ADSORPTION OF LEAD USING ACTIVATED CARBON PREPARED FROM COCONUT COIR

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CHEMICAL ENGINEERING
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Nurulsyahidah binti Abu Bakar

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
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CERTIFICATION OF APPROVAL

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TRONOH, PERAK

July 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NURULSYAHIDAH BINTI ABU BAKAR

ABSTRACT

The main objective of the project is to study on the feasibility of the activated carbon prepared from coconuts coir for lead removal. The removal of lead (Pb) using coconut coir as a low-cost alternative natural bio-adsorbent from aqueous solutions is investigated. For this project, both chemical and physical activation methods are used to prepare and activate the activated carbon prepared from coconut coir. Then the activated carbon is characterized using FTIR, SEM, TGA and XRD. Batch adsorption kinetic studies show the adsorption of lead metal ions (Pb^{2+}) is strongly affected by initial solution pH, initial metal ion concentration and adsorbent doses. It has been found that the amount of adsorption of lead increases with initial metal ion concentration, contact time and solution pH but decreases with the amount of adsorbent respectively. The adsorbent exhibited good sorption potential for lead at pH 6. Freundlich isotherm adsorption equations reasonably describe the adsorption isotherm within the metal ion concentration range. Perhaps coconut coir has the potential to be used for the large scale separation of lead from wastewater.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Heavy metal ions such as cadmium, zinc, copper, lead and nickel are considered to be hazardous to both human life and the environment. Lead poisoning in human causes severe damage to kidney, nervous system, reproductive system, liver and brain. Severe exposure to lead has been associated with sterility, abortion, stillbirths and neo-natal deaths (O.S. Amuda, A.A. Giwa, I.A. Bello, 2007). Contamination of water with lead heavy metal ions is a large environmental concern associated with the increasing of industrial processes which discharge aqueous effluents containing lead ions. Lead uses many industries such as acid battery manufacturing, metal plating and finishing, printing, photographic materials, explosive manufacturing, and tetraethyl lead manufacturing, and ceramic and glass industries (O.Gercel and H.F Gercel, 2007). Lead ions are not biodegradable and tend to accumulate in living organisms, causing various health problems. According to the World Health Organisation, the acceptable range of lead ions in water is 0.01 ppm (Waid Omar and Hossam Al- Rawi, 2007).

There are many conventional processes for the removal of lead and the other heavy metals from industrial wastewater streams such as precipitation, ion exchange, membrane filtration and reverse osmosis. However, these methods have somehow proven disadvantageous in as much as expensive equipments and continuous need of chemicals are required. Moreover, sometimes the above-mentioned methods fail to meet the Environment Protection Agency requirements. Considering the limitations of conventional methods for metal removal, the most promising alternative appears to be the adsorption process. This process is found to be more effective and attractive due to its lower costs and higher efficiency of heavy metal ions removal. Activated carbon (AC) is the most commonly used and most effective adsorbent. Nevertheless, its application fields are restricted due to its high cost. The use of low-cost wastes and agriculture by-products has been shown to provide economical solution to this problem.

Generally, the raw materials for the production of AC are those with high carbon but low inorganic contents such as wood, lignite, peat and coal. Beside that, lot of agricultural waste and by product have successfully converted to AC for examples macadamia nutshell, paper mill sludge and peach stones. In Malaysia there are potential raw materials resources for the production of the activated carbon. In this research, a local agricultural waste which is coconut coir is used to produce an activated carbon due to the availability and inexpensive material with high carbon and low inorganic content.

1.2 Problem Statement

The separation process for heavy metal removal in water contaminant is crucial in order to fulfil the environment requirement. Thus, the right action must be taken to overcome this problem. Although there are a lot of methods to separate it but economic factors must be considered. Some process need expensive equipments and continuous need of chemicals. The most relevant process to remove heavy metal is adsorption.

Adsorption is found to be the most promising alternative to remove the heavy metal from industrial wastewater. For this project, activated carbon will be used as adsorbent. Although there are other types of adsorbent such as synthetic polymeric and silica-based adsorbents but they are seldom used for wastewater adsorption because of their high cost. Activated carbon can be prepared from organic materials such as almond, coconut, walnut hulls, woods and coal but in this study, coconut coir has been chosen as the material to prepare the activated carbon. This is because of its low-cost natural adsorbent and it can be found easily in Malaysia.

1.3 Objectives and Scope of Study

The main objective of this project is to study on the feasibility of the activated carbon prepared from coconuts coir for lead removal. To achieve this objective, there are several objectives that need to be achieved:

i. To produce activated carbon from local coconut coir that can be used to remove heavy metals in solution.
ii. To investigate and study the characteristics of the produced activated carbon.
iii. To study on the adsorption process for lead removal from the aqueous solution by using low-cost organic material adsorbent.
iv. To analyse the effect of initial metal ion concentration, pH of the solution and amount of adsorbent used on the adsorption process.

The scope of study for this project starts with the knowledge gathering and theoretical studies. After that, the chemicals and equipments that will be used must be identified. Familiarization on the equipments is important to avoid faulty in the experiment. Since this project is a lab-based, several experiments have to be carried out in order to achieve the objectives. The scope is extended to the usage of the lab equipments. Results obtained from the laboratory tests will be analysed and interpreted.

1.4 Relevancy of the Project

The relevancy of the project can be seen in removing the lead ions in wastewater. This project can be applied in industrial since it provides a better process to treat the water before it can be discharged with a maximum allowable concentration.

CHAPTER 2

LITERATURE REVIEW

2.1 Preparation of Activated Carbon

The production of activated carbon is the first phase of this project. The activated carbon that is produced will be characterized. In this section, basic terms and theories which are related with the preparation of the activated carbon will be discussed.

2.1.1 Low- cost Activated Carbon

Among the practical solids used in industries, activated carbon is one of the most complex solids but it is the most versatile because of its extremely high surface area and micropore volume. Activated carbon (AC) is known as a solid, porous, black carbonaceous material and tasteless. Marsh (1989) defined AC as a porous carbon material, usually chars, which have been subjected to reaction with gases during or after carbonization in order to increase porosity. AC is distinguished from elemental carbon by the removal of all non-carbon impurities and the oxidation of the carbon surface. While according to Norlia Baharun (1999) AC is an organic material that has an essentially graphitic structure. The main features common to all AC are; graphite like planes which show varying degrees of disorientation and the resulting spaces between these planes which constitute porosity, and the unit built of condensed aromatic rings are referred to as Basic Structure Units (BSU) (Benaddi, 2000). Benaddi (2000) also stated that AC is predominantly an amorphous solid with a large internal surface area and pore volume. Cokes, chars and activated carbon are frequently termed amorphous carbon.

