ADSORPTION OF Cu(II), Zn(II) AND Fe(II) USING *CHENGAL* SAWDUST

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ADSORPTION OF Cu(II), Zn(II) AND Fe(II) USING *CHENGAL* SAWDUST

NOR HIDAYAH BINTI AHMAD NORDIN

A thesis submitted in fulfillment
of the requirements for the award of the Degree of
Bachelor of Chemical Engineering (Chemical)

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

January 2012

I declare that this thesis entitled “Adsorption of Cu(II), Zn(II) and Fe(II) Using *Chengal* Sawdust” is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.”

Signature :………………………………

Name : Nor Hidayah binti Ahmad Nordin

Date :

Dedicated to my beloved family

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Assalamualaikum wbt…

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ABSTRACT

Adsorption of heavy metals by sawdust, an alternative utilization of waste, chosen to investigate the effectiveness of using sawdust to remove heavy metal ions from aqueous solutions for the adsorbent's economical feasibility and availability in large quantity. In this study, *Chengal* tree sawdust, an inexpensive material, were investigated as an adsorbent for the removal of Cu(II), Zn(II) and Fe(II) ions from aqueous solutions using batch techniques. The purpose of this research is to investigate the potential of sawdust as natural low cost adsorbent to adsorb Cu(II), Zn(II) and Fe(II) heavy metal ions by the variation of solution pH, contact time, initial concentration and adsorbent dosage. The adsorbent was prepared by collecting the *Chengal* sawdust, dried and ground the sawdust using grinder and mechanical sieve to specify the adsorbent to certain size. Adsorbate were prepared by diluting 250 mg/L of stock heavy metal solutions then used for testing the effect of removal of the heavy metal ions. 0.5g of sawdust as adsorbent with 100 ml adsorbate was kept constant for this experiment where the adsorbate solution varied in pH2 to 10, using concentrated 0.1M NaOH and 0.1M HCl to change pH and put in a orbital shaker, operated at 150rpm for 145minutes. For variation in contact time, the solution was set on the shaker for different sets of time interval 20minutes from minute 5 until minute 145 at constant 150rpm and 25˚C, with adsorbate concentration of 250mg/L. Initial adsorbate solution concentration of 250 mg/L was used with adsorbent sample varying in weight, 0.1g to 0.5g, set on shaker at constant 150 rpm and 25˚C. Adsorption process using *Chengal* sawdust was found to be suitable at pH4. Kinetic studies were conducted where both pseudo-first order and pseudo-second order yield values of R^2 from 0.6964 to 0.9453 and from 0.9995 to 1.0 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 Cu(II), Zn(II), and Fe(II) using *Chengal* sawdust ($0.9360 < R^2 < 0.9905$), and the maximum adsorption capacity was highest for Cu(II) with 25.71mg/g.

ABSTRAK

Penjerapan logam berat oleh habuk papan, penggunaan alternatif bahan buangan, dipilih untuk menyiasat keberkesanannya untuk mengeluarkan ion logam berat daripada larutan. Dalam kajian ini, habuk papan dari pokok Chengal, bahan murah, yang sedang disiasat sebagai adsorben untuk penyingkiran Cu (II), Zn (II) dan Fe (II) ion daripada larutan akueus. Tujuan kajian ini adalah untuk menyiasat potensi habuk papan sebagai dsorben semula jadi yang kos rendah untuk menjerap Cu (II), Zn (II) dan Fe (II) ion-ion logam berat semula jadi menggunakan variasi larutan pH, masa, kepekatan awal dan jumlah adsorben. Adsorben disediakan dengan mengumpul habuk papan Chengal, dikeringkan dan dikisar kepada saiz tertentu. Adsorbate disediakan dengan mencairkan 250 mg / L logam berat yang akan digunakan untuk menguji kesan penyingkiran ion-ion logam berat. 0.5g habuk papan sebagai adsorben dan 100 ml adsorbate adalah malar bagi eksperimen ini di mana bagi larutan pH berbeza dalam kumpulan pH; 2-10, menggunakan pekat 0.1M NaOH dan HCl 0.1M untuk menukar pH dan dimasukkan ke dalam penggoncang orbit, yang beroperasi pada 150rpm untuk 145minit. Bagi perubahan dalam masa, larutan digoncang untuk pada setiap sela masa 20minit dari minit ke5 sehingga minit ke 145 pada 150rpm malar dan 25˚C, dengan kepekatan larutan 250mg / L. Larutan dengan kepekatan awal 250 mg / L telah digunakan dengan sampel adsorben yang berbeza-beza berat dalam skala, 0.1g 0.5g, ditetapkan pada penggoncang malar 150 rpm dan 25 ° C. Proses penjerapan menggunakan habuk kayu Chengal didapati sesuai pH4. Kajian kinetik telah dijalankan di mana kedua-dua pseudo-tertib pertama dan kedua di mana hasil nilai R^2 dalam lingkungan 0.6964-0.9453 dan 0.9995-1.0 bagi setiap tertib. Penjerapan bagi Cu (II), Zn (II) dan Fe (II) akan diterangkan oleh kedua-dua *Langmuir* dan *Freundlich* model.

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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

Heavy metals are any metallic chemical element that has a relatively high density and is toxic at low concentrations (Sekhar et al., 1997).The removal of toxic and heavy metal contaminants from aqueous waste streams and industrial effluents is one of the most important environmental issues being faced the world over. Malaysia has been one of the most industrial countries all over the world, where many of the rivers have been polluted due industrial waste discharge into local rivers and seas.

According to World Health Organization, some of examples of heavy metal include aluminum, chromium, magnesium, iron, copper, nickel, zinc, cadmium, mercury and plumbum. Ahmad et al., 2009, stated that iron is the most widely used metal where its low cost and high strength makes it favorable in engineering applications such as construction of machinery and machinery tools, automobiles and components for building. Irons could also be found in our daily food such as red meat, poultry, beans and green vegetables. However, large amounts of ingested iron could cause liver failure, long-term organ damage or even death. Copper's applications include in production of electrical wires, roofing and plumbing, industrial machinery and production of alloy. Exposure to particulates or solution may cause conjunctivitis, ulceration, and corneal abnormalities. Zinc is commonly used in alloy production and as an anti-corrosion agent by galvanization process. Excessive exposure to zinc can result in loss of appetite, decreased sense of taste and smell, slow wound healing and skin sores. Zinc-shortages can even cause birth defects. The search for alternate and innovative treatment techniques focused on natural byproduct which is environmental friendly that could be used as an adsorbent.

In order to combat this problem, the commonly used procedures for removing metal ions from dilute aqueous streams include chemical precipitation, ion-exchange, reverse osmosis and solvent extraction (Rich et al., 1987). However, these techniques have certain disadvantages such as incomplete metal removal, high reagent and energy requirements, generation of toxic sludge or other waste products that require disposal. The hazardous wastes generated from mining and smelting operations also need iron be decontaminated before entering the ecosystem.

Adsorption process is selected in this research because it is quite selective, effective, and able to remove various levels of soluble heavy metals in solution. In recent years, considerable attention has been focused on the removal of heavy metals using biosorbents derived from low-cost materials. Several biosorbents such as peat, tea waste, coconut husk, sewage sludge, and rice husk have been used for the treatment of metals in aqueous solution (Jang et al., 2005).

In this study, sawdust, which has environmental benefits in terms of the reuse of solid waste, was tested to evaluate its potential for the treatment of heavy metals. In the present investigation, the potential of a byproduct waste has been assessed for the removal of metal ions such as copper, zinc, and iron. The effect of various parameters such as solution pH, contact time and adsorbent doses with respect to the percentage of removal of metal ions has been studied. The objective of this study is to assess the feasibility of utilizing sawdust for the adsorption of heavy metals in aqueous solution by the variation of the given parameters.

1.2 PROBLEM STATEMENT

The increasing of industries in Malaysia lead to the increasing of industrial effluents discharged to local rivers and seas, where the increasing of waste disposal to water system with improper waste water treatment could cause water pollution. The usage of advanced technologies for wastewater treatment is too expensive and not economically feasible. Hence, another alternative of low cost adsorption system is tested using sawdust, unused residues that could hopefully help to reduce pollution.

