ADSORPTION OF Pb(II), Zn(II) AND Fe(II) USING RUBBER SEED SHELL

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ADSORPTION OF Pb(II), Zn(II) AND Fe(II) USING RUBBER SEED SHELL

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Thesis submitted in fulfilment of the requirements
for the award of the degree
in Bachelor of Chemical Engineering

Faculty of Chemical and Natural Resources
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Special dedication to my mum and dad that always inspire, love and stand beside me, and to my beloved friend.

Thank you for all your love, care and support.

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ABSTRACT

Adsorption of heavy metals by rubber seed shell, an alternative use of waste, chosen to investigate the their effectiveness of using rubber seed shell to remove heavy metal ions from aqueous solutions for the adsorbent's economical feasibility and availability in large quantity. Rubber seed shell a waste and an inexpensive material, were investigated as an adsorbent for the removal of Pb(II), Zn(II) and Fe(II) ions from aqueous solutions using batch techniques. The purpose of this research is to investigate the potential of rubber seed shell as natural low cost adsorbent to adsorb Pb(II), Zn(II) and Fe(II) heavy metal ions by the variation of solution pH, contact time and initial concentration. The adsorbent was prepared by collecting the rubber seed shell, dried and ground it using grinder and mechanical sieve to specify the adsorbent to certain size. Adsorbate were prepared by diluting 100 mg/L of stock heavy metal solutions then used for testing the effect of removal of the heavy metal ions. 0.3g of rubber seed shell as adsorbent with 100 ml adsorbate was kept constant for this experiment where the adsorbate solution varied in pH= 2 to pH=10, using concentrated 0.1M NaOH and 0.1M HCl to change pH and put in a stackable incubator shaker, operated at 150rpm for 120minutes. For variation in contact time, the solution was set on the shaker for different sets of time from 40 minute until 120 minutes at constant 150rpm and 25˚C, with adsorbate concentration of 100mg/L. Initial adsorbate solution concentration of 100 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 isotherms were described by both Langmuir and Freundlich isotherms. Freundlich equation was found to represent the equilibrium data for adsorption of Pb(II), Zn(II), and Fe(II) using rubber seed shell ($0.9391 < R^2 < 0.9857$), and the maximum adsorption capacity was highest for Pb(II) with 14.41mg/g.Adsorption process using rubber seed shell was found to be suitable at pH2. Adsorption kinetic were conducted where both pseudo-first order and pseudo-second order yield values of R^2 from 0.7081 to 0.9768 and from 0.9982 to 1.0 for each order.

ABSTRAK

Penjerapan logam berat oleh kulit biji getah, merupakan penggunaan alternatif bahan buangan, dipilih untuk menyiasat dan mengetahui keberkesanannya untuk mengeluarkan ion logam berat daripada larutan yang dikaji. Dalam kajian ini, kulit biji getah merupakan bahan buangan dan bahan murah, yang sedang disiasat sebagai adsorben untuk proses penyingkiran Pb (II), Zn (II) dan Fe (II) ion daripada larutan akueus. Tujuan kajian ini adalah untuk menyiasat potensi kulit biji getah sebagai adsorben semula jadi yang merupakan kos rendah untuk menjerap Pb (II), Zn (II) dan Fe (II) ion-ion logam berat semula jadi menggunakan variasi larutan pH, masa dan kepekatan awal. Adsorben disediakan dengan mengumpul kulit biji getah, dikeringkan dan dikisar kepada saiz tertentu. Adsorbate disediakan dengan mencairkan 100 mg / L logam berat yang akan digunakan untuk menguji kesan penyingkiran ion-ion logam berat. 0.3g kulit biji getah sebagai adsorben dan 100 ml adsorbate adalah malar bagi eksperimen ini di mana bagi larutan pH berbeza dalam kumpulan pH; 2-10, menggunakan kepekatan 0.1M NaOH dan HCl 0.1M untuk menukar nilai pH dan dimasukkan ke dalam penggoncang orbit, yang beroperasi pada 150rpm untuk 120minit. Bagi perubahan dalam masa, larutan digoncang bermula dari 40 minit dari jam pertama sehingga 120 minit pada 150rpm malar dan 25˚C, dengan kepekatan larutan 100mg / L. Larutan dengan kepekatan awal 100 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. Penjerapan bagi Pb (II), Zn (II) dan Fe (II) akan diterangkan oleh kedua-dua *Langmuir* dan *Freundlich* model. Persamaan *Freundlich* telah dijumpai untuk mewakili yang data keseimbangan untuk penjerapan Pb (II) Zn (II), dan Fe (II) menggunakan kulit biji getah (0,9391 $< R^2 <$ 0,9857), dan yang kapasiti penjerapan maksimum adalah tertinggi bagi Pb (II) dengan 14.41mg / g. Proses penjerapan menggunakan kulit biji getah didapati sesuai pH2. Penyerapan kinetik telah dijalankan di mana kedua-dua pseudo- tertib pertama dan kedua di mana hasil nilai R^2 dalam lingkungan 0.7081 to 0.9768 dan 0.9982 to 1.0 bagi setiap tertib.

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LIST OF ABBREVATIONS

AAS	Atomic Adsorption Spectrometer
Pb(II)	Lead (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 lead. 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. Lead is mostly used in building construction and lead-acid batteries production but exposure to lead through inhalation of fine dust and fumes or soluble lead compounds could lead to damages the nervous system and causes blood and brain disorder. A major application of zinc metal is most commonly used as an anti-corrosion agent. Although zinc is an essential requirement for good health, excess zinc can be harmful. The free zinc ion in solution is highly toxic to environments such as for plants, invertebrates, and even vertebrate fish. 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 fight 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).

Rubber seed shell 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 agriculture waste has been assessed for the removal of metal ions such as nickel, chromium, calcium 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 is to assess the feasibility of utilizing rubber seed shell for the adsorption of heavy metals in aqueous solution by the variation of the given parameters.

1.2 PROBLEM STATEMENT

The increasing use of metal and chemicals in Malaysia industries 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 rubber seed shell (RSS) that unused waste that could hopefully help to reduce pollution.