Although there are several types of adsorbents such as activated carbon, synthetic polymeric and silica-based adsorbents but synthetic polymeric and silica-based adsorbents are seldom used for wastewater adsorption because of their high cost. Activated carbon is the most commonly used and most effective adsorbent (R. Baccar, J. Bouzid, M.Feki and A. Montiel, 2009). However, its application fields are restricted due to its high cost. The use of low-cost wastes and agriculture by-products to produce activated carbon has been shown to provide economical solution to this problem (G. Cimino, R.M. Capello, C.Caristi, G.Toscazo, 2005), (O.Ioannidou, A.Zabaniotou, 2007). Numerous waste biomass sources are available in different parts of the world, on which some experimental adsorption properties have been reported e.g. rise husk (D. Kalderis et. al, 2008), peanut shells (W.Wafwoyo, C.W. Seo, W.E. Marshall, 1999), corn cobs (T.Vaughan, C.W. Seo, W.E. Marshall, 2001) and Tunisian olive-waste (R. Baccar, J. Bouzid, M.Feki and A. Montiel, 2009).

2.1.2 Preparation of Activated Carbon

There are 2 major preparation methods that have been used nearly 100 years ago, and are roughly classified as "chemical activation" and "thermal activation" (NISSIM, Stuart, H et al., 2000). The purpose of an activation process is to increase the volume and diameter of the pores, created during the process of carbonization. In the chemical activation, both processes of carbonization and activation take place simultaneously. There are two major modern preparation methods have been patented and are roughly classified as chemical activation and physical activation (NISSIM, Stuart, H et al., 2000). The chemical methods involve activation with acids such as H_3PO_4 or combinations of H_2SO_4 and HNO_3, or mixing with $ZnCl_2$ as well as many other compounds. Chemical activation presents two important advantages: (1) a much higher yield and (2) the surface of the fibres prepared by chemical activation are less damaged than by physical activation (J.A. Macia-Agullo, B.C. Moore, D. Cazorla-Amoros, A. Linares-Solano, 2003).

Thermal activation is also called as physical activation. The physical activation of agricultural waste product-based carbon with steam or carbon dioxide is a conventional manufacturing process of the activated carbon (R.C. Bansal, J.P.

Donnet, F.Stoeckli, 1988). The physical activation process consists of a preliminary thermal treatment of the carbonaceous precursor in an inert environment, called pyrolysis followed by suitable activation of the resulting char using steam or carbon dioxide. It was reported in literature that air is not widespread used as oxidant, although lower activation temperatures could be used, as carbon–oxygen reactions are exothermic and can result in thermal runaway and extreme burn off (Turkan Kopac, Atakan Toprak, 2007). The carbonized material is mixed with gases at high temperature to activate it. The source material can be several carbonic materials, e.g. coconut shell, nutshells, wood, coal. It's also generally done by using one or combining carbonization and activation/oxidation process. Carbonization process happened when the material with carbon content is pyrolysed at temperatures in the range 600-900 °C, in absence of air while activation/oxidation happened when raw material or carbonized material is exposed to oxidizing atmospheres (carbon dioxide, oxygen, or steam) at temperature range 600-1200 °C.

2.1.3 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a tool that recognizes types of functional groups in a molecule (Masita Mohammad, 2008). It is by producing an infrared absorption spectrum that is like a molecular "fingerprint". FTIR is used to identify chemicals that are either organic or inorganic. By interpreting the infrared absorption spectrum, the functional groups in a molecule can be determined. FTIR spectra of pure compounds are so unique that they are called a molecular "fingerprint". Organic compounds have very rich, detailed spectra where else inorganic compounds are usually much simpler.

Figure 2.1: Fourier Transform Infrared Spectroscopy (FTIR)

Bonds and groups of bonds vibrate at specific frequencies. A molecule that is exposed to infrared rays absorbs infrared energy at frequencies which are specific to that molecule. FTIR spectroscopy does not require a vacuum condition since neither oxygen nor nitrogen can absorb infrared rays. FTIR analysis can be applied to small quantities of materials, whether solid, liquid, or gaseous. The adsorption capacity of activated carbon depends upon porosity as well as the chemical reactivity of functional groups at the surface. Knowledge on surface functional groups would give insight to the adsorption capability of the produced activated chars. FTIR spectra are collected for qualitative characterization of surface functional groups of porous carbons activated by both physical and chemical methods (Viboon Sricharoenchaikul, Chiravoot Pechyen, Duangdao Aht-ong and Duangduen Atong, 2007).

2.1.4 Scanning Electron Microscope (SEM)

Conventional light microscopes use a series of glass lenses to bend light waves and create a magnified image. SEM creates the magnified images by using electrons instead of light waves. The SEM shows 3-dimensional images at much higher magnifications. The images are very detailed. The images created without light waves are rendered black and white (Karine Flogeac, Emmanuel Guillon, Michel Aplincourt, 2005). It can be used to estimate the pore size of the activated carbon quantitatively. Pore entrances, clearly visible as dark irregular shapes on lighter membrane background.

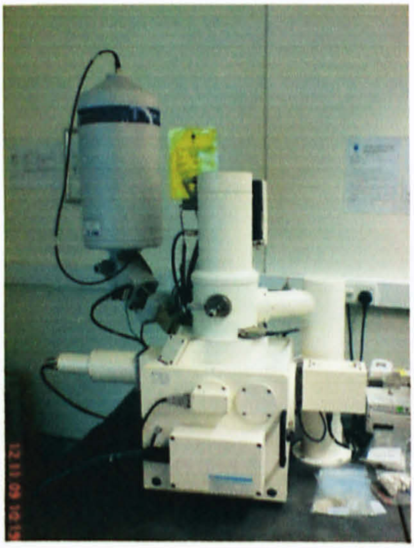


Figure 2.2: Scanning Electron Microscope (SEM)

2.1.5 Thermo Gravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) used to measure the quantity and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Measurements are used to determine the composition of materials and to predict their thermal stability at temperatures up to 1000°C. The technique can characterize materials that demonstrate weight loss or gain due to decomposition, oxidation, or dehydration. Facts like thermal stability of materials, oxidative stability of materials, composition of multi-component system, decomposition kinetics of materials, effects of reactive or corrosive atmospheres on materials and finally moisture and volatiles content of materials can be found by using TGA.

2.1.6 X-ray Diffraction (XRD)

X-ray diffraction (XRD) is used to reveal the chemical structural changes associated with the sorption process. X-ray Diffraction (XRD) is one of the most powerful techniques for qualitative and quantitative analysis of crystalline compounds. The technique provides information that cannot be obtained any other way. The information obtained includes types and nature of crystalline phases present, structural makeup of phases, degree of crystallinity, amount of amorphous content, microstrain & size and orientation of crystallites. The use of XRD for this project is to investigate the surface inorganic components of the prepared activated carbons.