Malaysia, a tropical country is one of the world’s largest wood suppliers through nation due to its location surrounded with tropical equatorial rainforest. Lumber factories that produces waste from lumber cutting which is sawdust is being thrown away or burned, some are used for production of wood based panels, mushroom production, animal husbandry, and building products. 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 sawdust or waste product. Thus, this research will determine whether sawdust could be an effective adsorbent for the removal of heavy metal ions contained in wastewater discharged by the industries.

1.3 OBJECTIVE

To investigate the potential of sawdust as natural adsorbent to adsorb Cu(II),Zn(II) and Fe (II) ions by the variation of solution pH, contact time, initial concentration and adsorbent doses.

1.4 SCOPE

1.41 Analyzation of the potential of sawdust as a low cost natural adsorbent to adsorb heavy metal ions from solution.

1.42 Investigation and observation of the process condition effect for Cu(II), Zn(II) and Fe(II) that can be removed by using sawdust.

1.42 Determination of the effect of process parameters to the percentage removal efficiency of heavymetal ions by analyzing the result of initial and final concentration for each variable using Atomic Adsorption Spectrometer:

1.4.2.1 Solution pH

1.4.2.2 Contact time

1.4.2.3 Dosage of adsorbent

1.4.2.4 Initial Concentration

1.5 RATIONALE AND SIGNIFICANCE

In this experiment, heavy metal ions, Cu(II), Zn(II) and Fe (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. Several biomaterials such as tea waste, rice husk, coconut husk, oil palm fibre and sawdust are low cost waste residues and easily available in large quantities in Malaysia.

The utilization of natural and agriculture by-product as adsorbent not only economically feasible but instead of throwing away waste, it could be functional to be used as heavy metal ions adsorbent to reduce pollution. Sawdust 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 lumber factories, and also benefit for the environment. Hence, from sawdust which ones was a waste, we could conventionalize it to become wealth.

CHAPTER 2

LITERATURE REVIEW

Ahmad et al. (2009) investigated the removal of Cu (II) and Pb (II) ions from aqueous solutions by adsorption on sawdust of meranti wood. 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.

Balkaya et al. (2006) had studied the adsorption of cadmium on phosphogypsum, a waste material from the manufacture of phosphoric acid by wet process. Before batch adsorption study, phosphogypsum was pre-conditioned by milk of lime. Effect of initial pH on cadmium adsorption was investigated. It was found that cadmium adsorption was dependent on solution pH and maximum cadmium removal was observed in the pH range of

9.5 and 11.5. The Langmuir and Freundlich theories were used to describe the cadmium adsorption process, and the Freundlich isotherm showed the best fit to the process. Maximum adsorption capacity of lime-preconditioned phosphogypsum was found to be 131.58 mg/g.

Yasemin et al. (2006) had studied the adsorption of lead, cadmium and nice1 from aqueous solution by sawdust of walnut. The effect of contact time, initial metal ion concentration and temperature on metal ions removal has been studied. The equilibrium time was found to be of the order of 60 min. Kinetics fit pseudo first-order, second-order and intraparticle diffusion models, hence adsorption rate constants were calculated. The adsorption data of metal ions at temperatures of 25, 45 and 60°C have been described by the Freundlich and Langmuir isotherm model. The thermodynamic parameters such as energy, entropy and enthalpy changes for the adsorption of heavy metal ions have also been manipulated and discussed. Ion exchange is probably one of the major adsorption mechanisms for binding divalent metal ions to the sawdust. The selectivity order of the adsorbent is Pb (II)> Cd (II)> Ni II). From these results, it can be concluded that the sawdust of walnut could be a good adsorbent for the metal ions from aqueous solutions.

Memon et al. (2006) had studied the ability of sawdust (treated and untreated) waste, a waste material derived from the commercial processing of cedrus deodar wood for furniture production, to remove/preconcentrate Cd (II) ions from aqueous solution. Sorption was found to be rapid (97% within 8 min). The binding of metal ions was found to be pH dependent, optimal sorption accruing at around pH 4–8. Potentiometric titrations of sawdust revealed two distinct pKa values, the first having the value similar to carboxylic groups (3.3–4.8) and second comparable with that of amines (8.53–10.2) with the densities 1.99×10^{-4} and 7.94×10^{-5}, respectively. Retained Cd (II) ions were eluted with 5ml of 0.1 mol l^{-1} HCl. Detection limit of 0.016 $\mu g ml^{-1}$ was achieved with enrichment factors of 120. Recovery was quantitative using sample volume of 600 ml. The Langmuir and D–R isotherm equations were used to describe partitioning behaviour for the system at different temperatures. Kinetic and thermodynamic behaviour of sawdust for Cd (II) ions removal was also studied.

M.M.El Jamal et. al. (2011) explained the kinetic and equilibrium study of adsorption of some dyes onto feldspar by the effect of dye concentration, pH, mass of adsorbent, temperature and shaking speed. His experimental data fitted the pseudo-second order kinetics and maximum adsorption capacity is 0.66mg/g at 40˚C by using Langmuir isotherm study, with endothermic process.

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 R2 =0.9946 and the maximum monolayer adsorption capacity was found to be 40.0 mg g-1 at 298K.

CHAPTER 3

METHODOLOGY

3.1 MATERIALS

i. Sawdust
ii. Copper(II) Sulphate $CuSO4$
iii. Ferrous (II) Sulphate $FeSO4$
iv. Zinc(II) Sulphate $ZnSO4$
v. 0.1N NaOH
vi. 0.1N HCl

3.2 APPARATUS

i. 100ml Beaker
ii. 1L and 100ml Measuring cylinder
iii. 100ml Conical flask
iv. Aluminium Foil
v. pH meter
vi. Atomic Adsorption Spectrometer
vii. Electronic balance
viii. Sieve
ix. Oven
x. Tyler Mesh (to label samples)
xi. Glass Rod
xii. Dropper
xiii. 1L Volumetric Flask
xiv. Funnel
xv. Whatman Filter Paper 125mm

3.3 EXPERIMENTAL PROCEDURES

3.3.1 Preparation of Sawdust

The sawdust used in the present investigation as an adsorbent collected from lumber factories. The sawmill produced during lumber cutting is called sawdust. The collected materials were then washed up with tap water followed by distilled water for several times to remove dirt particles. Then, the washed sawdust was then dried at 60˚C for 24 hours in oven. The dried material is then crushed and sieved in size range of 100-150 µm particle size using a sieve and stored in bottle for use. (Basiru et al. 2010)