Despite, Malaysia being a major rubber growing country, Malaysia is the leading producer of natural rubber in the world. About 46% of the total world's rubber is produced in Malaysia. The pip of rubber-seed is sent to oil-mills, but a huge amount of rubber-seed shells as agricultural waste which has become an environment problem; environment contamination problems in rubber tree plantations. Rubber seed shell can found at any rubber tree plantation areas. Besides that, this alternative that we use for removal heavy metal, rubber seed shell can take the part that is one of the valuable biomass wastes. As the low cost, rubber seed shell is an attractive and inexpensive alternative for the adsorption removal of dissolved metals. From rubber seed shell which ones was a waste, we could conventionalize it to become wealth.

1.3 OBJECTIVE

- To investigate the potential of rubber seed shell as natural adsorbent to adsorb Pb (II), Zn (II) and Fe (II) ions by the variation of solution pH, contact time, and adsorbent doses.

- To find the adsorption kinetics of Pb (II), Zn (II) and Fe(II) from aqueous solution

1.4 SCOPE

1.4.1 Analyzation of the potential of rubber seed shell as a low cost natural adsorbent to adsorb heavy metal ions from aqueous solution.

1.4.2 Observation and investigation of the effect of process condition for Lead (II), Zinc (II) and Iron (II) that can be removed by using rubber seed shell.

1.4.3 Determination of the percentage removal efficiency of heavy-metal ions by analyzing the result of initial and final concentration for each variable using Atomic Adsorption Spectrometer:

1.4.3.1: Solution pH (pH 2, pH 4, pH 6, pH 8, pH 10)

1.4.3.2: Contact time (40min, 60min, 80min, 100min, and 120min)

1.4.3.3: Initial Concentration (20ppm, 40ppm, 60ppm, 80ppm, 100ppm)

1.4.4 Determination of Langmuir and Freundlich adsorption isotherm for Lead (II), Zinc (II) and Iron (II) that can be removed by using rubber seed shell.

1.4.5 Determination the Pseudo-first order and Pseudo-second order adsorption kinetic for Pb(II), Zn(II) and Fe(II) using rubber seed shell.

1.5 RATIONALE AND SIGNIFICANCE

In this experiment, heavy metal ions, Pb (II) Fe (II) and Zn (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, orange peel and sawdust are low cost waste residues and easily available in large quantities in Malaysia.

The utilization of natural and agriculture waste 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. Rubber seed shell is chosen in this research because it is selective towards metal ion adsorption, effective, economically feasible because rubber seed shell can be easily found as waste product at rubber production, where orange peel can found at local food industries and local plantation field that have benefit for the environment. Hence, from rubber seed shell which ones was a waste, we could conventionalize it to become wealth.

CHAPTER 2

LITERATURE REVIEW

Okieiman et al. (2005) had studied the adsorption of heavy metal and organic compound from aqueous solution by rubber seed shell. The effect of pH determination, bulk density determination, adsorption of zinc ion and organics has been studied. This studies are used the values of partition coefficient of Zn (II) ions between the active carbon and continuous aqueous phase are in the range 0.786 – 0.190 L/g for the range of initial metal ion concentrations (0.5 – 2.5 $mmolL^{-1}$). The adsorption data of metal ions at temperatures of 30, 40, 50 and 60°C have been described by the Freundlich and Langmuir isotherm model. From the results, it can concluded that powdered activated carbon from using rubber seed shell and ammonium chloride as chemical activator has been used in removing Zn (II) ions from aqueous solutions. And that show that shell carbon is an effective remover of metal ions and organic compounds.

Oladoja et al. (2008) had studies the process of adsorption using 5 different isotherm models (Langmuir, Freundlich, Temkin, Harkins–Jura, and Halsey isotherm equations). The highest values of r2 were obtained when the experimental data were fitted into Freundlich and Halsey isotherm equations (0.999). Analysis of the data obtained from the different sorption studies revealed that the data fitted better to the pseudo-second order model than any other kinetic model, indicating that the sorption process will include chemisorptions on rubber seed shell.

Jamal et. al. (2011) explained the kinetic and equilibrium study of adsorption of some dyes onto feldspar by the effect of dye concentration, pH, and 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, with endothermic process.

Li et al. (2007) investigated the removal of Cd, Zn, Co, and Ni ions from aqueous solutions by adsorption on an orange peel. Batch kinetics and isotherm studies were carried out under varying the solution of pH, contact time and adsorbent dosage. Adsorption isotherms of Pb (II), Ni (II), Zn (II), Cu (II) and Co (II) ions on adsorbents were determined and correlated with common isotherm equations such as Langmuir and Freundlich isotherms models. The Langmuir and Freundlich models were used to determine adsorption isotherm data. The thermodynamic parameters like equilibrium constant in Langmuir model, maximum theoretical metal uptake capacity (mol/kg) and values of correlation coefficient, R^2 for the adsorption of Co (II), Ni (II), Zn (II), and Cd (II) ions have also been computed and discussed.

Ajmal et al. (2000) had studied and gave that the process is endothermic showing monolayer adsorption of Ni (II), with a maximum adsorption of 96% at 50^{o}C for an initial concentration of 50mgl^{-1} at pH 6. Kinetic and thermodynamic behaviour of rubber seed shell for Pb (II), Zinc (II) and Iron (II) ions removal was also studied. Thermodynamic parameters such as standard free energy change, enthalpy change and entropy change were also calculated and computed. The process is endothermic and follows Langmuir and Freundlich isotherm. Respectively, desorption was possible with 0.05M HCL and was found to be 95.83% in column and 76% in batch process.

Mohammad et.al. (2011) explained 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. Rubber Seed Shell (RSS)

ii. Lead (II) Sulfate

iii. Zinc (II) Sulphate

iv. Ferrous (II) Sulphate

v. 0.1N NaOH

vi. 0.1N HCl

3.2 Apparatus

i. 1L and 100ml Measuring cylinder
ii. 1L Volumetric Flask
iii. 250ml Beaker
iv. 250ml Volumetric flask
v. 100ml Conical flask
vi. Glass rod
vii. Dropper
viii. Funnel
ix. Whatman Filter Paper 125mm
x. Aluminium foil
xi. pH meter
xii. Oven
xiii. Electronic balance
xiv. Atomic Adsorption Spectrometer
xv. Sieve
xvi. Oven
xvii. Tyler Mesh (to label samples)

3.3 Experimental Procedures

3.3.1: Rubber Seed Shell

The rubber seed shell was washed with deionised water, to remove any attached dirt and soluble impurities. Then, the washed rubber seed shells were dried in the open air and then pulverize. The powder was sieved using a laboratory sieve and the 160 μm fraction was separated. This fraction was washed several times with deionised water until the washings were free of colour and turbidity. The powder was later dried in the oven at 80 ˚C and kept in a sealed polythene bag pending usage as an adsorbent.

