY OVERVIEW

**Appearance:** colourless. Caution! May cause digestive tract irritation. Causes eye and skin irritation. Causes respiratory tract irritation.

**Target Organs:** None.

##### Potential Health Effects

**Eye:** Causes eye irritation.

**Skin:** Causes skin irritation.

**Ingestion:** May cause irritation of the digestive tract.

**Inhalation:** Causes respiratory tract irritation.

**Chronic:** Not available. None

#### 3. First-Aid Measures

**Eyes:** Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid.

**Skin:** Get medical aid. Flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes.

**Ingestion:** If victim is conscious and alert, give 2-4 cupfuls of milk or water. Never give anything by mouth to an unconscious person. Get medical aid.

**Inhalation:** Remove from exposure to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid.

**Notes to Physician:** Treat symptomatically and supportively.

**Antidote:** None reported.

#### 4. Fire-Fighting Measures

**General Information:** As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear.

**Extinguishing Media:** Substance is noncombustible; use agent most appropriate to extinguish surrounding fire.

#### 5. Accidental Release Measures

**General Information:** Use proper personal protective equipment as indicated in Section 8.

**Spills/Leaks:** Sweep up, then place into a suitable container for disposal. Avoid generating dusty conditions.

#### 6. Handling and Storage

**Handling:** Wash thoroughly after handling. Use with adequate ventilation. Minimize dust generation and accumulation. Avoid contact with eyes, skin, and clothing. Avoid ingestion and inhalation.

**Storage:** Store in a dry area. Store in a cool, dry, well-ventilated area away from incompatible substances.

#### 7. Physical and Chemical Properties

**Physical State:** Solid

**Appearance:** colourless

**Odor:** odorless

**pH:** Not available.

**Vapor Pressure:** Not available.

**Vapor Density:** Not available.

**Evaporation Rate:**Not available.

**Viscosity:** Not available.

**Boiling Point:** Not available.

**Freezing/Melting Point:**122 deg F

**Autoignition Temperature:** Not applicable.

**Flash Point:** Not applicable.

**Decomposition Temperature:**Not available.

**NFPA Rating:** Not published.

**Explosion Limits, Lower:**Not available.

**Upper:** Not available.

**Solubility:** Soluble in water

**Specific Gravity/Density:**1.96

**Molecular Formula:**ZnSO<sub>4</sub>.7H<sub>2</sub>O

**Molecular Weight:**287.5314

## 8. Solubility and Reactivity

**Chemical Stability:** Stable.

**Conditions to Avoid:** Excess heat.

**Incompatibilities with Other Materials:** None reported with materials and contaminants which the material may reasonably come into contact.

**Hazardous Decomposition Products:** Oxides of sulfur, toxic fumes of zinc oxide.

**Hazardous Polymerization:** Has not been reported.

## Appendix B: Material Safety Data Sheet of Iron(II) Sulfate

### 1. Chemical Product and Information

**MSDS Name:** Iron(II) sulfate heptahydrate

**Synonyms:** Green vitrol; Ferrous sulfate heptahydrate; Iron protosulfate.

CAS#	Chemical Name	Percent	EINECS/ELINCS
7782-63-0	Iron(II) sulfate heptahydrate	>99	unlisted

### 2. Hazards Identification

#### EMERGENCY OVERVIEW

**Appearance:** blue-green solid.

**Caution!** May cause eye and skin irritation. May cause respiratory tract irritation. May be harmful if swallowed. Air sensitive. Moisture sensitive.

**Target Organs:** Liver, gastrointestinal system, eyes, skin, mucous membranes.

#### Potential Health Effects

**Eye:** May cause mild eye irritation.

**Skin:** May cause skin irritation.

**Ingestion:** Ingestion of large amounts may cause gastrointestinal irritation. May be harmful if swallowed. Ingestion may result in irritation of the esophagus, bleeding of the stomach and ulcer formation. G.I. disturbances (e.g., gastric distress, colic, constipation, diarrhea) may occur if swallowed. In children, ingestion of large quantities of ferrous sulfate may cause vomiting, vomiting of blood, liver damage, rapid heart rate, peripheral vascular collapse.