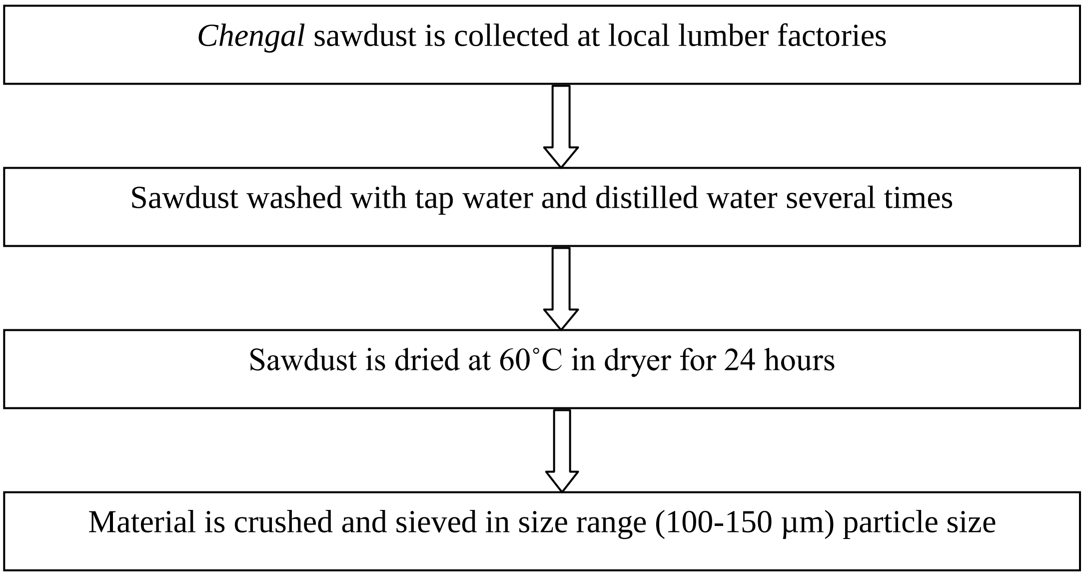
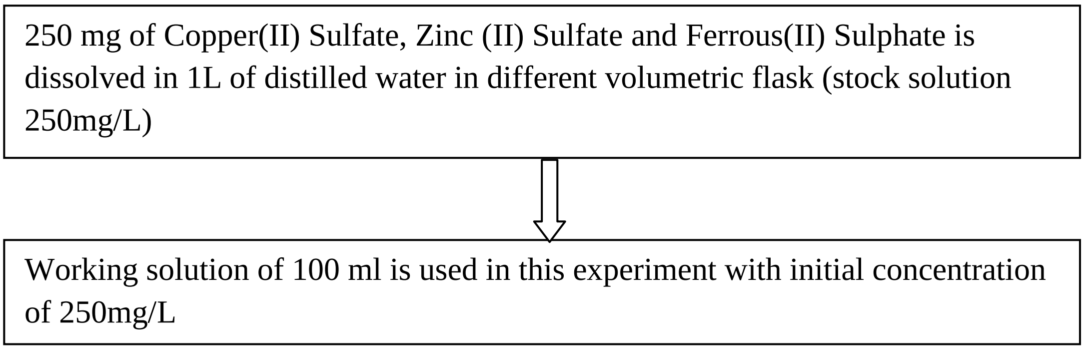


Figure 3.1 Flow Diagram for Preparation of Sawdust

3.3.2 Preparation of Adsorbate

Solution will be prepared by dissolving 250 mg of Copper(II) Sulfate, Zinc(II) Sulfate and Ferrous(II) Sulphate in 1L of distilled water in different 1L volumetric flask.



250 mg of Copper(II) Sulfate, Zinc (II) Sulfate and Ferrous(II) Sulphate is dissolved in 1L of distilled water in different volumetric flask (stock solution 250mg/L)

Working solution of 100 ml is used in this experiment with initial concentration of 250mg/L

Figure 3.2 Flow Diagram for Preparation of Adsorbate

3.3.3 Effect of pH

1 g of sawdust as adsorbent with 100 ml adsorbate was kept constant for this experiment. Concentrated 0.1M NaOH and 0.1M HCl is used to change pH from 2 to 10 so that the change in volume of the solution is negligible. Adsorbate solution shaked at 25˚C on an orbital shaker operated at 150rpm for 145 minutes. Sample is filtered using Whatman Filter Paper 125mm to be analyzed at AAS.

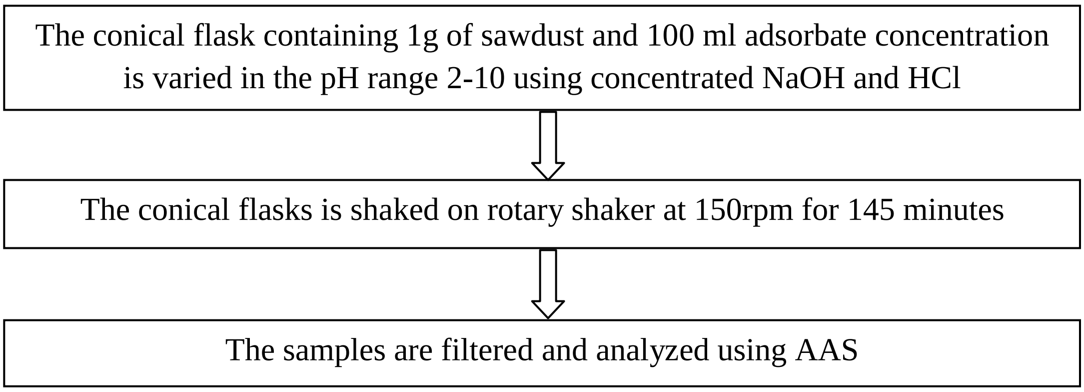


Figure 3.3 Flow Diagram of The Effect of Solution pH

3.3.4 Effect of Contact Time

In this experiment, the adsorbate solution concentration is 250mg/L. The effect of contact time investigated for 5min, 25min, 45min, 65min, 85min, 105min, 125min and 145min at the pH4 constant and sample of 0.5g adsorbent dosage and shake on an orbital shaker operated at 150 rpm and 25˚C. Samples are filtered using Whatman filter paper and analyzed using AAS.

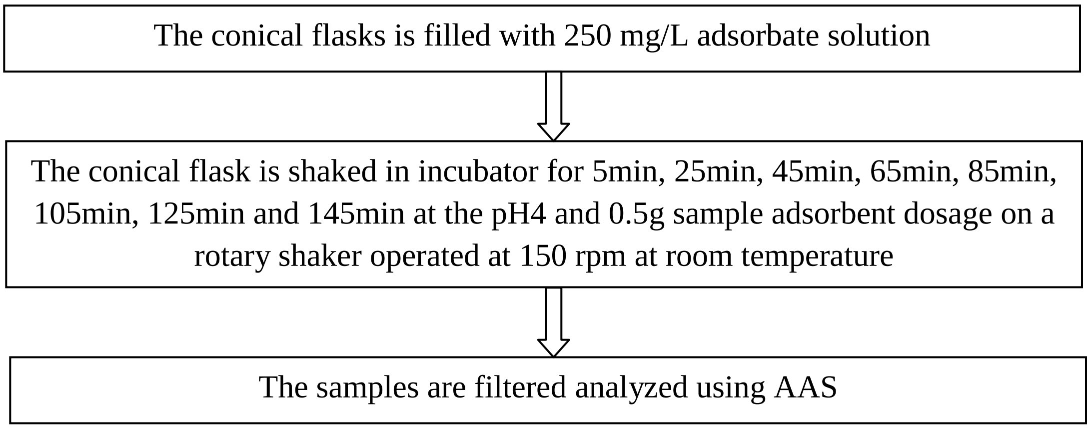


Figure 3.4 Flow Diagram of The Effect of Contact Time

3.3.5 Effect of Adsorbent Dosage

Initial adsorbate solution concentration of 250 mg/L was used with adsorbent sample varying in weight, 0.1g, 0.2g, 0.3g, 0.4 g, and 0.5g. The contact time and pH kept constant at 145min and pH4 that can be obtained from previous experiment before conducting the effect of sawdust dosage experiment at 25˚C. . Samples are filtered using Whatman filter paper and analyzed using AAS.

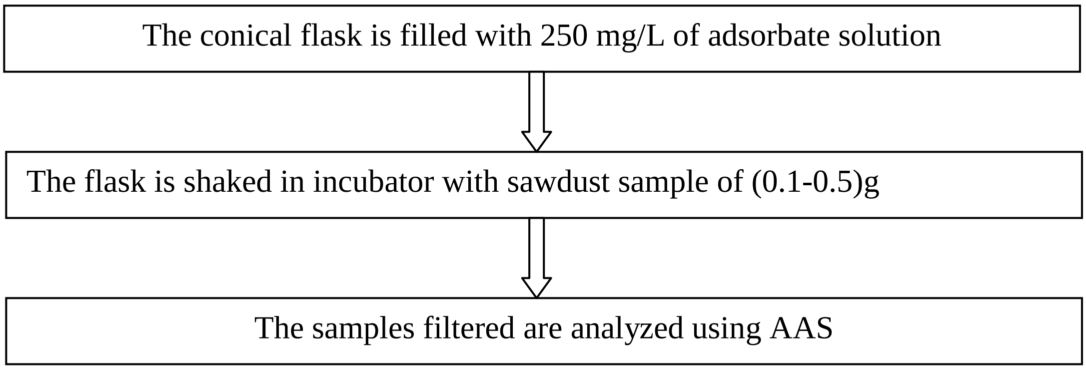


Figure 3.5 Flow Diagram of The Effect of Adsorbent Dosage

3.3.6 Effect Of Initial Concentration

In this experiment, the initial adsorbate solution concentration was varied from 50mg/L, 100mg/L, 150mg/L, 200mg/L and 250mg/L. The effect initial concentration is analyzed using constant solution pH of pH4, 0.5g of adsorbent dosage and sample contact time of 145min and shake sample on an orbital shaker operated at 150 rpm and 25˚C.