Rubber seed shell is collected at rubber tree plantation

⇓

Rubber seed shell washed with deionised water for several times to remove dust

⇓

Rubber seed shell is dried at open air then grind

⇓

The powder was sieved using a laboratory sieve and the 100 μm fraction was separated.

⇩

This portion was washed several times with deionised water until the washings were free of colour and turbidity

⇩

The powder was later dried in the oven at 80 ˚C and then kept in a sealed polythene bag pending usage as an adsorbent.

Figure 3.1: Flow diagram of preparation of rubber seed shell (RSS)

3.3.2: Preparation of Adsorbate

Solution will be prepared by dissolving 100 mg of Lead (II) Sulfate, Zinc (II) Sulphate and Ferrous (II) Sulphate in 1L of distilled water in different volumetric flask.

100 mg of Lead (II) Sulfate, Zinc (II) Sulfate and Ferrous (II) Sulphate is dissolved in 1L of distilled water in different volumetric flask (stock solution 100mg/L)

⇩

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

Figure 3.2: Flow diagram of preparation of adsorbate

3.3.3: pH Solution

0.3 g of rubber seed shell as adsorbent with 100 ml adsorbate was kept constant for this experiment. Use concentrated 0.1N NaOH and 0.1N HCl to change pH from pH=2 to pH=10 so that the change in volume of the solution is negligible. Adsorbate solutions were performed at 25˚C at the variable pH which are pH=2, pH= 4, pH=6, pH=8, pH=10, on a rotary shaker operated at 150rpm for 2 hours. Then, sample are filtered using Whatman Filter Paper 125mm to be analyzed using Atomic Adsorption Spectrometry.

The conical flask containing 0.3g of rubber seed shell and 100 ml adsorbate concentration is varied in the pH range 2-10 were adjusted using concentrated NaOH and HCl

⇩

The conical flasks is shaked on rotary shaker at 150rpm for 2 hours

⇩

The samples are analyzed using Atomic Adsorption Spectrometry

Figure 3.3: Flow diagram of pH solution

3.3.4: Contact Time

In this experiment, the adsorbate solution concentration is 100mg/L. The effect of contact time investigated for 40min, 60min, 80min, 100min, and 120min at the optimum pH= 2, 0.3g/100ml sample of adsorbent dosage and shaked on a rotary shaker operated at 150 rpm and 25˚C. Then, sample are filtered using Whatman Filter Paper 125mm to be analyzed using Atomic Adsorption Spectrometry.

The conical flasks is filled with 100 mg/L adsorbate solution

⇩

The conical flask is shaked in incubator for 40min, 60min, 80min, 100min, and 120min at the pH=2 and 0.3 g sample adsorbent dosage on a rotary shaker operated at 150 rpm at 25˚C

⇩

The samples are analyzed using Atomic Adsorption Spectrometry

Figure 3.4: Flow diagram of contact time

3.3.5: Adsorbent Dosage

Initial adsorbate solution concentration of 100 mg/L was used with adsorbent sample varying in weight, 0.1grams, 0.2grams, 0.3grams, 0.4grams, 0.5grams. The contact time and pH kept constant at 120min and pH=2 that can be obtained from previous experiment before conducting the effect of rubber seed shell dosage experiment at 25˚C. . Samples are filtered using Whatman filter paper and analyzed using AAS.

The conical flask is filled with 100 mg/L of adsorbate solution

The flask is shaked in incubator with rubber seed shell sample of (0.1g -0.5g)

The samples are analyzed using Atomic Adsorption Spectrometry

Figure 3.5: Flow diagram of adsorbent dosage

3.3.6: Initial Concentration

In this experiment, the initial adsorbate solution concentration was varied from 20mg/L, 40mg/L, 60mg/L, 80mg/L and 100mg/L. The effect of initial concentration is analyzed using constant solution pH of pH=2, 0.3g of adsorbent dosage and sample contact time of 120min and shake sample on an incubator shaker operated at 150rpm and 25^0C.

The conical flask is filled with 100 mg/L of adsorbate solution

⇓

The flask is shake in incubator shaker at constant 0.3g sample adsorbent, pH2 for 120min and operated at 150rpm, 25^0C

⇓

The samples are analyzed using Atomic Adsorption Spectrometry

Figure 3.6: Flow diagram of initial concentration

Experiment on the four variables, effect of pH solution, contact time, adsorbent dosage and initial concentration was done for adsorbate, Lead (II) Sulfate, Zinc (II) Sulfate and Ferrous (II) Sulfate solution.

The rubber seed shell is prepared

The adsorbate is prepared

The adsorbent and adsorbate is tested for effect of different solution pH

The adsorbent and adsorbate is tested for effect of different contact time

The adsorbent and adsorbate is tested for effect of different adsorbent dosage

The adsorbent and adsorbate is tested for effect of different initial concentration

The result is tabulated and compared to obtain the suitable pH solution, contact time and adsorbent dosage for maximum adsorption of Lead (II), Zinc (II) and Fe (II) using rubber seed shell

Figure 3.7: Flow diagram of adsorption of Pb (II), Zn (II) and Fe (II) using rubber seed shell 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.

$$\boxed{M_1V_1 = M_2V_2}$$

Samples that are filtered will be diluted to 10mg/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 are obtained.