2.2 Adsorption Experiment

In order to study the effectiveness of the activated carbon produced, adsorption experiment must be conducted.

2.2.1 Adsorption Process

Adsorption is the process of accumulating substances that are in solution on a suitable interface. Adsorption is a mass transfer operation in that a constituent in the liquid phase is transferred to the solid phase. Heavy metals are nowadays among the most important pollutants in source and treated water, and are becoming a severe public health problem, can be toxic to aquatic life and cause natural waters to be unsuitable as potable water sources (Serpil Cetin and Erol Pehlivan, 2007). Heavy metal removal from aqueous solutions has been commonly carried out by several processes: chemical precipitation, solvent extraction, ion-exchange, reverse osmosis or adsorption. Among these processes, the adsorption with the selection of a suitable adsorbent can be an effective technique for the removal of heavy metals from wastewater (R. Baccar, J. Bouzid, M.Feki and A. Montiel, 2009). Although these suggested adsorbents are activated carbon, alumina, silica and ferric oxide, which generally have high metal adsorption capacity, they are expensive and difficult to be separated from the wastewater after use. Therefore, considering the cost economics, recent research has focused on the development of alternative low-cost adsorbents using various agricultural, industrial, natural/biological waste materials. Another reason for this interest is that importance of adsorption on solid surfaces in many industrial applications in order to improve efficiency and economy. Therefore, it is essential to understand adsorption characteristics such as mechanism and kinetics of adsorption.

2.2.2 Heavy Metal (Lead)

Lead is a main group element with a symbol Pb (Latin: plumbum). Lead has the atomic number 82. Lead is a soft, malleable poor metal, also considered to be one of the heavy metals. Lead is a poisonous metal that can damage nervous connections (especially in young children) and cause blood and brain disorders. Because of its low reactivity and solubility, lead poisoning usually only occurs in cases when the lead is dispersed, like when sanding lead based paint, or long term exposure in the case of pewter tableware. Long term exposure to lead or its salts (especially soluble salts or the strong oxidant PbO_2) can cause nephropathy, and colic-like abdominal pains. Lead can also be found listed as a criteria pollutant in the United States Clean Air Act section 108. Lead that is emitted into the atmosphere can be inhaled, or it can be ingested after it settles out of the air. It is rapidly absorbed into the bloodstream and is believed to have adverse effects on the central nervous system, the cardiovascular system, kidneys, and the immune system.

2.2.3 Adsorption Experiment

A measured quantity of the adsorbent and 25 ml of the metal ion solution will be shaken in a constant temperature shaker at 120 rpm for a given time and then the suspensions will be filtered through a 0.45 μm syringe filter (Tarun Kumar Naiya, Ashim Kumar Bhattacharya, Sailendranath Mandal and Sudip Kumar Das, 2009). The filtrate was analysed using flame atomic absorption spectrophotometer with air-acetylene flame. The pH of the liquid is adjusted with dilute HCl and NaOH solution. The experiments will be carried out by varying the initial metal ion concentration, amount of adsorbent and the pH. Adsorption rate is measured according to predefined procedure with both metal ion concentration ranging from 5.0 to 50 mg/L (Tarun Kumar Naiya, Ashim Kumar Bhattacharya, Sailendranath Mandal and Sudip Kumar Das, 2009). The metal ion concentration retained in the adsorbent phase is calculated according to

$$q_t = \frac{(C_o - C_t)V}{m}$$

Where C_o (mg/L) and C_t are the concentration in the solution at time t=0 and at time, t, V is the volume of solution (L) and m is the amount of adsorbent (g) added (Masita Mohammad, 2008).

2.2.4 Adsorption Isotherm

Adsorption isotherms were derived from batch experiments. Isotherms are empirical relations, which are used to predict how much solute can be adsorbed by activated carbon. The three most well known isotherms are the Freundlich, Langmuir and Linear. The quantity of adsorbate that can be taken up by an adsorbent is a function of both the characteristics and concentration of adsorbate and the temperature. Normally, the resulting function of the amount of the concentration at a constant temperature is called an adsorption isotherm. Adsorption isotherms are developed by exposing a given amount of adsorbate in a fixed volume of liquid with vary amounts of activated carbon. The equilibrium data are analyzed in accordance with the Langmuir and Freundlich sorption isotherms.

CHAPTER 3

METHODOLOGY

3.1 Project Work Flow

Before proceeding with the lab work, steps are drawn out diagrammatically to clear out on the project flow.

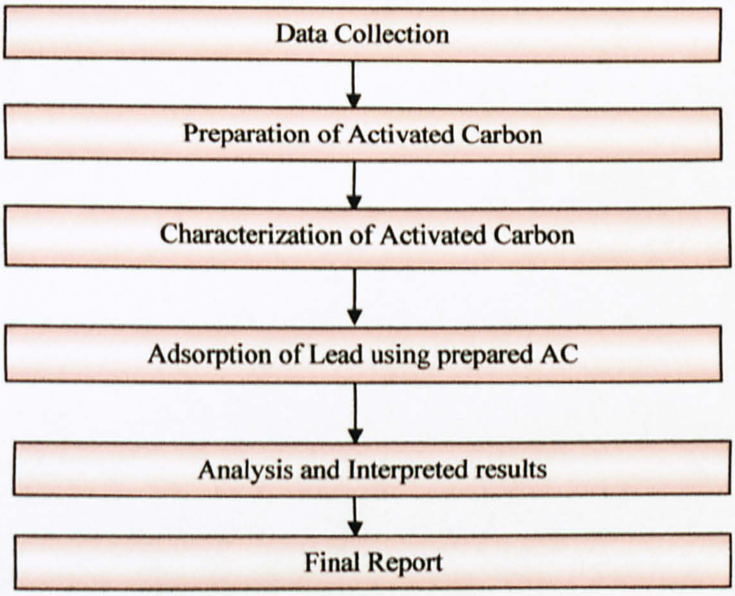


Figure 3.1: Flowchart of Project Work Flow

3.2 Chemicals

Plumbum nitrate, $Pb(NO_3)_2$, Hydrochloric acid, HCl, Sodium hydroxide, NaOH, Sodium bicarbonate, $NaHCO_3$, and activated carbon prepared from coconut coir.

3.3 Apparatus

1000ml volumetric flask, 100ml bottle sample, pipette, magnetic stirrer, rotary shaker, 5ml sample bottle, electronic balance, spatula, pH meter, graduated cylinder, Atomic Adsorption Spectrophotometer (AAS), X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Scanning Electron Microscope (SEM), Energy Dispersive X-ray Spectroscopy (EDX), Fourier Transform Infrared Spectroscopy (FTIR).