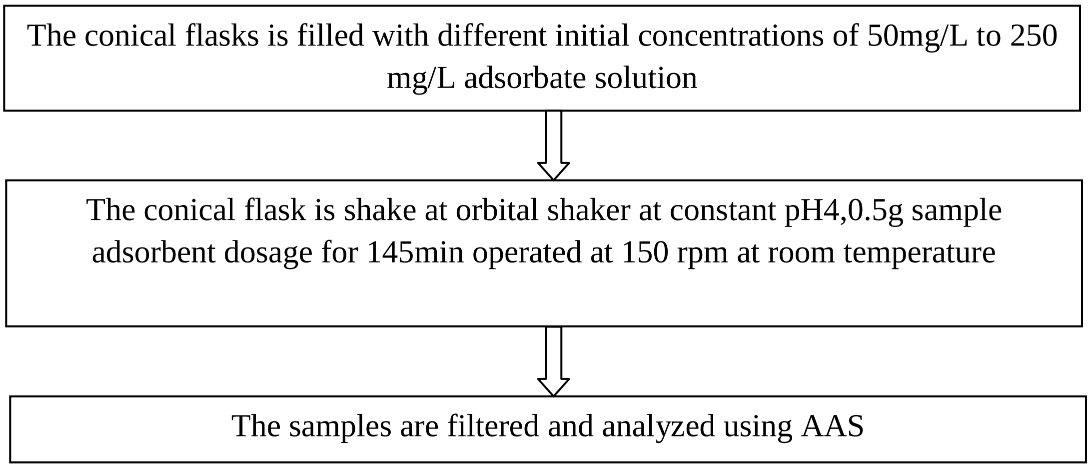


Figure 3.6 Figure Diagram of The Effect of Initial Concentration

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.

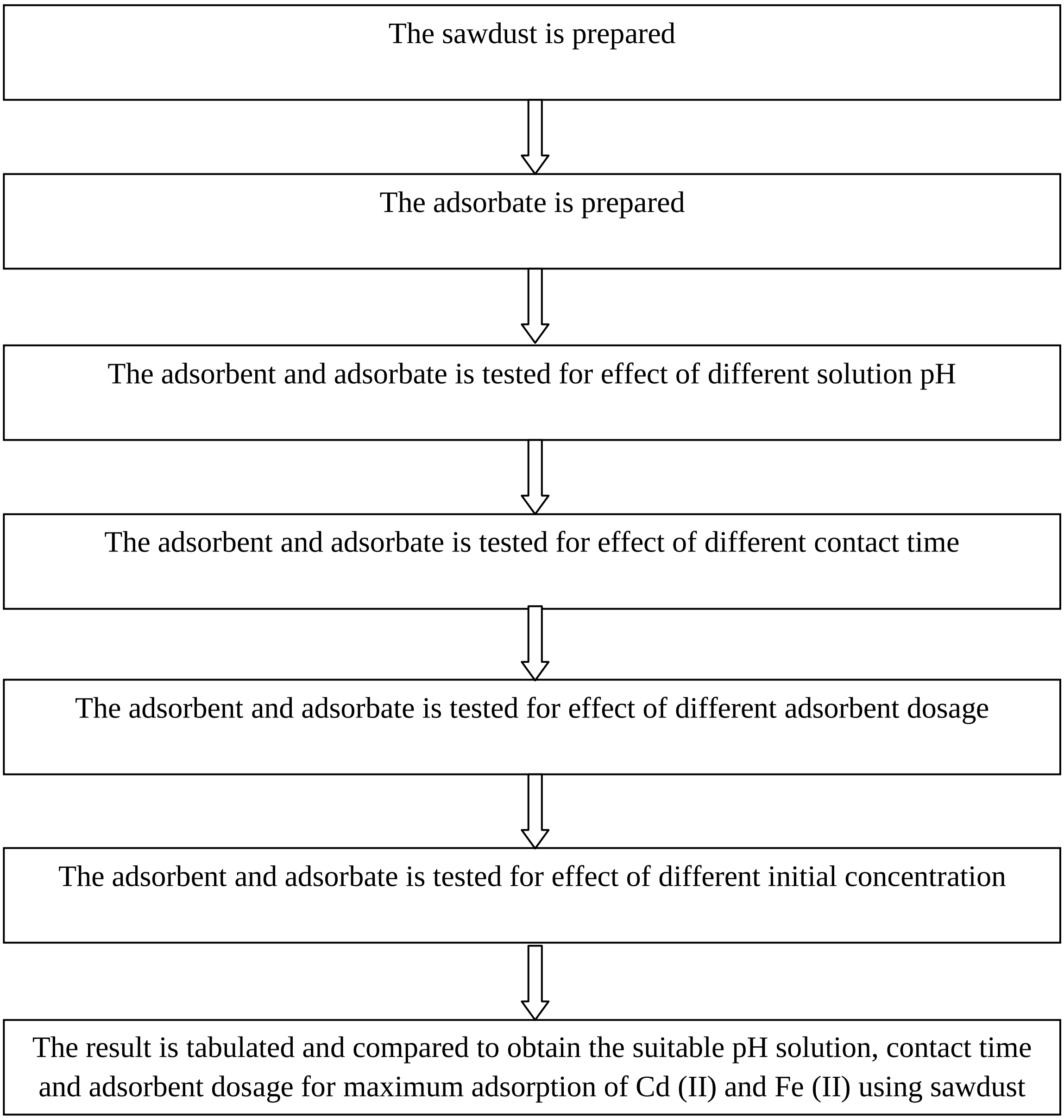


Figure 3.7 Flow Diagram of Adsorption of Cu(II), Zn(II) and Fe(II) Using Sawdust Process

3.3.7 Analyzing Sample

Standard solutions are prepared before running the Atomic Adsorption Spectrometer. Solutions are prepared by diluting 1000mg/L of standard into a 100ml volumetric flask for concentration 50mg/l with standard solution ranging from 0 to 10 by using the equation.

$$M1V1=M2V2$$

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 & DISCUSSION

4.1 Effect of Solution pH

Adsorbate solution pH is an important parameter that can influence heavy metal adsorption. It affects both the surface charge of adsorbent and the degree of ionization of the heavy metal in solution (Aksu, 2005). The effect of the adsorbent dosage was studied at room temperature (25˚C) by varying the solution pH, for pH2 pH4, pH6, pH8 and pH10 while the parameters of adsorbent dosage, contact time and initial concentration was kept constant for 0.5g, 145minutes, and 250mg/L.