CHAPTER 4

RESULT & DISCUSSION

4.1 Effect of Solution pH

The pH of the solution has a major impact on the uptake of heavy metal, since it determines the surface charge of the adsorbent which means the degree of ionization and speciation of the adsorbate (El-Ashtoukhy, 2008). In order to establish the effect of pH was studied at different pH values which were conducted by varying the optimum pH range from pH=2 to pH=10 for heavy metals. The graph heavy metal removal efficiency, % versus solution pH is plotted by using formula below

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

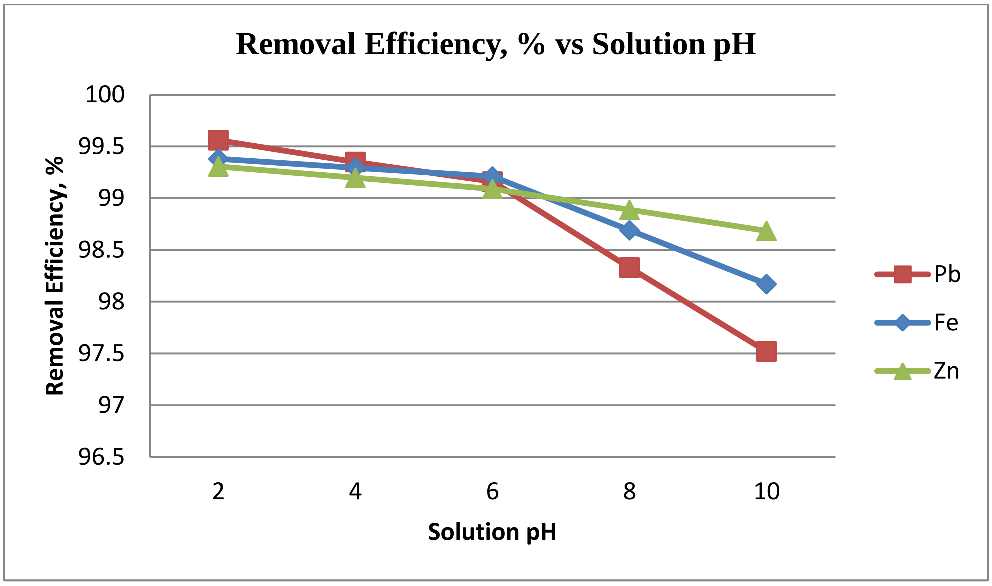

Figure 4.1: Effect of pH on Pb (II), Zn(II), Fe(II).

Initial concentration = 100 mg/L, Adsorbent dosage = 0.3g/L,

Contact time = 2hrs

From figure 4.1 shows that the effect of solution pH on the adsorption of heavy metal and the maximum removal of heavy metal were observed at pH= 2. The decrease in adsorption at high pH values may be due to the competitiveness of hydrogen and heavy metal ions on the sorption sites. Referring to Low et.al, 1993 could be attributed to the hydrogen ions competing with metal ions for sorption 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 Adsorbent Dosage

One of the parameters that strongly affect the sorption capacity is the concentration of the adsorbents. With the fixed metal initial concentration, it can easily be inferred that the percent removal of metal ions increases with increasing weight of adsorbents as shown from figure below. Amount of adsorbent was varied from 0.1g – 0.5g with fixed initial concentration, pH=2 and contact time which is 100mg/L and 120 minutes. The results indicated the percent removal of metal increased with the increase in the amount of adsorbent and removal efficiency. The graph heavy metal removal efficiency, % versus adsorbent dosage is plotted by using formula below

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

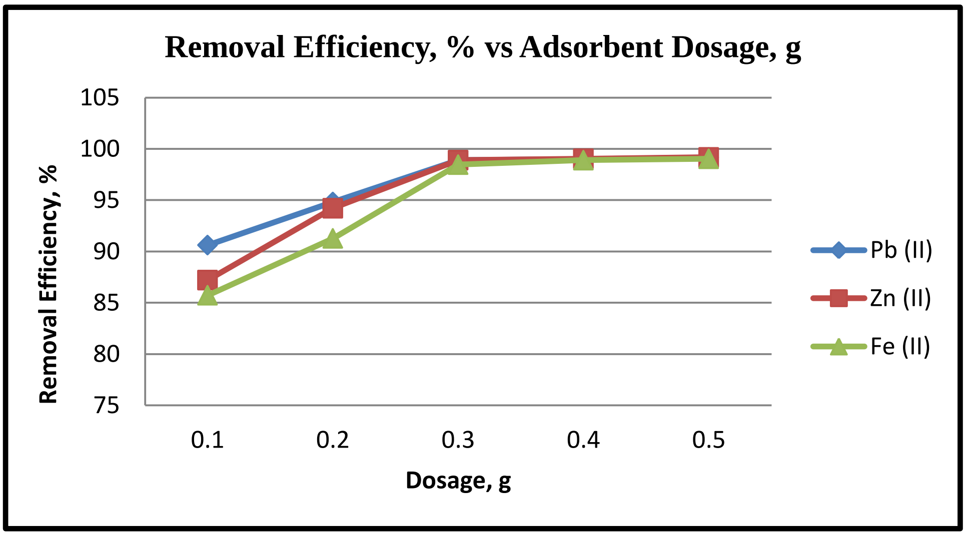

Figure 4.2: Effect of adsorbent dosage on Pb (II), Zn(II), Fe(II).

Initial concentration = 100 mg/L, pH = 2, Contact time = 2hrs

From figure above, it shown that increasing of adsorbent dosage improve the removal metal until the equilibrium was reached at 0.3g for every each metal. This happened due to availability of surface area (Aoyama et al., 2003). More quantity of adsorbent result in increasing of surface area which is causes removal of more metal ions. This can be explained by the fact that a fixed mass of adsorbent can only adsorp a certain amount of metal.

4.3 Effect of Contact Time

The equilibrium time required for the adsorption of heavy metals Pb(II), Zn(II) and Fe(II) with 0.3g/L of the rubber seed shell adsorbent and initial concentration 100mg/L at different time intervals were studied. The graph of heavy metal removal efficiency, % versus contact time, minute were plotted by using formula below