3.4 Preparation of activated carbon

Grinded coconut coir was used as raw material for the production of activated carbons via both chemical and physical activation. First of all, the grinded coconut coir was washed with distilled water and was oven dried for 24 hours at 105 °C. 10% sodium hydroxide (NaOH) solution was mixed with dried coconut coir in 1:1 ratio (for example 50 g of dried coir is treated with 50 ml of 10% NaOH) and then was placed in the over for 1 day. After that it was washed with distilled water and placed in 2% $NaHCO_3$ solution overnight. The coconut coir was then washed with distilled water and placed in the oven at 105 °C for 24 hours. Next the physical activation was carried out in a furnace at 30 °C up to 900 °C in the presence of nitrogen gas at flow of 150 ml/min for 30 minutes (30 minutes was started to count when the temperature reached 900 °C). After the cooling, it was washed with 10% hydrochloric acid (HCl) and distilled water. The sample was then dried at 150 °C overnight and sieved to particle size ranging 300-500 µm.

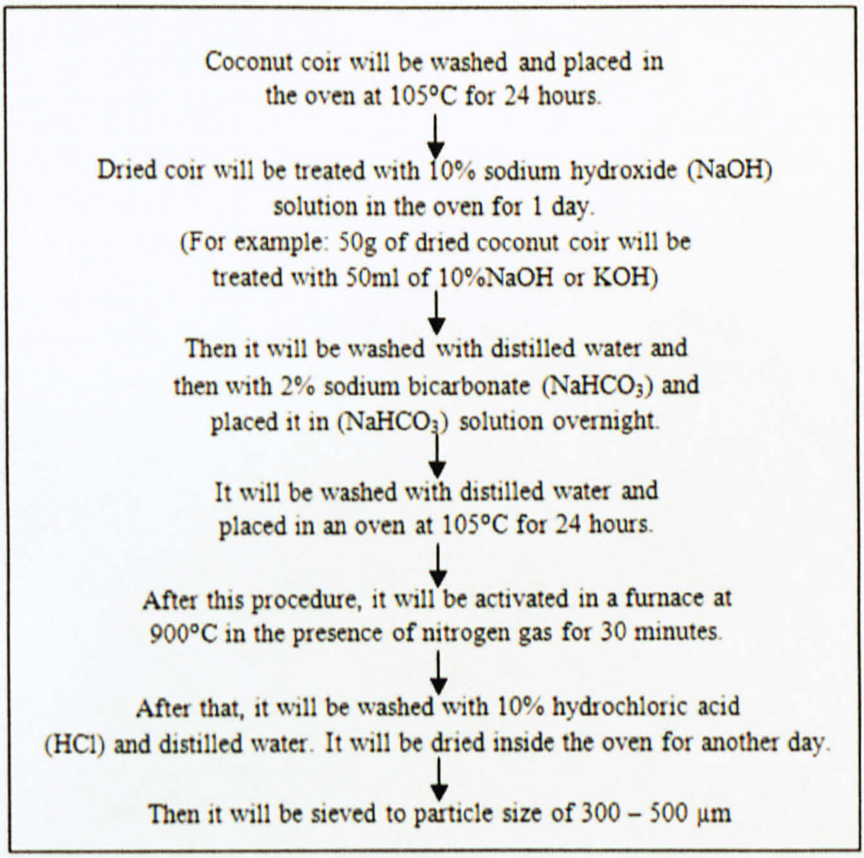
3.4.1 Activation of AC using Fixed Bed Activation Unit (FBAU)

Thermal activation process was taken place in FBAU in the presence of nitrogen (N_2). The procedure to operate the FBAU is listed below.

1. The treated coconut coir was placed in the activation unit.
2. The main MCB which is placed in the control panel was switched 'ON'.
3. The 'ON' pushbutton on the Control Panel was pressed to power on the system.

4. The pushbutton for Valve 1 and Valve 5 were pressed.
5. Nitrogen gas was supplied to the activation unit at flow of 0.15 (150ml/min).
6. The temperature was set at 900°C. *(Note: The temperature was increased by 100).*
7. 30 minutes was started to count when the temperature had reached 900°C.
8. Sample was taken out when temperature is less than 100°C.

Figure 3.2 Fixed Bed Activation Unit (FBAU)



Coconut coir will be washed and placed in the oven at 105°C for 24 hours.

↓

Dried coir will be treated with 10% sodium hydroxide (NaOH) solution in the oven for 1 day.
(For example: 50g of dried coconut coir will be treated with 50ml of 10%NaOH or KOH)

↓

Then it will be washed with distilled water and then with 2% sodium bicarbonate ($NaHCO_3$) and placed it in ($NaHCO_3$) solution overnight.

↓

It will be washed with distilled water and placed in an oven at 105°C for 24 hours.

↓

After this procedure, it will be activated in a furnace at 900°C in the presence of nitrogen gas for 30 minutes.

↓

After that, it will be washed with 10% hydrochloric acid (HCl) and distilled water. It will be dried inside the oven for another day.

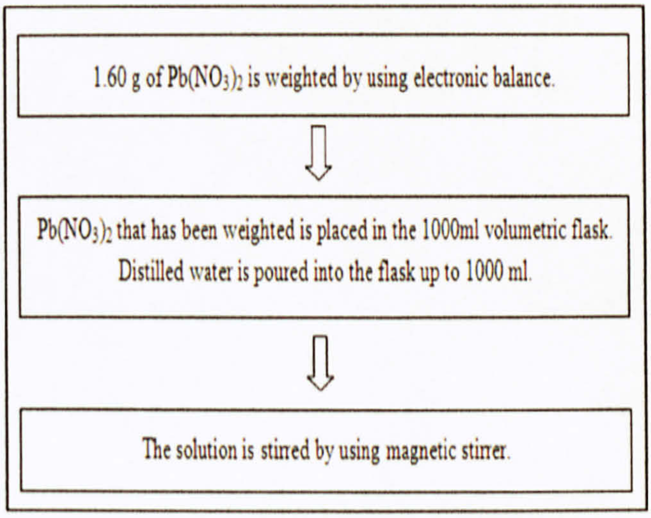
↓

Then it will be sieved to particle size of 300 – 500 μm

Figure 3.3: Summary the Methodology of AC Preparation

3.5 Synthetic Waster water Preparation

Stock solution which is 1000 ppm for lead must be prepared. The salt used for the preparation of lead solution is lead nitrate, $Pb(NO_3)_2$ because it is soluble in water. The stock solution containing 1000 mg/L of standard Pb(II) were prepared by dissolving 1.60 g of $Pb(NO_3)_2$ (Tarun Kumar Naiya, Ashim Kumar Bhattacharya, Sailendranath Mandal and Sudip Kumar Das, 2009). Standard solution of particular Pb(II) concentration was prepared by proper dilution with 1000 mL of distilled water.



1.60 g of $Pb(NO_3)_2$ is weighted by using electronic balance.