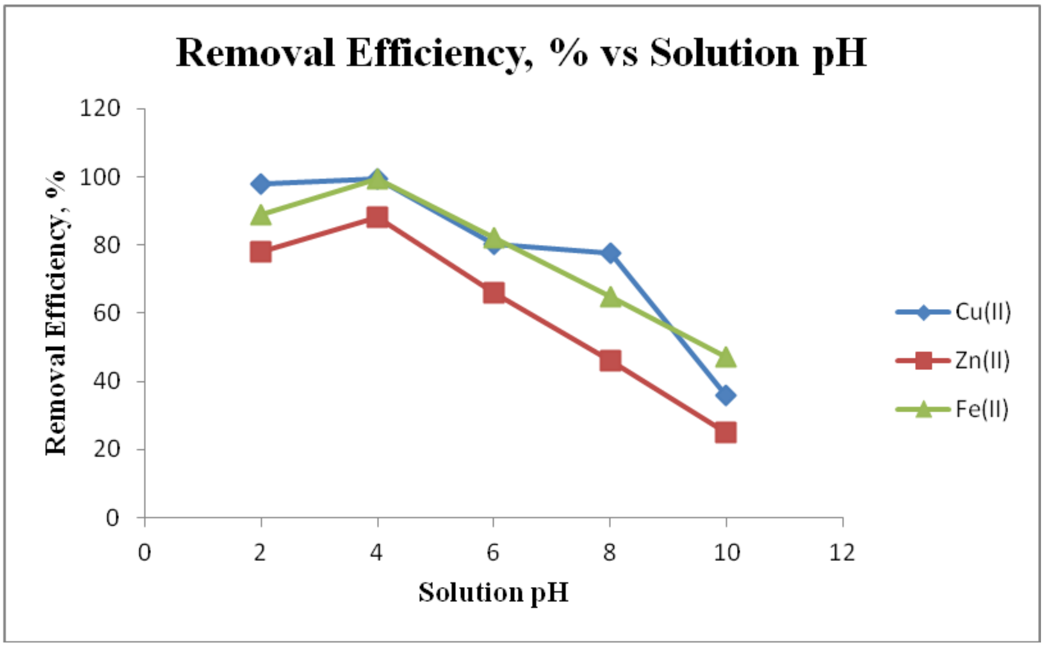


Figure 4.1 Graph of Removal Efficiency, % vs Solution pH

Figure above represents the effect of initial pH of the solution on the adsorption of heavy metal where it is observed that the maximum heavy metal removed happens at solution pH 4. This may be attributed to the competition between the hydrogen and heavy metal ions on the sorption sites, at low pH values. At pH value of 4, the concentration of proton decreased which reduces the competition with heavy metal ions for adsorption sites. As solution pH further increased, heavy metal became insoluble and produces precipitation which makes it impossible for adsorption process to happen resulting in the decreasing of removal efficiency.

4.2 Effect of Contact Time

Adsorption of heavy metals Cu(II), Zn(II), and Fe(II) was measured at different contact time for 8 interval time of 20 minute, starting from minute 5 to minute 145 at constant initial concentration of heavy metal solution of 250mg/L, amount of 0.5g *Chengal* sawdust, solution pH 4 and let to shake at 150 rpm and 25˚C using mechanical shaker. The graph if heavy metal removal efficiency, % versus contact time, minute is plotted by using formula below

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

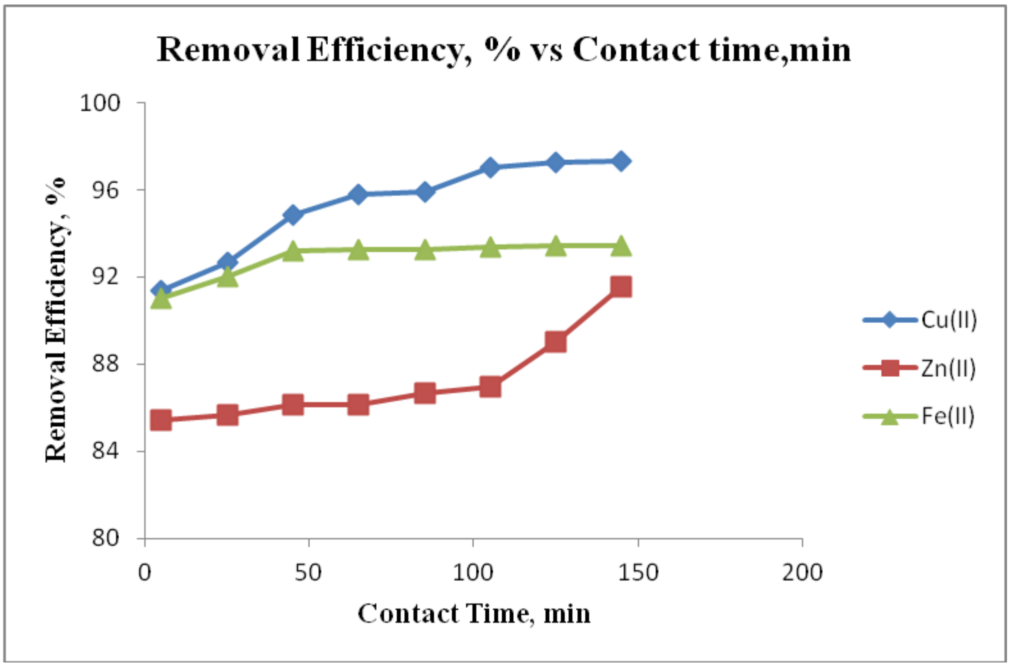


Figure 4.2 Graph of Heavy Metal Efficiency, % vs Contact Time, min

Figure and Table below shows that the removal efficiency for Cu(II) is the highest with the maximum removal efficiency of 97.32%, followed by Fe(II) with 93.44% and Zn(II) with 91.52%. 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 sawdust hence increasing the removal efficiency of heavy metal. Most of the maximum percent heavy metal removal was attained after about 145 min of contact time with the sawdust.

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 0.1g to 0.5g. For all these runs, initial concentration of heavy metal, solution pH and contact time was fixed as 250mg/L, pH4 and 145 minutes of contact time.

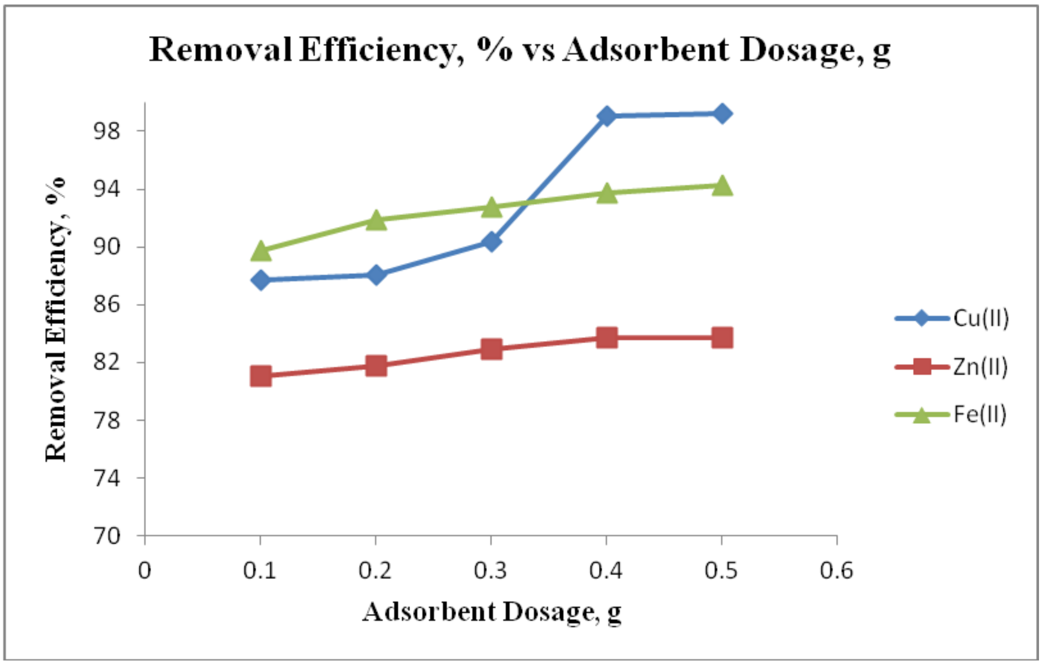


Figure 4.3 Graph of Removal Efficiency, % vs Adsorbent Dosage, g

Based on the graph above, removal efficiency for Cu(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 ion uptake.

4.4 Effect of Initial Concentration

The effect of heavy metal concentration in the range of 50 to 250 mg/L on removal efficiency was observed with constant solution pH4, contact time of 145minutes and 0.5g of adsorbent.