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

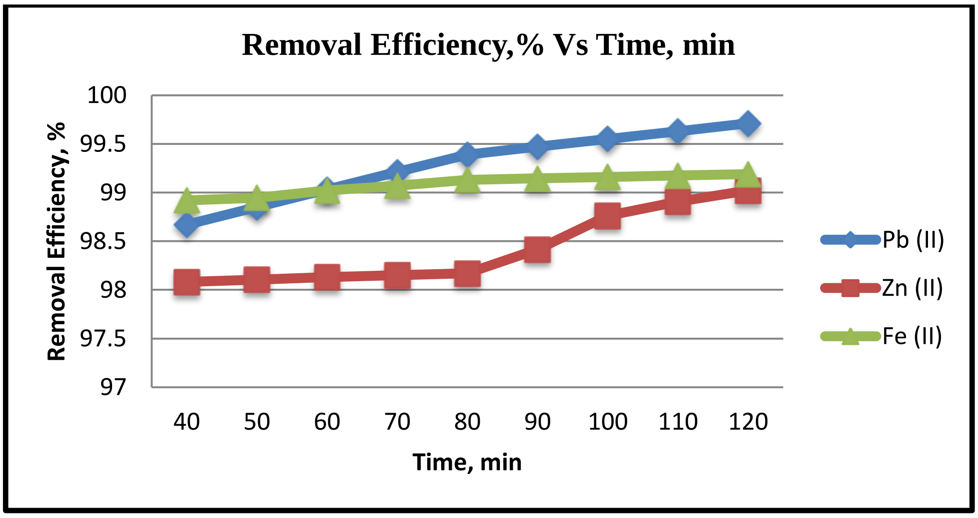

Figure 4.3 Effect of time on Pb (II), Zn(II), Fe(II)

Adsorbent dosage = 0.3 g, pH = 2, Contact time = 2hrs

Figure above represents the percent removal efficiency of Pb(II), Zn(II) and Fe(II) versus the contact time for the initial concentration and by using the optimum pH value which obtained for these heavy metals. Pb(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 of heavy metal ions increases with time and altains saturation on about 120minutes. Basically, the removal of sorbate is rapid, but it gradually decreases with time until it reaches equilibrium. The removal rate of Pb(II), Zn(II) and Fe(II) heavy metal ions increases of the adsorption time. However, it remains constant after an equilibrium time of 120min. The rate of percent metal removal is higher in the beginning due to a larger availability surface area of the adsorbent for the adsorption of the metals.

4.4 Effect of Initial Concentration

The effect of heavy metal concentration in the range of 20 to 100 mg/L on removal efficiency was observed with constant solution pH=2, contact time of 120minutes and 0.3g of adsorbent. The graph of heavy metal removal efficiency, % initial concentration, mg/L were plotted by using formula below

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

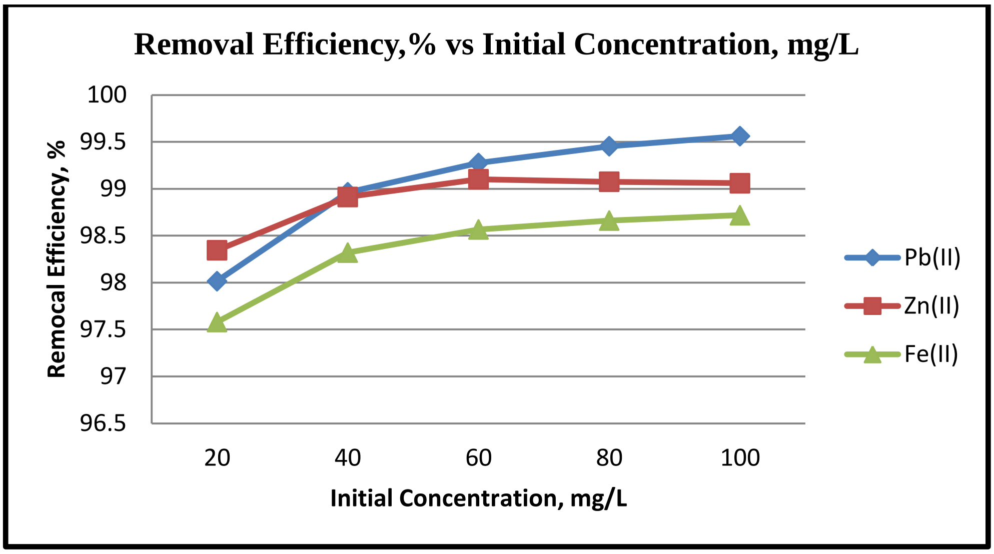

Figure 4.4 Effect of initial concentration on Pb (II), Zn(II), Fe(II).

Adsorbent dosage = 0.3 g, pH = 2, Contact time = 2hrs

From the figure above, the removal efficiency increase with the increasing amount of initial concentration. At higher concentration, the available sites of adsorption process become fewer, and hence the percentage removal of metal ions depends upon the initial concentration. The amount of metal ions adsorbed per unit mass of all the adsorbents increased with the initial metal concentration as expected. Uptake of Pb(II), Zn(II) and Fe(II) heavy metal ions increased with increase in concentration and remained nearly equilibrium after equilibrium time. And from figure above, it also showed that the adsorption was rapid in the initial stages and gradually decreased with adsorption progress.

4.5 Adsorption Equilibrium

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 (Kumar et al., 2009). Isotherm used to calculate maximum heavy metal ions uptake are Langmuir and Freundlich isotherms. Experimental isotherm data were conducted at an equilibrium time of 120 minute, constant adsorbent dosage of 0.3g, and adsorbate solution with pH=2. Interact with adsorbents and is critical in optimizing the use of adsorbents.

4.5.1 The Langmuir isotherm

The Langmuir adsorption isotherm model was used to describe the relationship between the amount of heavy metal adsorbed and its equilibrium concentration in solutions. The Langmuir isotherm is valid for monolayer adsorption on a surface containing a finite number of identical sites. The model assumes uniform adsorption on the surface and no transmigration in the plane of the surface (Hall et al., 1996). The linear form of the Langmuir isotherm can be represented by the following equation,

$$q_e = \frac{q_m\ K_L C_e}{1 + K_L C_e}$$ (Kumar et al., 2009)

Where Ce (mg/L) is the equilibrium concentration of the adsorbate, qe (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, K_L the Langmuir adsorption constant in L/mg.

The constants in the Langmuir isotherm can be determined by plotting (1/qe) versus (1/Ce) and making use of above equation rewritten as (Kumar et al., 2009):

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

Where qm is the maximum adsorption capacity for heavy metal ions uptake, mg/g and K_L the Langmuir adsorption constant in L/mg. The graph $\frac{1}{q_e}$ of plotted against $\frac{1}{C_e}$ yielding the value of ranging from 1.28 mg/g to 9.89 mg/g.