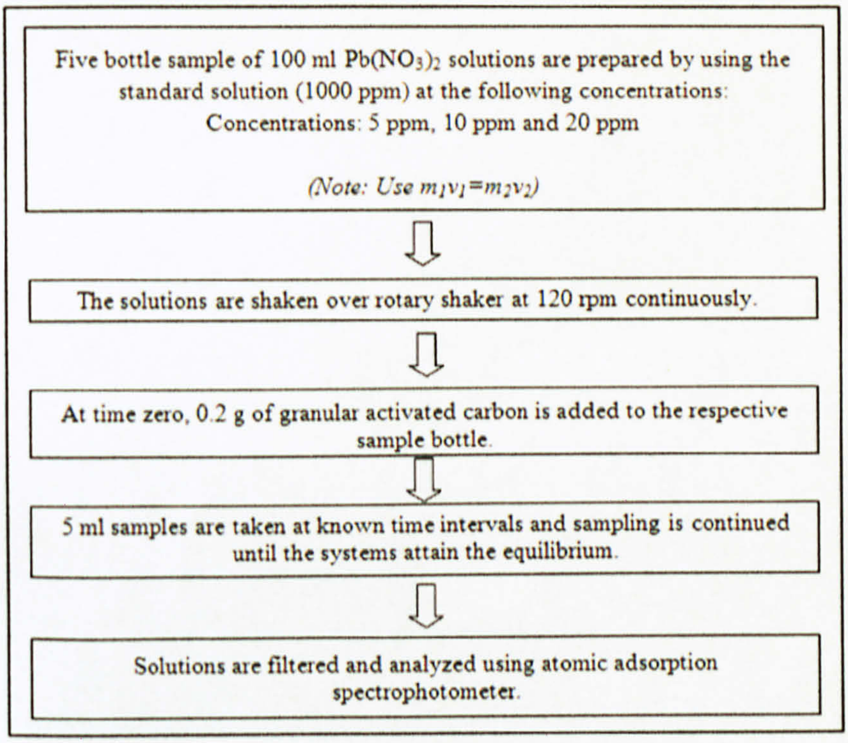
$Pb(NO_3)_2$ that has been weighted is placed in the 1000ml volumetric flask. Distilled water is poured into the flask up to 1000 ml.

The solution is stirred by using magnetic stirrer.

Figure 3.4: Summary of Preparation of 1000 ppm of $Pb(NO_3)_2$

3.6 Isotherm Experiment

The adsorption isotherm is determined by allowing lead ions solutions of known concentration to be mixed with accurately weighted amount of coconut coir activated carbon in a tightly closed bottle sample. Equilibrium isotherm experiments are conducted by mixing 0.2 g of coconut coir activated carbon with 100 ml solution for initial metal concentrations of 5 ppm, 10 ppm and 20 ppm. Those metal solutions can be prepared using the formula of $m_1v_1=m_2v_2$. Continuous mixing is needed during the experimental period with a constant agitation speed of 120rpm for better mass transfer with high interfacial area of contact. The remaining concentration of Pb(II) in each sample after adsorption at different time intervals is determined by atomic-absorption spectroscopy after filtering the adsorbent with 0.45μm syringe filter to make it carbon free.



Five bottle sample of 100 ml $Pb(NO_3)_2$ solutions are prepared by using the standard solution (1000 ppm) at the following concentrations:
Concentrations: 5 ppm, 10 ppm and 20 ppm

(Note: Use $m_1v_1=m_2v_2$)

The solutions are shaken over rotary shaker at 120 rpm continuously.

At time zero, 0.2 g of granular activated carbon is added to the respective sample bottle.

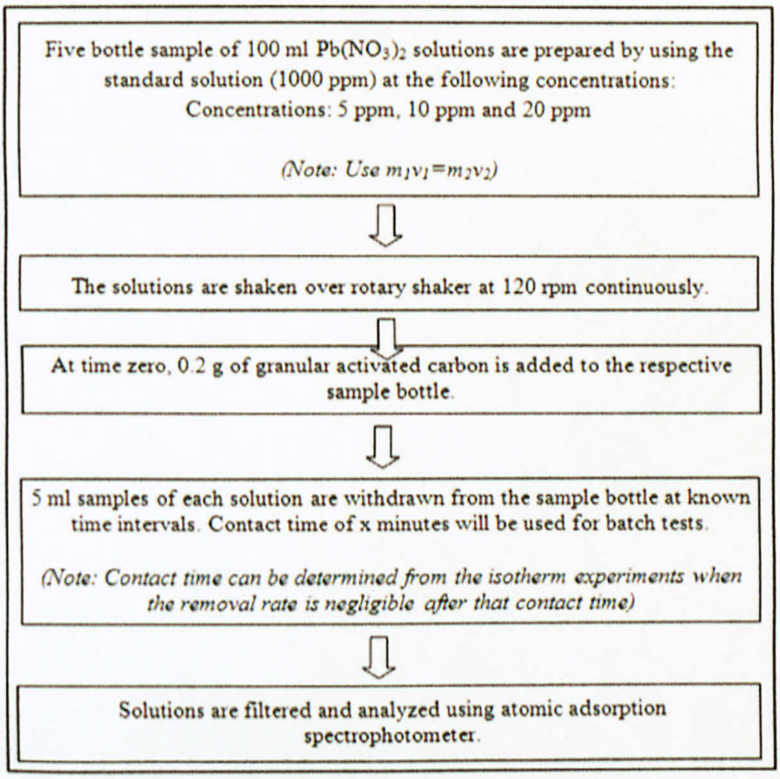
5 ml samples are taken at known time intervals and sampling is continued until the systems attain the equilibrium.

Solutions are filtered and analyzed using atomic adsorption spectrophotometer.

Figure 3.5: Summary of Isotherm Experiment

3.7 Effect of Initial Metal Ion Concentration

Batch adsorption experiments were performed by contacting 0.2 g of the coconut coir activated carbon with 100ml of the aqueous solution of different initial concentration (5ppm, 10ppm and 20ppm). The mixture was shaken in a rotary shaker and 5 ml samples of solution were withdrawn from the bottle sample at known time intervals. Preliminary experiment (Isotherm Experiment) was used to determine the contact time for the batch tests. The sample was filtered to remove any fine particles and analyzed for the metal ions.



Five bottle sample of 100 ml $Pb(NO_3)_2$ solutions are prepared by using the standard solution (1000 ppm) at the following concentrations:
Concentrations: 5 ppm, 10 ppm and 20 ppm

(Note: Use $m_1v_1=m_2v_2$)

The solutions are shaken over rotary shaker at 120 rpm continuously.

At time zero, 0.2 g of granular activated carbon is added to the respective sample bottle.