**Inhalation:** May cause respiratory tract irritation.

**Chronic:** Repeated exposure may increase iron levels in the liver, spleen and lymphatic system. Damage may occur in the spleen and liver. Oral doses of 960 mg/kg given intermittently over a 9 week period produced jaundice in humans.

### 3. First-Aid Measures

**Eyes:** In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical aid.

**Skin:** In case of contact, flush skin with plenty of water. Remove contaminated clothing and shoes. Get medical aid if irritation develops and persists. Wash clothing before reuse.

**Ingestion:** If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Get medical aid.

**Inhalation:** If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid.

**Notes to Physician:** Treat symptomatically and supportively.

#### 4. Fire-Fighting Measures

**General Information:** As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. Substance is noncombustible.

**Extinguishing Media:** Substance is noncombustible; use agent most appropriate to extinguish surrounding fire.

**Flash Point:** Not applicable.

**Autoignition Temperature:** Not applicable.

**Explosion Limits, Lower:**Not available.

**Upper:** Not available.

**NFPA Rating:** (estimated) Health: 1; Flammability: 0; Instability: 0

#### 5. Accidental Release Measures

**General Information:** Use proper personal protective equipment as indicated in Section 8.

**Spills/Leaks:** Vacuum or sweep up material and place into a suitable disposal container. Clean up spills immediately, observing precautions in the Protective Equipment section. Avoid generating dusty conditions. Provide ventilation.

#### 6. Handling and Storage

**Handling:** Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Use with adequate ventilation. Minimize dust generation and accumulation. Avoid contact with eyes, skin, and clothing. Avoid breathing dust.

**Storage:** Do not store in direct sunlight. Store in a cool, dry area away from incompatible substances.

## 7. Physical and Chemical Properties

**Physical State:** Solid

**Appearance:** blue-green

**Odor:** odorless

**pH:** 3-5 (5% aq. sol.)

**Vapor Pressure:** Not available.

**Vapor Density:** Not available.

**Evaporation Rate:** Negligible.

**Viscosity:** Not available.

**Boiling Point:** 300 deg C (dec)

**Freezing/Melting Point:** 64 deg C

**Decomposition Temperature:** > 300 deg C

**Solubility:** 48.6g/100g water at 50C

**Specific Gravity/Density:** 1.898

**Molecular Formula:** FeSO<sub>4</sub>·7H<sub>2</sub>O

**Molecular Weight:** 278.01

## 8. Solubility and Reactivity

**Chemical Stability:** Stable at room temperature in closed containers under normal storage and handling conditions. FeSO<sub>4</sub>·7H<sub>2</sub>O oxidizes in moist air forming a brown coating of basic ferric sulfate. Aqueous solutions are oxidized slowly by air when cold, rapidly when hot; rate of oxidation increased by addition of base or exposure to light.

**Conditions to Avoid:** Light, dust generation, moisture, excess heat, prolonged exposure to air.

**Incompatibilities with Other Materials:** Strong oxidizing agents, bases, lead acetate, silver salts, lime water, carbonates, potassium tartrate, gold salts, potassium iodide, sodium borate, sodium tartrate, tannin.