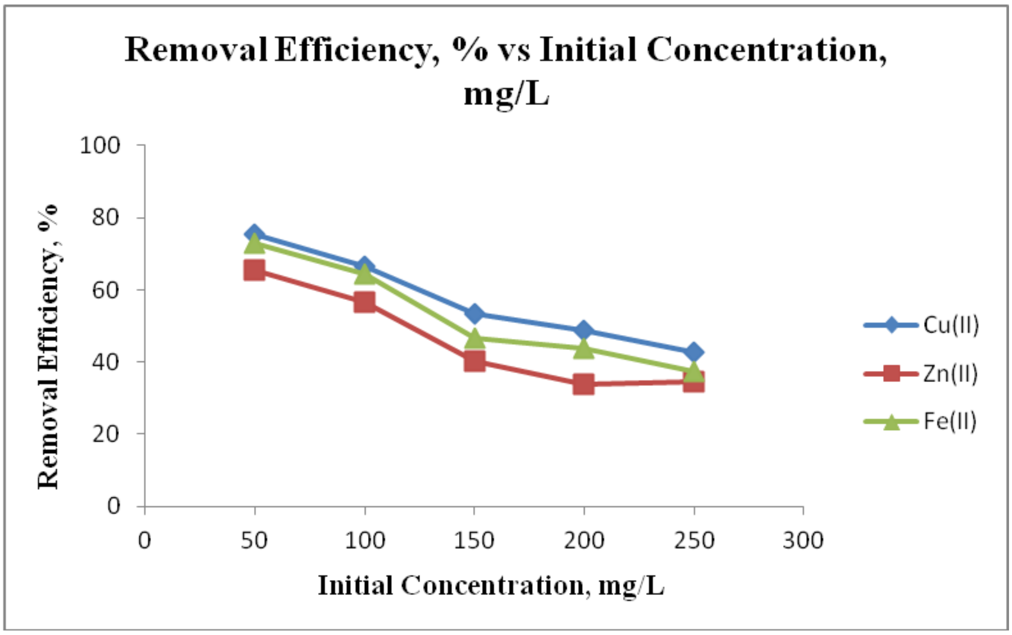


Figure 4.4 Graph of Removal Efficiency, % vs Initial Concentration, mg/L

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 250mg/L of adsorbate, with the removal efficiency of 42.64%, 34.38% and 37.44% for Cu(II), Zn(II) and Fe(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, *qe* 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 *qe* 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

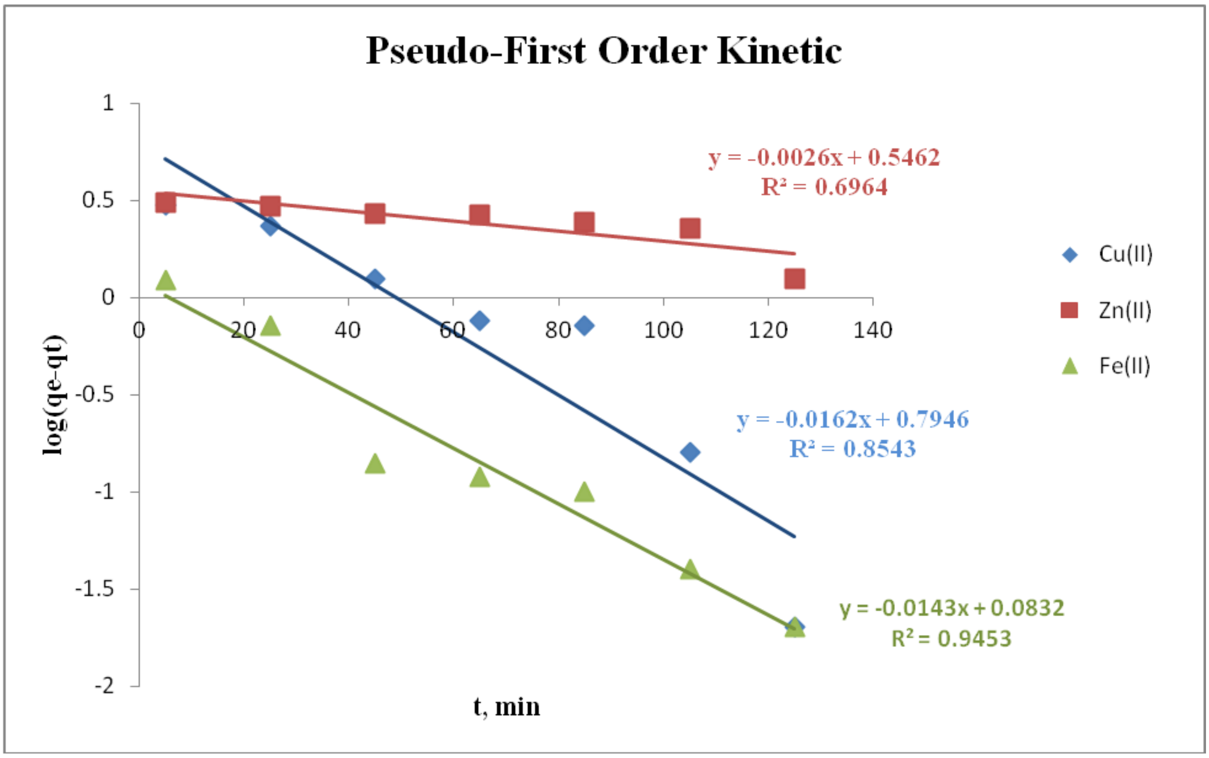


Figure 4.5 Pseudo-First Order Kinetic for Cu(II), Zn(II) and Fe(II) Adsorption Using *Chengal* Sawdust

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 absorbed 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.

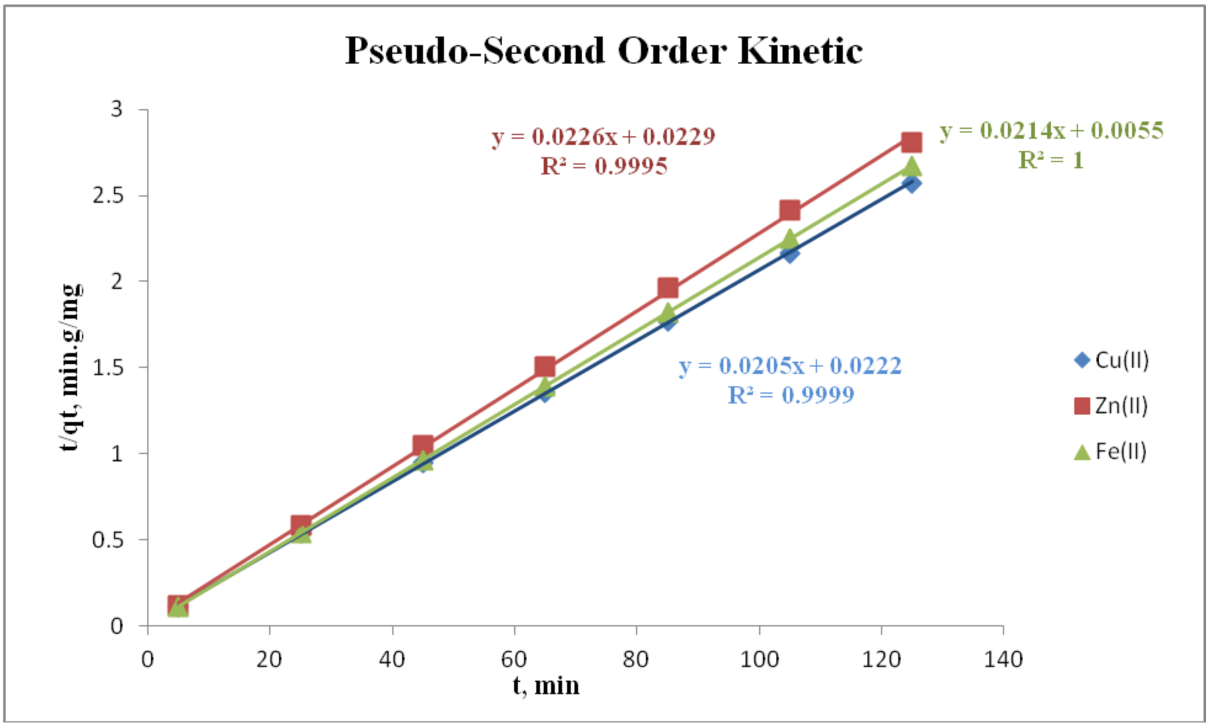


Figure 4.6 Pseudo-Second Order Kinetic for Cu(II), Zn(II) and Fe(II) Adsorption Using *Chengal* Sawdust

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

Table 4.1 Pseudo-First Order and Pseudo-Second Order Kinetic Model Constants

Heavy metal	Pseudo-First Order Kinetic		Pseudo-Second Order Kinetic	
	k1ads, L/min	R2	k2ads, g/mg.min	R2
Cu(II)	0.0373086	0.8543	0.01893018	0.9999
Zn(II)	0.0059878	0.6964	0.023007207	0.9995
Fe(II)	0.0329329	0.9453	0.020628829	1

From the calculated values above, the adsorption process of Cu(II), Zn(II) and Fe(II) heavy metal ions are more favourable towards pseudo-second order kinetics due to higher value of R2 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 *qm* 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.