5 ml samples of each solution are withdrawn from the sample bottle at known time intervals. Contact time of x minutes will be used for batch tests.

(Note: Contact time can be determined from the isotherm experiments when the removal rate is negligible after that contact time)

Solutions are filtered and analyzed using atomic adsorption spectrophotometer.

Figure 3.6: Summary of Effect of Initial Metal Ion Concentration Experiment

3.8 Effect of pH

Effect of initial solution pH on adsorption was determined by mixing 0.2 g of adsorbent with 100 ml of solution containing metal concentration of 20 ppm at various pH values ranging from 3.0 to 6.0. Solution pH was adjusted with 0.5M HCl and NaOH solutions. The mixture was shaken for 1 hour and the solution was filtered and analysed.

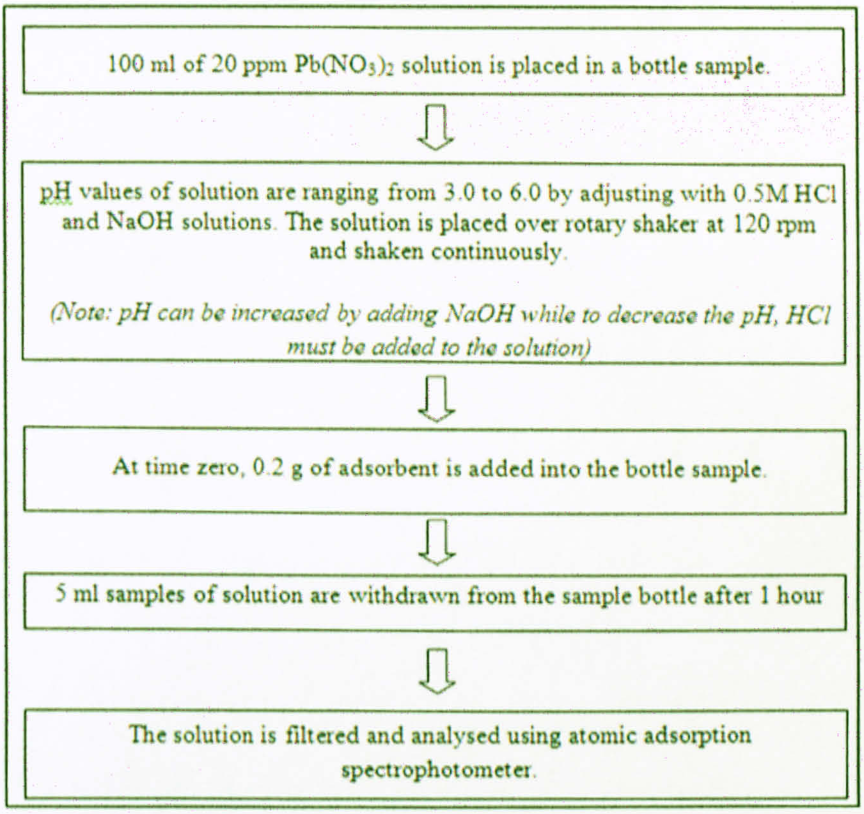
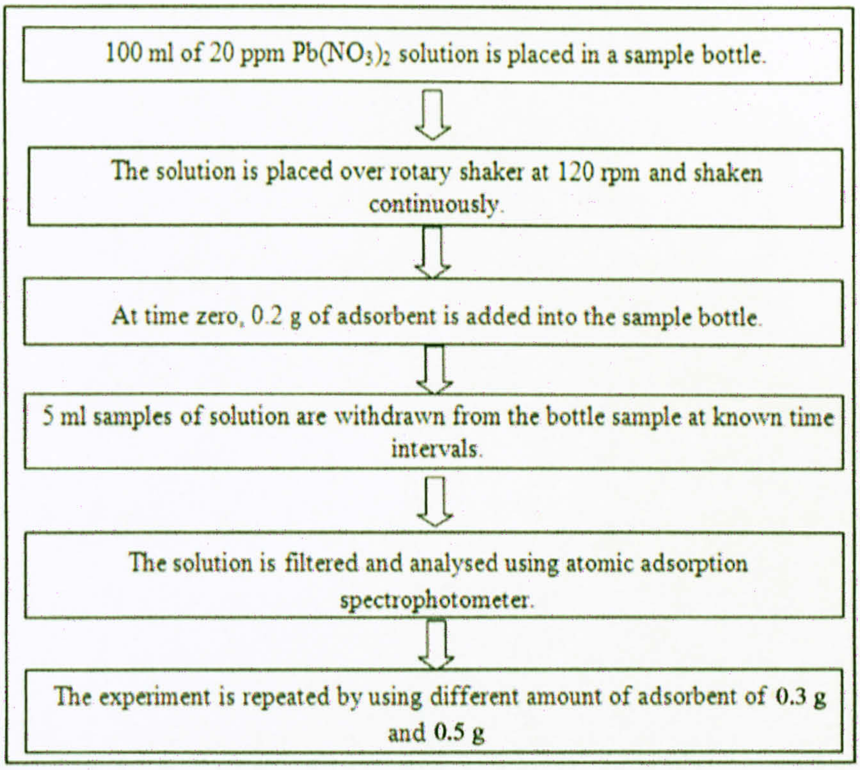


Figure 3.7: Summary of Effect of pH Experiment

3.9 Effect of Adsorbent Dose

The experiment was determined by batch experiment of 0.2 g adsorbent in 100 ml of 20 ppm Pb(II) solutions. The solution was shaken continuously over rotary shaker at constant speed of 120 rpm. 5ml samples of solution were withdrawn from the sample bottle at known time intervals. The sample was filtered and analyzed. The experiment was repeated by using different amount of adsorbent of 0.3 g and 0.5 g.



100 ml of 20 ppm $Pb(NO_3)_2$ solution is placed in a sample bottle.

The solution is placed over rotary shaker at 120 rpm and shaken continuously.

At time zero, 0.2 g of adsorbent is added into the sample bottle.

5 ml samples of solution are withdrawn from the bottle sample at known time intervals.

The solution is filtered and analysed using atomic adsorption spectrophotometer.