**Hazardous Decomposition Products:** Oxides of sulfur, oxides of iron.

**Hazardous Polymerization:** Has not been reported.



### Appendix C: Result Data

**Table C.1** Effect of Contact Time for Fe(II)

Contact Time (min)	Initial Concentration, $C_o$ , (mg/l)	Final Concentration, $C_f$ (mg/l)	Removal (%)
10	100	63.10	36.90
20	100	44.40	55.60
30	100	37.50	62.50
60	100	29.10	70.90
90	100	27.00	73.00
120	100	24.60	74.70

**Table C.2** Effect of Adsorbent Dosage on Fe(II)

Adsorbent Dosage, g	Initial Concentration, $C_o$ , (mg/l)	Final Concentration, $C_f$ (mg/l)	Removal (%)
0.3 g	100	70.20	29.80
0.6 g	100	57.80	42.20
0.9 g	100	44.40	55.60
1.2 g	100	29.90	70.10
1.5 g	100	26.60	73.40
1.8 g	100	24.10	75.90

**Table C.3** Effect of Initial Concentration on Fe(II)

Concentration, mg/l	Final Concentration, $C_f$ (mg/l)	Removal (%)
10	8.9	91.10
25	18.50	81.50
50	41.80	58.20
75	60.00	40.00
100	75.89	24.11
125	82.00	18.00

**Table C.4** Effect of Contact Time for Zn(II)

Contact Time (min)	Initial Concentration, $C_o$ (mg/l)	Final Concentration, $C_f$ (mg/l)	Removal (%)
10	100	76.10	23.90
20	100	54.50	45.50
30	100	30.20	69.80
60	100	19.70	80.30
90	100	14.90	85.10
120	100	12.40	87.60

**Table C.5** Effect of Adsorbent Dosage on Zn(II)

Adsorbent Dosage, g	Initial Concentration, $C_o$ , (mg/l)	Final Concentration, $C_f$ (mg/l)	Removal (%)
0.3 g	100	63.60	36.40
0.6 g	100	50.20	49.80
0.9 g	100	40.20	59.80
1.2 g	100	25.70	74.30
1.5 g	100	21.10	78.90
1.8 g	100	19.80	80.20

**Table C.6** Effect of Initial Concentration on Zn(II)

Concentration, mg/l	Final Concentration, $C_f$ (mg/l)	Removal (%)
10	5.40	94.60
25	18.70	81.30
50	47.50	52.50
75	73.30	26.70
100	85.00	15.00
125	89.70	10.30

**Table C.7** Effect of Contact Time for Cd(II)

Contact Time (min)	Initial Concentration, $C_o$ , (mg/l)	Final Concentration, $C_f$ (mg/l)	Removal (%)
10	100	48.90	51.10
20	100	36.70	63.30
30	100	28.30	71.70
60	100	14.80	85.20
90	100	9.60	90.40
120	100	8.70	91.30

**Table C.8** Effect of Adsorbent Dosage on Cd(II)

Adsorbent Dosage, g	Initial Concentration, $C_o$ , (mg/l)	Final Concentration, $C_f$ (mg/l)	Removal (%)
0.3 g	100	59.80	40.20
0.6 g	100	31.90	68.10
0.9 g	100	20.70	79.30
1.2 g	100	17.90	82.10
1.5 g	100	16.20	83.80
1.8 g	100	17.60	85.40

**Table C.9** Effect of Initial Concentration on Cd(II)

Concentration, mg/l	Final Concentration, $C_f$ (mg/l)	Removal (%)
10	8.40	91.60
25	21.60	78.40
50	46.70	53.30
75	67.80	32.20
100	78.50	21.50
125	80.00	20.00

**Table C.10** Calibration Curve on Fe(II)

Concentration (mg/l)	Absorption (ml)
0.00	0.0000
2.00	0.0304
4.00	0.0402
6.00	0.0508
8.00	0.0592
10.00	0.0756

**Table C.11** Calibration Curve on Zn(II)

Concentration (mg/l)	Absorption (ml)
0.00	0.0002
2.00	0.0311
4.00	0.0598
6.00	0.1123
8.00	0.1678
10.00	0.1950

**Table C.12** Calibration Curve on Cd(II)

Concentration (mg/l)	ABS
0.00	0.0001
2.00	0.0511
4.00	0.0899
6.00	0.1360
8.00	0.1700
10.00	0.1890

