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

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A report submitted in partial fulfillment of the

requirements for the award of the degree of

Bachelor of Chemical Engineering

Faculty of Chemical Engineering & Natural Resources

University Malaysia Pahang

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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 0.961and 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.

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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 penggoncang 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 penggoncang 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 penggoncang 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 ABBREVATIONS

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.

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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

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(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 (Acemioglu and Alma, 2001), corn cobs (Hawrhorne-Costa et al., 1995) , apple wastes (Maranon and Sastre, 1991), wool fibers (Balkose and Baltacioglu, 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 Champangne, 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 aloe. 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).

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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.

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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.

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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.

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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,

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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.

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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 [ΔH = 31.47 kJ/mol for Cu (II) and ΔH = 20.07 kJ/mol for Pb (II)] implied that the adsorption was endothermic in nature.

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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.

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 μm. Bulk density and heating value of RHA were found to be 104.9 kg/m^3 and 9.68 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 ≥400 °C. In the paper mills, rice husk is heated at temperatures ≥700 °C to generate steam. Therefore, it is expected that the amount of carbon will be very small in rice husk ash.

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CHAPTER 3

RESEARCH METHODOLOGY

3.1 MATERIAL

i. Rice Husk
ii. Ferrous (II) Sulphate $FeSO_4$
iii. Zinc (II) Sulphate $ZnSO_4$
iv. Cadmium (II) Sulphate $CdSO_4$
v. 0.1 M NaOH
vi. 0.1 M HCl
vii. Distilled Water

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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 1000µL
x. Atomic Adsorption Spectrophotometer (AAS)
xi. Orbital shaker
xii. Oven
xiii. Grinder
xiv. pH Meter
xv. Syringe filter

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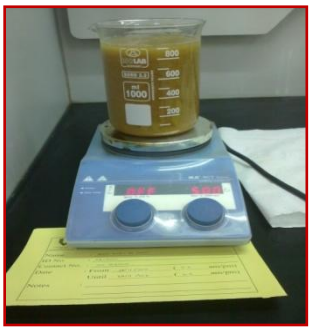
3.3 FLOWCHART

Paddy Tree

Rice Husk

Rice husk were dried at 60°C for 24 hours.



Rice husk were treated with 0.6M acid citric.

Rice husk were filtered and dried at 60°C for 2 hours.

Rice husk were grinded to get the smaller size.

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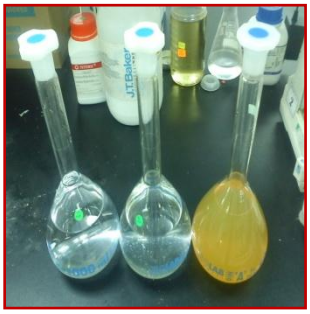
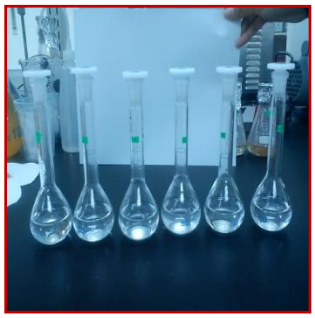
Rice husk were sieved into 1.0 mm

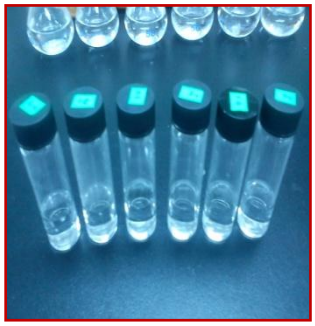


Ferum (II) sulphate, Zinc (II) sulphate and Cadmium (II) sulphate solution



Calibration Curve of Ferum (II), Zinc (II) and Cadmium (II) solution

Working solution of Ferum (II), Zinc (II) and Cadmium (II)



Samples of Ferum (II), Zinc (II) and Cadmium (II) solution

The percent removal was analyzed using AAS

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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.

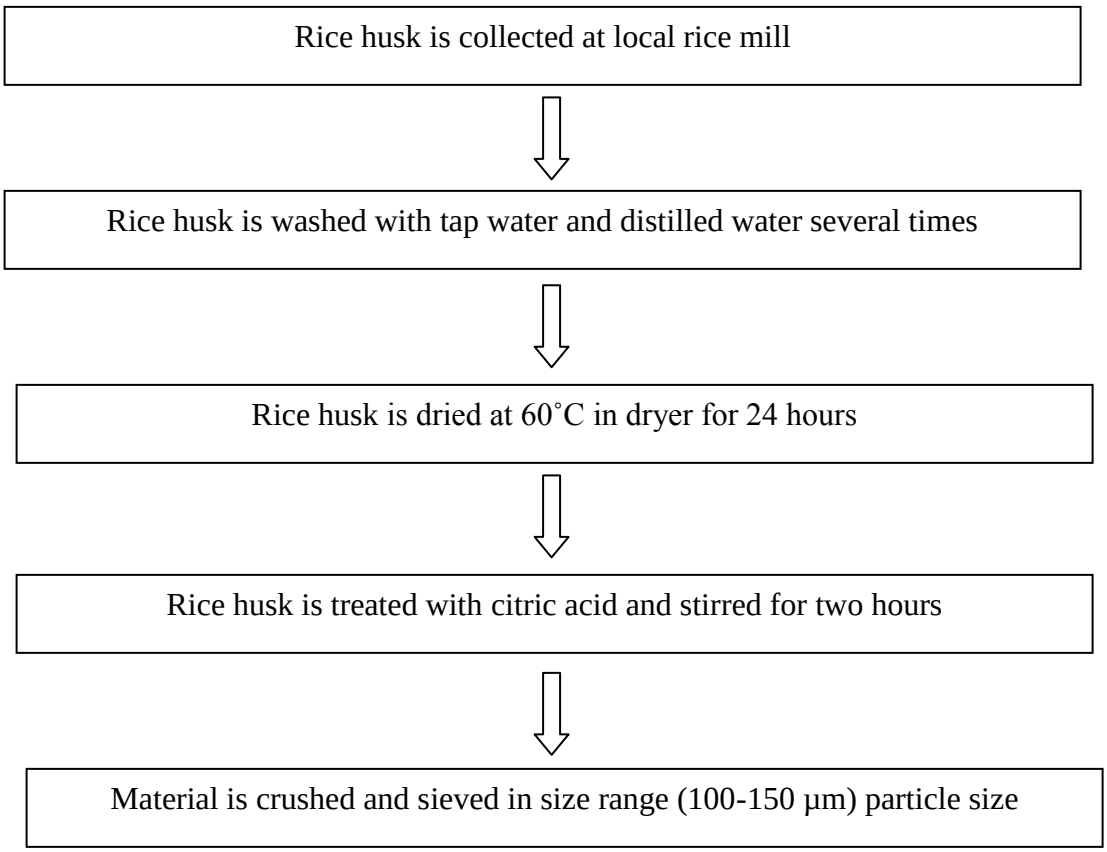

Figure 3.4.1 Flow Diagram for Preparation of Adsorbent

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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 FeSO4. xH_2O, $ZnSO_4$. H_2O and $CdSO_4.xH_2O$ 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 shaked using orbital shaker at constant speed of 150 rpm at 25˚C for different variation of

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