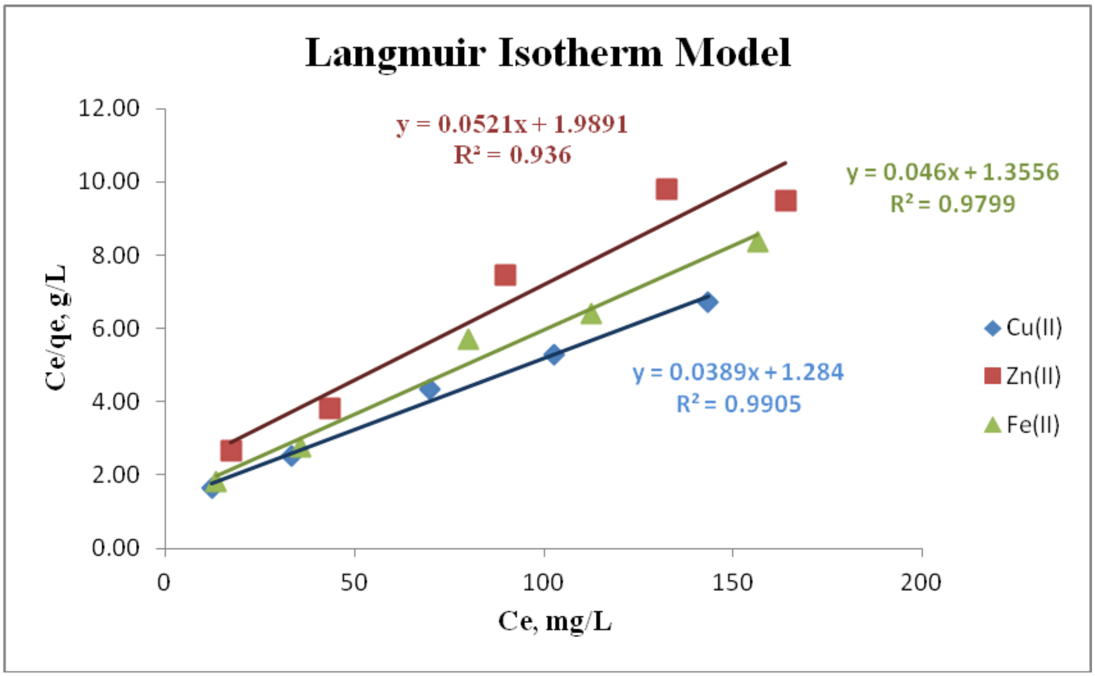


Figure 4.7 Langmuir Isotherm for Cu(II), Zn(II) and Fe(II) Adsorption Using *Chengal* Sawdust

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, *Kf* 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, *Kf* and n are obtained by plotting the graph between of ln *qe* versus ln *Ce*.

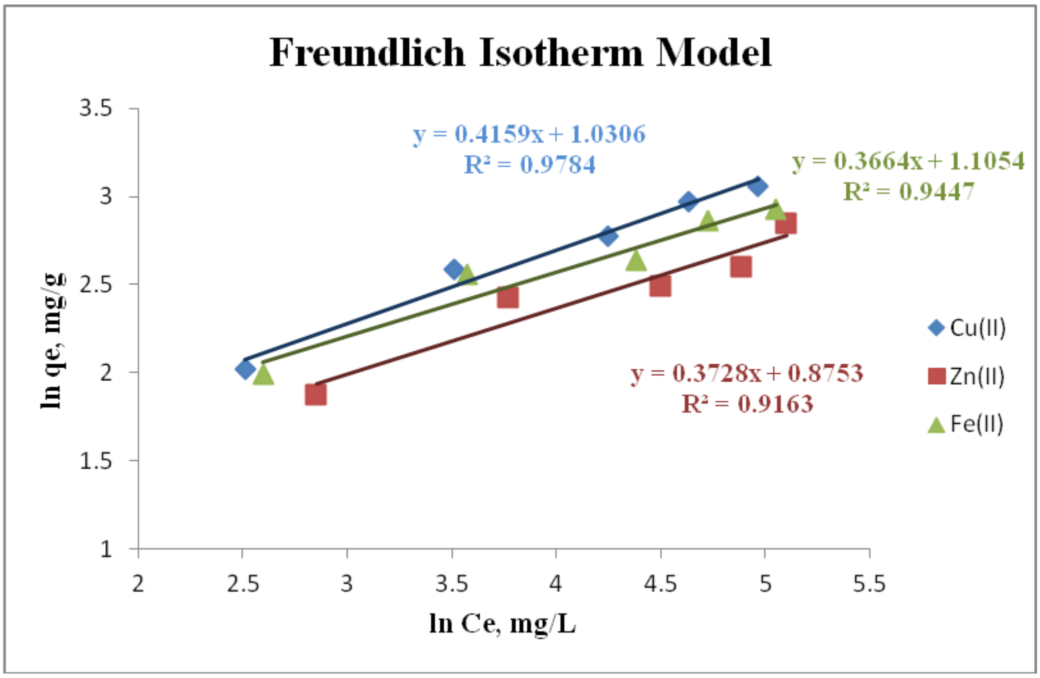


Figure 4.8 Freundlich Isotherm for Cu(II), Zn(II) and Fe(II) Adsorption Using *Chengal* Sawdust

Cu(II) yields higher value of R2compared to Fe(II) and Zn(II) ions where it is adsorbed more onto the sawdust, with *Kf* 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 4.2 Langmuir and Freundlich Adsorption Isotherm Model Constants

	Langmuir				**Freundlich**		
	qmax, mg/g	**KL, l/mg**	**RL**	**R2**	**Kf, (mg/g)(l/mg)^(1/n)**	**n**	**R2**
Cu(II)	25.7069	20.0210	0.0002	0.9905	2.8027	2.4044	0.9784
Zn(II)	19.1939	9.6495	0.0004	0.9360	2.3996	2.6824	0.9163
Fe(II)	21.7391	16.0365	0.0002	0.9799	3.0204	2.7293	0.9447

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 0.9360 to 0.9905.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The study has successfully proven that sawdust of *Chengal* is an effective adsorbent for Cu(II), Zn(II) and Fe(II) removal from aqueous solutions. The results obtained shows that the adsorption of Cu(II) is the highest among all three heavy metal ions. Adsorption process is also strongly dependent on contact time, adsorbent dosage, solution pH and initial adsorbate concentration. In this study, adsorption process occurs maximum removal efficiency at contact time 145 minutes, adsorbent dosage of 0.5g, solution pH of 4 and initial adsorbate concentration of 150mg/L. 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 R2 values from 0.99 to 1.00 and k values of 0.01893-0.0230. Isotherm studies using Langmuir and Freundlich model shows that this experiment is well fitted with Langmuir model with maximum heavy metal uptake between 19.19 to 25.73 mg/g and Langmuir constants ranging from 9.65 to 20.02 L/mg.

This study should be tested by using real wastewater to observe the removal efficiency of heavy metal ions from the wastewater. Heavy metal, in general, exhibits harmful effects on humans and the environment. They are moderately toxic compound. Exposure to heavy metal can occur through inhalation, ingestion, and eye or skin contact causes irritation, redness, and tearing of the eyes and irritation of the nose and skin sores. Personal Protective Equipments such as gloves, face mask, *etc.* should be worn when deal with heavy metal, as a safety precaution.

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APPENDICES

Appendix A: Flowchart Experiment

Chengal sawdust preparation

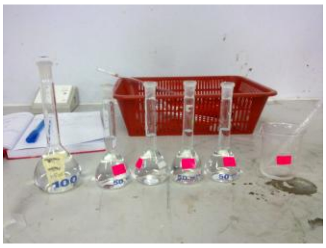
Adsorbate preparation

Cu(II), Fe(II) and Zn(II) weighed at 0.25g for adsorbate preparation.