The experiment is repeated by using different amount of adsorbent of 0.3 g and 0.5 g

Figure 3.8: Summary of Effect of Adsorbent Dose

3.10 Project Activities and Gantt Chart

3.10.1 Milestone for the First Semester of 2-Semester Final Year Project

No.	Activities/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	🟥	🟥						Mid-semester break							
2	Preliminary Research Work		🟥	🟥	🟥											
3	Submission of Preliminary Report				●											
4	Seminar 1 (optional)					🟥	🟥	🟥								
5	Project Work					🟥	🟥	🟥								
6	Submission of Progress Report									●						
7	Seminar 2 (compulsory)										🟥	🟥	🟥	🟥		
8	Project work continues									🟥	🟥	🟥	🟥	🟥		
9	Submission of Interim Report Final Draft														●	
10	Oral Presentation															●

● Key milestone

3.10.2 Milestone for the Second Semester of 2-Semester Final Year Project

No.	Activities/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Project Work Continue	🟥	🟥	🟥					**Mid-Semester Break**							
2	Submission of Progress Report 1				●											
3	Project Work Continue				🟥	🟥	🟥	🟥		●						
4	Submission of Progress Report 2															
5	Seminar (compulsory)										🟥	🟥	🟥			
6	Project work continue									🟥	🟥	🟥	🟥			
7	Poster Exhibition											●				
8	Submission of Dissertation (soft bound)													●		
9	Oral Presentation														●	
10	Submission of Project Dissertation (Hard Bound)															●

● Key milestone

CHAPTER 4

RESULTS AND DISCUSSION

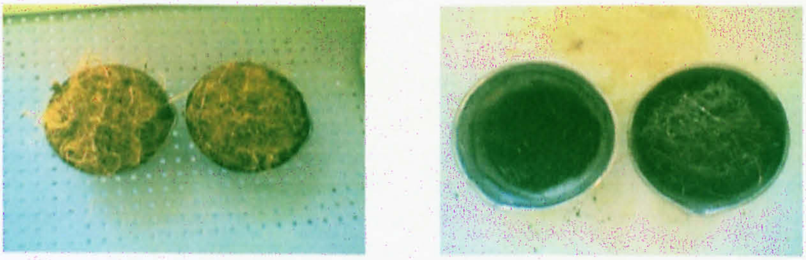
4.1 Preparation of Activated Carbon

The preparation of activated carbon (AC) from coconut coir took about 2 to 3 weeks.

(a) (b)

Figure 4.1 (a) 10% Sodium Hydroxide, NaOH was poured into the dried coconut coir (b) Coconut coir that had been washed with Sodium Bicarbonate, $NaHCO_3$

The amount of the AC lost during the activation (refer to Figure 4.2).



(a) (b)

Figure 4.2 (a) Dried treated coconut coir before activation process (b) Dried treated coconut coir after activation process

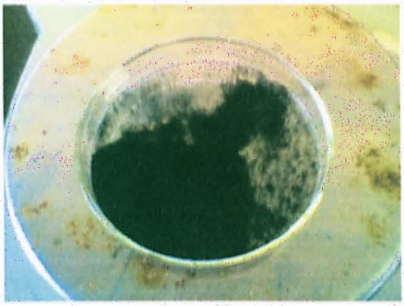


Figure 4.3 Activated carbon prepared from coconut coir

4.2 Characterization of Activated Carbon

4.2.1 Fourier Transform Infrared Spectroscopy (FTIR)

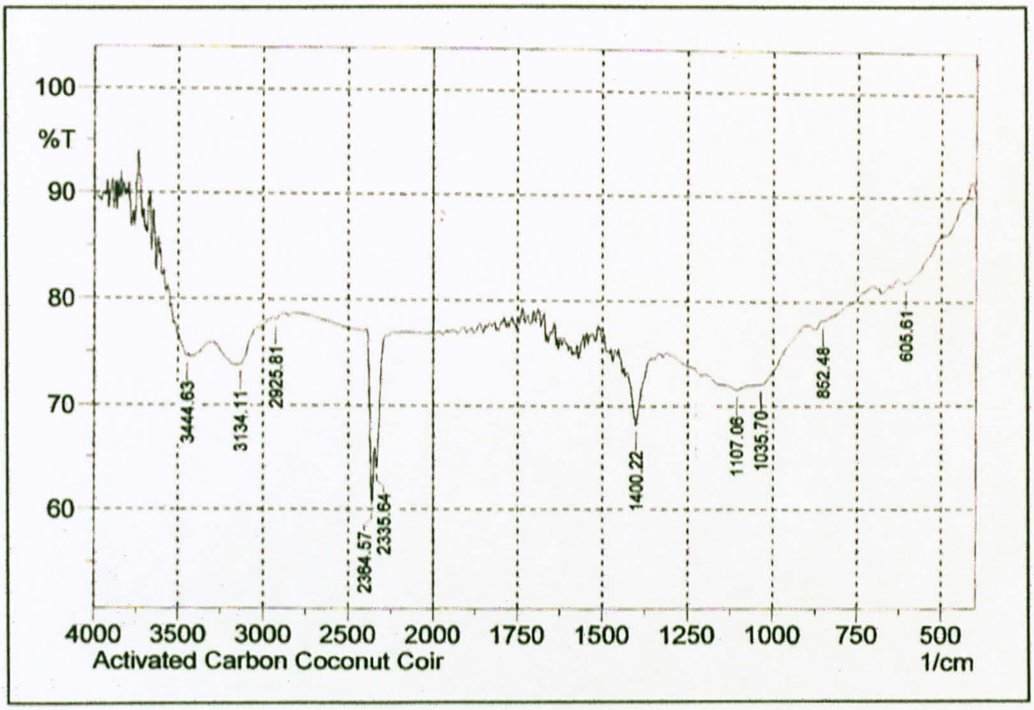


Figure 4.4: FTIR spectrum of activated carbon

A quantitative analysis of activated carbon was conducted by obtaining FTIR transmission spectra of carbon samples. The adsorption capacity of activated carbon depends upon porosity as well as the chemical reactivity of functional groups at the surface. This reactivity created an imbalance between forces at the surface as compared to those within the body, thus leading to molecular adsorption by the van der Waals force. Knowledge on surface functional groups would give insight to the adsorption capability of the produced activated chars. FTIR spectra were collected for qualitative characterization of surface functional groups of porous carbons activated by both physical and chemical methods. Figure 4.4 shows the functional groups of activated carbon prepared from coconut coir.

The FTIR spectrum of coconut coir AC reveals a broad peak at 3444.63 cm^{-1} and 3134.11 cm^{-1} which can be attributed to the O-H group (Masita Mohammad, 2008). The peak observed at 2925.81 cm^{-1} corresponds to the C-H stretching group (Parinda, Suksabye, Paitip Thiravetyan, Woranan Nakbanpote, Supanee Chayabutra, 2006). The alkynes group of C≡C was found around 2364.57 cm^{-1} to 2335.64 cm^{-1} (Jeremias de Souza Macedo, Nivan Bezerra da Costa Júnior, Luis Eduardo Almeida, Eunice Fragoso da Silva Vieira, Antonio Reinaldo Cestari, Iara de Fátima Gimenez, Neftali Lênin Villarreal Carreño and Ledjane Silva Barreto, 2006). The vibration at 1400.22 cm^{-1} is assigned to the alkanes group of C-H. The peaks from 1035.70 cm^{-1} to 1035.70 cm^{-1} are assigned to the carbonyl stretching group, C-OH stretching in primary alcohol (1035.70 cm^{-1}) and secondary alcohol (1107.36 cm^{-1})(Parinda, Suksabye, Paitip Thiravetyan, Woranan Nakbanpote, Supanee Chayabutra, 2006).