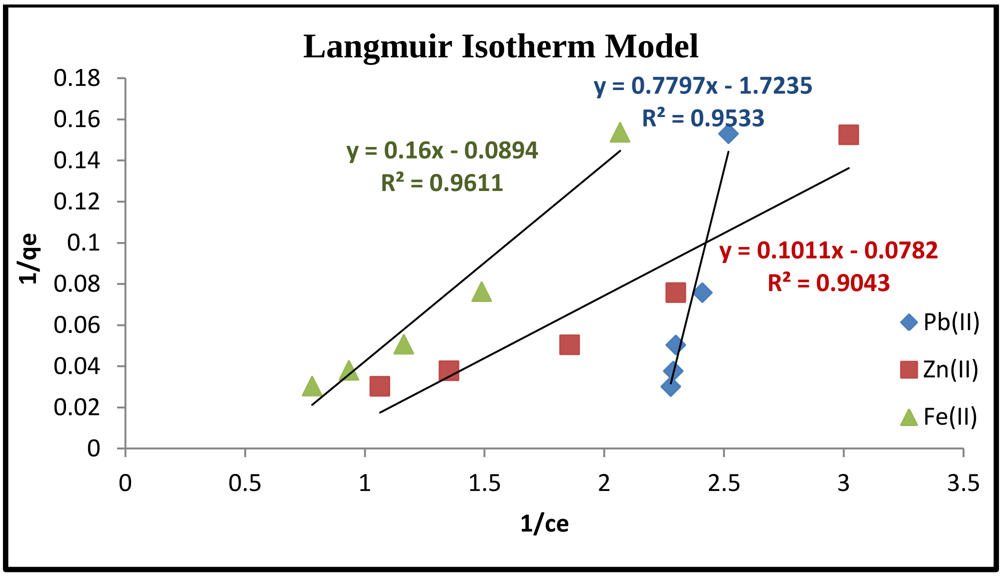

Figure 4.5: Langmuir isotherm of Pb(II), Zn(II) and Fe(II) adsorption using rubber seed shell

4.5.2 The Freundlich isotherm

The freundlich isotherm model is an empirical relationship describing the adsorption of solute from a liquid to a solid surface and assumes that different sites with several adsorption energies are involved. Freundlich adsorption isotherm is the relationship between the amounts of Pb(II), Zn(II) and Fe(II) heavy metals adsorbed per unit mass of adsorbent, qe and the concentration of the nickel at equilibrium, Ce.

The Freundlich isotherm is expressed by (Tangjuank et al., 2009)

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

The logarithmic form given as,

$$logq_e = \log K_f + \frac{1}{n} logC_e$$

K_f and n are the Freundlich adsorption isotherm constants. Where, K_f is known as the indicators of the adsorption capacity and n is the heterogeneity factor representing the deviation from linearity of adsorption and is also known as Freundlich coefficient. The ability of Freundlich model to fit the experimental data was examined. Graph was plotted by log *Ce* vs. log *qe* to construct the intercept value of K_f and the slope of n. From Fig. 4.6 the Freundlich constants K_f values ranging from 0.13 to 0.82 and n were found to be 0.06 to 0.67. The magnitudes of K_f and n show easy separation of Pb(II), Zn(II) and Fe(II) ions from the aqueous solution. The value of K_f interception is an indicating of the adsorption capacity progress of the adsorbent. And value for the slope of 1/n indicates the effect of concentration on the adsorption capacity and represents as adsorption intensity. Freundlich isotherm fitted well with the correlation coefficient of 0.9857.

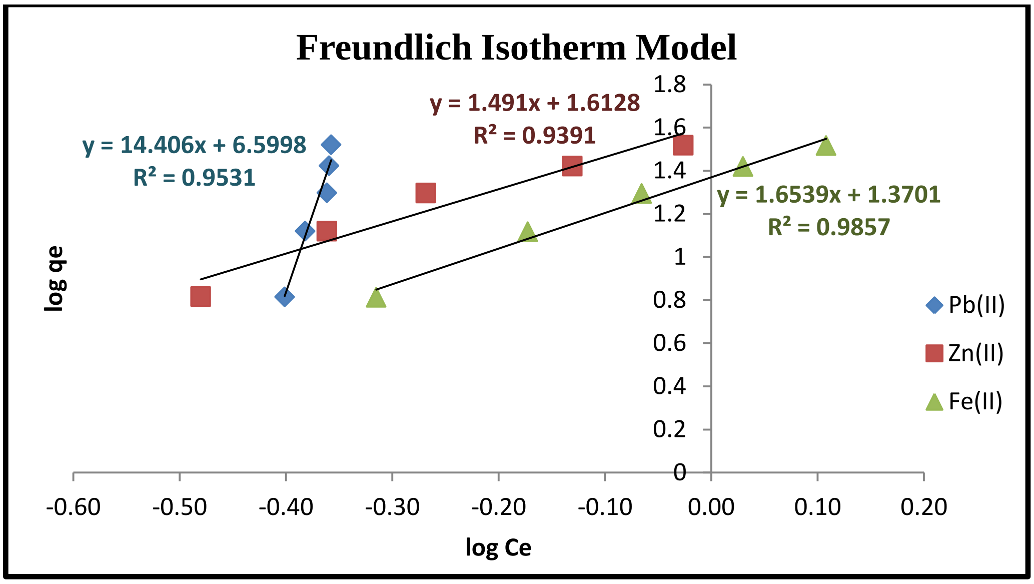

Figure 4.6 Freundlich isotherm of Pb(II), Zn(II) and Fe(II) adsorption using rubber seed shell

From the graph, corresponding isotherm the Langmuir and Freundlich adsorption constats with the correlation coefficients were calculated and presented in Table 4.1

Table 4.1 Langmuir and Freundlich adsorption isotherm model constants

Heavy Metal	Langmuir			Freundlich		
	q_{max}, mg/g	K_L, l/mg	R^2	K_f, (mg/g)(l/mg)^(1/n)	1/n	R^2
Pb(II)	1.2825	2.2105	0.9533	0.8195	14.4060	0.9531
Zn(II)	9.8912	0.7735	0.9043	0.2076	1.4910	0.9391
Fe(II)	6.2500	0.5588	0.9611	0.1368	1.6539	0.9857