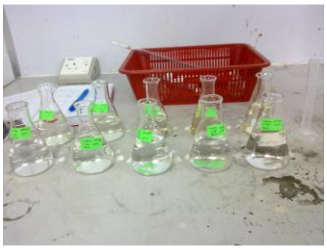
Preparation of sample based on parameter tested

Sample placed onto orbital shaker

Solution filtered using Whatman filter paper



Standard solution preparation



Sample dilution preparation

Sample analyzed using AAS

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

1. Chemical Product and Information

MSDS Name: Copper (II) Sulfate Anhydrous
Synonyms: Copper monosulfate; Cupric sulfate; Cupric sulfate anhydrous; Sulfuric acid, copper(2+) salt (1:1).

CAS#	Chemical Name	Percent	EINECS/ELINCS
7758-98-7	Copper(II) sulfate	>97	231-847-6

2. Hazards Identification

Appearance: light gray powder.

Warning! Harmful if swallowed. Causes eye and skin irritation and possible burns. Causes digestive and respiratory tract irritation with possible burns. Hygroscopic (absorbs moisture from the air). Severe marine pollutant.

Target Organs: Blood, kidneys, liver.

Potential Health Effects

Eye: Exposure to particulates or solution may cause conjunctivitis, ulceration, and corneal abnormalities. Causes eye irritation and possible burns.

Skin: Causes skin irritation and possible burns.

Ingestion: Harmful if swallowed. May cause severe gastrointestinal tract irritation with nausea, vomiting and possible burns. Ingestion of large amounts of copper salts may cause bloody stools and vomit, low blood pressure, jaundice and coma. Ingestion of copper compounds may produce systemic toxic effects to the kidney and liver and central nervous excitation followed by depression.

Inhalation: May cause ulceration and perforation of the nasal septum if inhaled in excessive quantities. Causes respiratory tract irritation with possible burns.

Chronic: May cause liver and kidney damage. May cause anemia and other blood cell abnormalities. Individuals with Wilson's disease are unable to metabolize copper. Thus, copper accumulates in various tissues and may result in liver, kidney, and brain damage. Adverse reproductive effects have been reported in animals. Laboratory experiments have resulted in mutagenic effects. Chronic copper poisoning in man is recognized in the form of Wilson's disease.

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 water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse.
Ingestion: Do not induce vomiting. 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 immediately.
Inhalation: Remove from exposure and move to fresh air immediately. If breathing is difficult, give oxygen. Get medical aid. Do NOT use mouth-to-mouth resuscitation. If breathing has ceased apply artificial respiration using oxygen and a suitable mechanical device such as a bag and a mask.
Notes to Physician: Individuals with Wilson’s disease are more susceptible to chronic copper poisoning.
Antidote: The use of d-Penicillamine as a chelating agent should be determined by qualified medical personnel.

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. During a fire, irritating and highly toxic gases may be generated by thermal decomposition or combustion. Substance is on-combustible.
Extinguishing Media: Use extinguishing media most appropriate for the surrounding fire.
Flash Point: Not applicable.
Autoignition Temperature: Not applicable.
Explosion Limits, Lower: Not available.
Upper: Not available.
NFPA Rating: (estimated) Health: 2; 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. Avoid runoff into storm sewers and ditches which lead to waterways. Clean up spills immediately, observing precautions in the Protective Equipment

section. Avoid generating dusty conditions. Provide ventilation. U.S. regulations require reporting spills and releases to soil, water and air in excess of reportable quantities.

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: Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Store protected from moisture.

7. Physical and Chemical Properties

Physical State: Powder
Appearance: light gray
Odor: Odorless
pH: Not available.
Vapor Pressure: Not available.
Vapor Density: Not applicable.
Evaporation Rate: Negligible.
Viscosity: Not available.
Boiling Point: Not available.
Freezing/Melting Point:200 deg C
Decomposition Temperature:560 deg C
Solubility: Soluble.
Specific Gravity/Density:3.6
Molecular Formula: CuO4S
Molecular Weight:159.61

8. Solubility and Reactivity

Chemical Stability: Stable at room temperature in closed containers under normal storage and handling conditions.
Conditions to Avoid: High temperatures, dust generation, exposure to moist air or water.
Incompatibilities with Other Materials: Aqueous solution of copper(2+) sulfate is an acid. Incompatible with strong bases, hydroxylamine, magnesium..
Hazardous Decomposition Products: Oxides of sulfur, copper fumes.
Hazardous Polymerization: Will not occur

Appendix C: 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:$ZnSO_4.7H_2O$

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 D: 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 a t 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:$FeSO_4 \cdot 7H_2O$

Molecular Weight:278.01

8. Solubility and Reactivity

Chemical Stability: Stable at room temperature in closed containers under normal storage and handling conditions. $FeSO_4 \cdot 7H_2O$ 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 E: Result Data

Table E.1 Effect of Contact Time for Cu(II)

Cu(II)			
Contact Time, min	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
5	250	21.6	91.36
25	250	18.4	92.64
45	250	12.9	94.84
65	250	10.5	95.80
85	250	10.3	95.88
105	250	7.5	97.00
125	250	6.8	97.28
145	250	6.7	97.32

Table E.2 Effect of Contact Time for Zn(II)

Zn(II)			
Contact Time, min	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
5	250	36.5	85.40
25	250	35.9	85.64
45	250	34.7	86.12
65	250	34.6	86.16
85	250	33.4	86.64
105	250	32.6	86.96
125	250	27.4	89.04
145	250	21.2	91.52

Table E.3 Effect of Contact Time for Fe(II)

Fe(II)			
Contact Time, min	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
5	250	22.4	91.04
25	250	19.9	92.04
45	250	17.0	93.2
65	250	16.9	93.24
85	250	16.8	93.28
105	250	16.5	93.4
125	250	16.4	93.44
145	250	16.4	93.44

Table E.4 Effect of Adsorbent Dosage on Cu(II)

Cu(II)			
Adsorbent Dosage, g	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
0.1	250	30.7	87.72
0.2	250	29.8	88.08
0.3	250	24	90.4
0.4	250	2.4	99.04
0.5	250	1.9	99.24

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

Zn(II)			
Adsorbent Dosage, g	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
0.1	250	47.3	81.08
0.2	250	45.6	81.76
0.3	250	42.6	82.96
0.4	250	40.8	83.68
0.5	250	40.6	83.76

Table E.6 Effect of Adsorbent Dosage on Fe(II)

Fe(II)			
Adsorbent Dosage, g	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
0.1	250	25.6	89.76
0.2	250	20.3	91.88
0.3	250	18.1	92.76
0.4	250	15.7	93.72
0.5	250	14.3	94.28

Table E.7 Effect of Solution pH on Cu(II)

Cu(II)			
Solution pH, pH	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
2	250	4.9	98.04
4	250	1.2	99.52
6	250	49.6	80.16
8	250	55.5	77.8
10	250	160.3	35.88

Table E.8 Effect of Solution pH on Zn(II)

Zn(II)			
Solution pH, pH	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
2	250	55	78
4	250	29.9	88.04
6	250	85.2	65.92
8	250	134.7	46.12
10	250	187.3	25.08

Table E.9 Effect of Solution pH on Fe(II)

Fe(II)			
Solution pH, pH	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
2	250	27.6	88.96
4	250	1.6	99.36
6	250	45.1	81.96
8	250	87.6	64.96
10	250	132.2	47.12

Table E.10 Effect of Initial Concentration on Cu(II)

Cu(II)		
Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
50	12.31	75.38
100	33.4	66.6
150	69.73	53.51333333
200	102.7	48.65
250	143.4	42.64

Table E.11 Effect of Initial Concentration on Zn(II)

Zn(II)		
Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
50	17.31	65.38
100	43.4	56.6
150	89.73	40.18
200	132.5	33.75
250	163.8	34.48

Table E.12 Effect of Initial Concentration on Fe(II)

Fe(II)		
Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
50	13.44	73.12
100	35.68	64.32
150	79.99	46.67333333
200	112.4	43.8
250	156.4	37.44