4.6 Adsorption Kinetics

Kinetic models have been used in order to investigate the controlling mechanism of adsorption processes such as mass transfer and chemical reaction, the pseudo-first-order and pseudo-second order equations are applied to model the kinetics of Pb(II), Zn(II) and Fe(II) heavy metals adsorption onto rubber seed shell. The pseudo-first-order rate equation is given as (Kumar et al., 2009):

$$\log(q_e - q_t) = \log q_e - \frac{k_{1ad}}{2.303} t$$

Where, *qe* is the amount of heavy metal ion adsorbed at equilibrium (mg/g), q_t is the amount adsorbed at time *t* (mg/g), K_{1ad} is the rate constant of first order adsorption (L/min). 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 Pb (II), Zn (II) and Fe (II) heavy metals ions are observed in the graph below

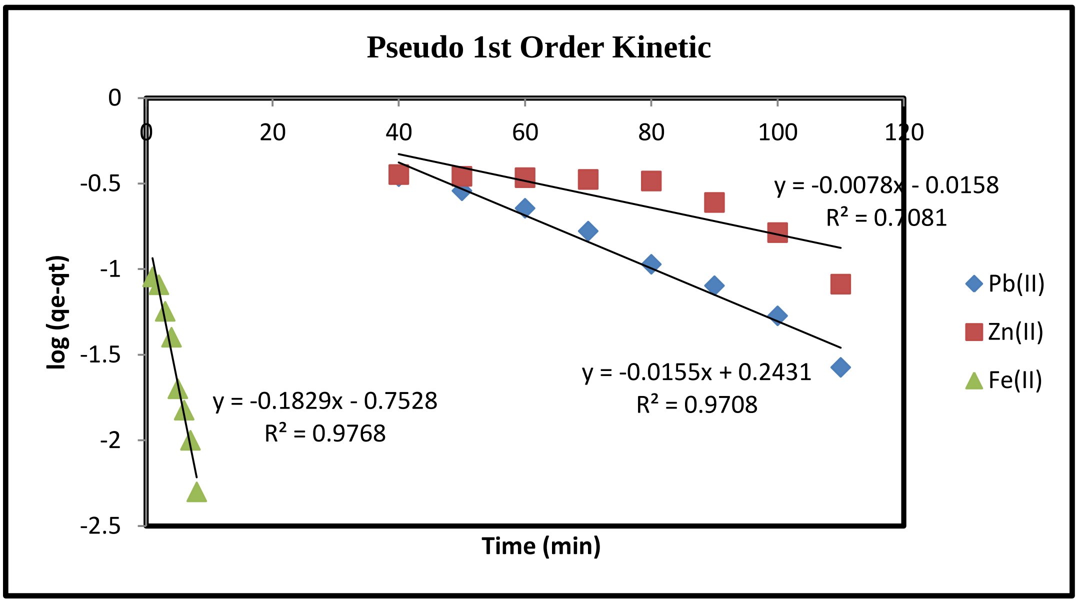

Figure 4.7 Pseudo-first order kinetic for Pb(II), Zn(II) and Fe(II) adsorption using rubber seed shell

The pseudo-second-order equation is expressed as (Kumar et al., 2009);

$$\frac{1}{q_t} = \frac{1}{h} + \frac{1}{q_e}t$$

Where $h = kq_e^2$ (mg $g^{-1}min^{-1}$) expressed as the initial adsorption rate as $t \rightarrow 0$ and k is the rate constant of pseudo-second-order adsorption (g $mg^{-1}min^{-1}$). Graph t/qt versus t were plotted should give a straight line if pseudo-second-order kinetics is applicable (Kumar et al., 2009). And for qe, k and h it can be determined from the slope and intercept of the plot from the graph. The plots of the linearized form of the pseudo-second order adsorption kinetic at different Pb(II), Zn(II) and Fe(II) concentrations using rubber seed shell are shown in Fig. 4.8. The pseudo-first-order pseudo-second-order rate constants and corresponding correlation coefficients determined from Figs. 4.7 and 4.8 are presented in Table 4.2. From the calculated values below, the adsorption process of Pb (II), Zn(II) and Fe(II) heavy metal ions are more favourable towards pseudo-second order kinetics due to higher value of R^2 and in the range of 0 to 1.

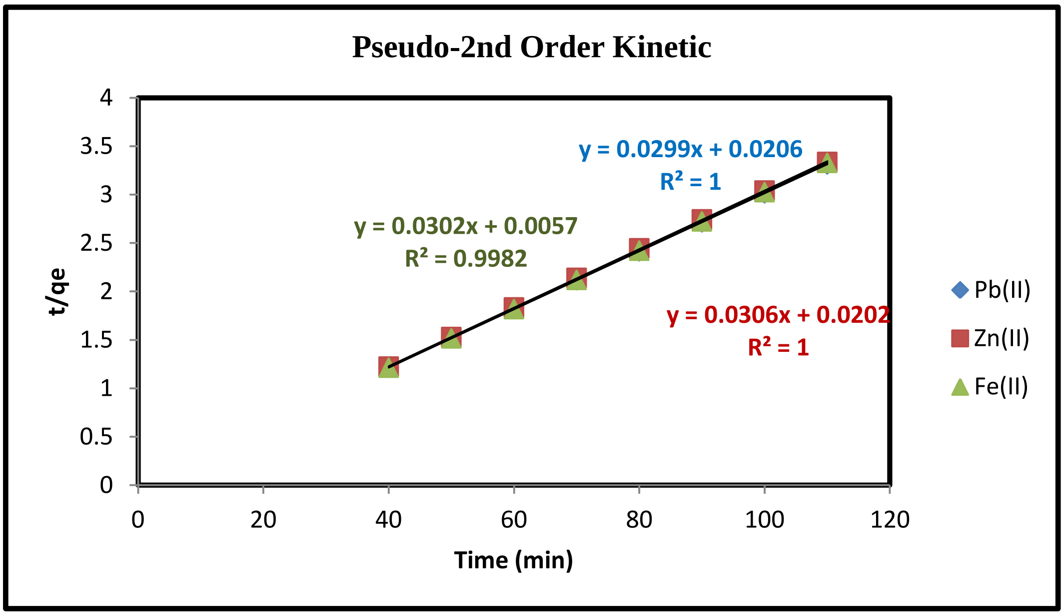

Figure 4.8 Pseudo-second order kinetic for Pb (II), Zn (II) and Fe (II) adsorption using rubber seed shell

Table 4.2 Pseudo-first order and Pseudo-second order kinetic model constant

Heavy metal	Pseudo-First Order Kinetic				Pseudo-Second Order Kinetic		
	q_e exp, mg/g	q_e, mg/g	k_{1ads}, L/min	R^2	q_e, mg/g	k_{2ads}, g/mg.min	R^2
Pb(II)	33.2367	1.7502	0.0357	0.9708	33.4448	0.0434	1.0000
Zn(II)	33.0500	1.0371	0.0180	0.7081	32.6797	0.0455	1.0000
Fe(II)	33.0633	5.6598	0.4212	0.9768	33.1126	0.0443	0.9982

CHAPTER 5

CONCLUSION

5.1 Conclusion

From the result we can conclude rubber seed shell has been successfully approved to act as an adsorbent for adsorption of Pb(II), Zn(II) and Fe(II) ions from aqueous solution. The adsorption process has been affected by the variation of solution pH, contact time, and adsorbent dosage. The highest adsorption was found by the time of 120 minutes, pH of 2 and adsorbent dosage of 0.3g. Adsorption of Pb(II), Zn(II) and Fe(II) ions from aqueous solution approved the second-order kinetic yielding good R2 values from 0.99 to 1.00 and k values of 0.0434 - 0.0455.

5.2 Recommendation

Some recommendations have been made to improve the result are given for future work.

- It will be effective if there are more experiment on various adsorbent could be done continuously.
- Try to add another economically adsorbents

CHAPTER 6

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APPENDIX A

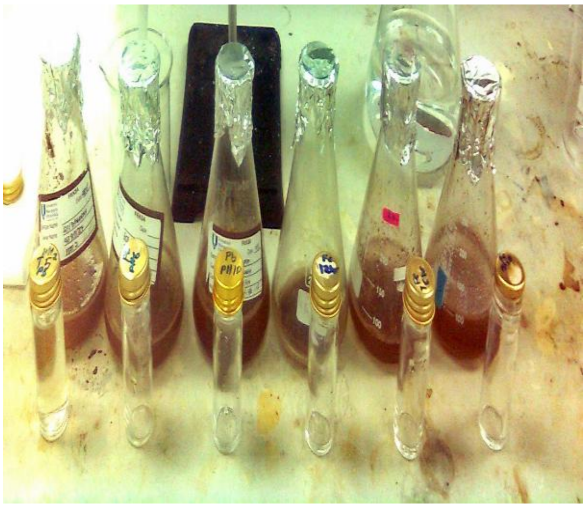
Rubber Seed Shell

Drying of Rubber Seed Shell

Rubber Seed Shell Preparation

Lead (II) Sulfate, Zinc(II) Sulphate and Ferum(II) Sulphate

Adsorbate preparation



Preparation samples based on parameter tested

Solution filtered using Whatman filter paper

Sample solution after react with reagent

APPENDIX B: RESULT DATA

Table B1: Effect of Solution pH on Pb(II)

Pb(II)			
Solution pH, pH	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
2	100	0.64	99.36
4	100	0.55	99.45
6	100	0.84	99.16
8	100	1.67	98.33
10	100	1.98	98.02

Table B2: Effect of Solution pH on Zn(II)

Zn(II)			
Solution pH, pH	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
2	100	0.793	99.207
4	100	0.802	99.198
6	100	0.91	99.09
8	100	1.063	98.937
10	100	1.116	98.884

Table B3: Effect of Solution pH on Fe(II)

Fe(II)			
Solution pH, pH	Initial Concentration, mg/L	Final Concentration, mg/L	Removal Efficiency, %
2	100	0.62	99.38
4	100	0.706	99.294
6	100	0.99	99.01
8	100	1.21	98.79
10	100	1.53	98.47

Table B4: Effect of Adsorbent Dosage on Pb(II)

Pb(II)

Adsorbent Dosage, g	Initial Concentration, (mg/L)	Final Concentration, (mg/L)	Removal Efficiency,%
0.1	100	9.37	90.63
0.2	100	5.174	94.826
0.3	100	1.097	98.903
0.4	100	0.994	99.006
0.5	100	0.835	99.165

Table B5: Effect of Adsorbent Dosage on Zn(II)

Zn(II)

Adsorbent Dosage, g	Initial Concentration, (mg/L)	Final Concentration, (mg/L)	Removal Efficiency, %
0.1	100	12.784	87.216
0.2	100	5.782	94.218
0.3	100	1.097	98.903
0.4	100	0.962	99.038
0.5	100	0.827	99.173

Table B6: Effect of Adsorbent Dosage on Fe(II)

Fe(II)

Adsorbent Dosage, g	Initial Concentration, (mg/L)	Final Concentration, (mg/L)	Removal Efficiency, %
0.1	100	14.26	85.74
0.2	100	8.717	91.283
0.3	100	1.527	98.473
0.4	100	1.094	98.906
0.5	100	0.959	99.041

Table B7: Effect of Contact Time on Pb(II)

Pb(II)			
Contact Time, (min)	Initial Concentration, mg/L	Final Concentration, (mg/L)	Removal Efficiency, %
40	100	1.33	98.67
50	100	1.15	98.85
60	100	0.97	99.03
70	100	0.79	99.21
80	100	0.61	99.39
90	100	0.53	99.47
100	100	0.45	99.55
110	100	0.37	99.63
120	100	0.29	99.71

Table B8: Effect of Contact Time on Zn(II)

Zn(II)			
Contact Time, min	Initial Concentration, (mg/L)	Final Concentration, (mg/L)	Removal Efficiency, %
40	100	1.92	98.08
50	100	1.895	98.105
60	100	1.87	98.13
70	100	1.85	98.15
80	100	1.83	98.17
90	100	1.585	98.415
100	100	1.24	98.76
110	100	1.095	98.905
120	100	0.98	99.02

Table B9: Effect of Contact Time on Fe(II)

Fe(II)			
Contact Time, min	Initial Concentration, mg/L	Final Concentration, (mg/L)	Removal Efficiency, %
40	100	1.08	98.92
50	100	1.053	98.947
60	100	0.98	99.02
70	100	0.93	99.07
80	100	0.87	99.13
90	100	0.855	99.145
100	100	0.84	99.16
110	100	0.825	99.175
120	100	0.81	99.19