4.2.2 X-ray Diffraction (XRD)

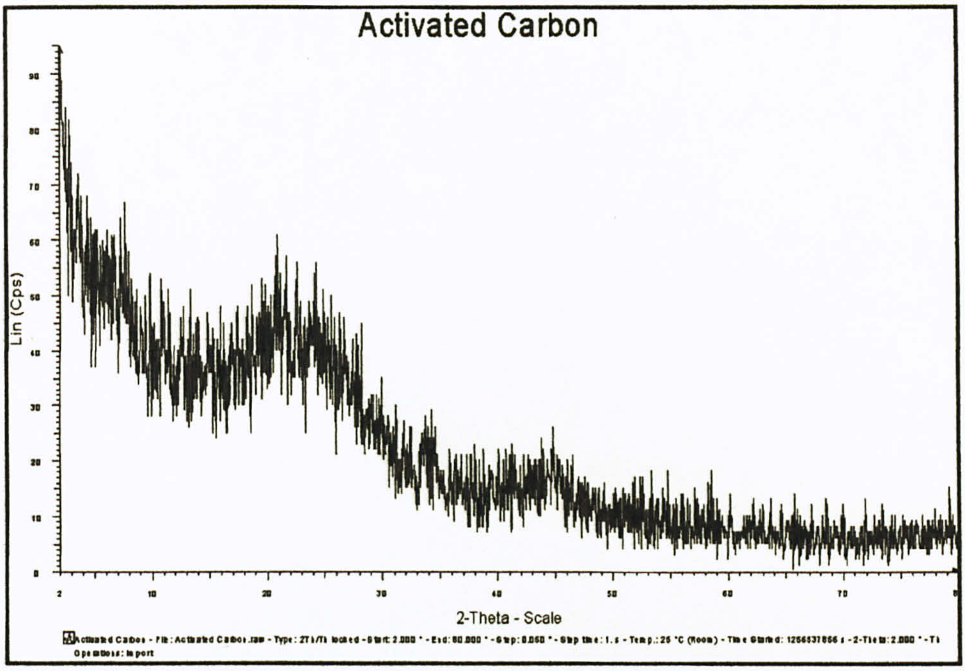


Figure 4.5: X-ray diffractogram for activated carbon from coconut coir

The crystal structure characteristics of the result samples were analyzed by X-ray Diffraction (XRD) as a radiation source. The measurement was carried out in a scale 2θ and long duration scan 15s. The x-ray diffraction patterns of activated carbon sample are shown in Figure 4.5. From the figure, a broad peak existed at $2\theta = 22^{\circ}$ with relative intensity of 58 cps for coconut coir correspond to the lattice plane cellulose. Reasonably, the peaks for the coconut coir increased with density which is commonly observed in nano sized carbon. The sharp lines superimposed on the broad carbon peaks are identified as silica, perhaps unavoidable impurity in activated carbon (Yong Lei, Qinglin Wu, Fei Yao and Yanjun Xu, 2007).

The cell walls of most of the natural fibers mainly consist of cellulose, hemicellulose, and lignin. Cellulose has both amorphous and crystalline regions, although hemicellulose and lignin are amorphous. The intensity of the peaks did change, suggesting differences in crystallinity and also their crystallite size depend on the heat treatment. The XRD pattern of activated carbon fiber shows almost no diffraction lines and is confirmed to be the amorphous material (Roger M. Rowell, James S. Han and Jeffrey S. Rowell, 2000). The XRD pattern of result sample after synthesis shows the diffraction lines corresponding to cellulose in a face centered cubic structure.

Due to their thinness (or small "crystallite size") and the polycrystalline morphology of the coconut coir, their x-ray diffraction diagrams do not exhibit sufficiently detailed diffraction data to permit a full structure analysis to be completed, such as can be done with large single crystals (Sarko). The relative low temperature allows the formation of small amounts of stable structure which is evidenced by the peak located at around diffraction angle of 50°. In addition to the diffraction lines, a broad peak at low diffraction angles is observed corresponding to the presence of amorphous phase in the sample (Z. Al-Qodah and R. Shawabkah, 2009). That could be attributing to the unreacted remaining carbon. When the coconut coir is treated with acids the majority of those peaks disappear due to leaching out the corresponding minerals during activation and washing with water.

4.2.3 Scanning Electron Microscope (SEM)

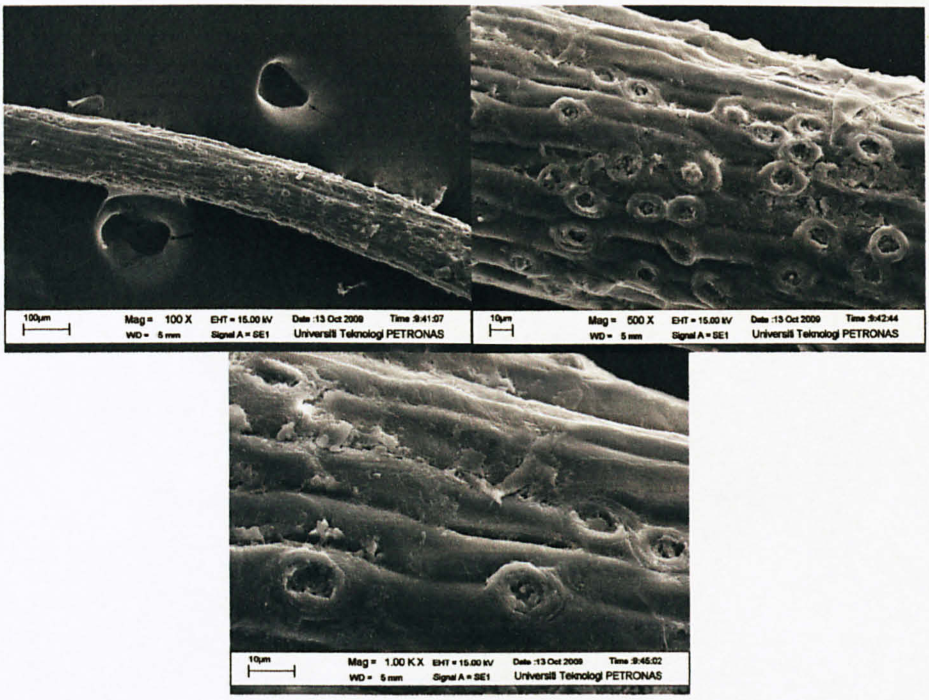


Figure 4.6: Images of long pieces of raw coconut coir

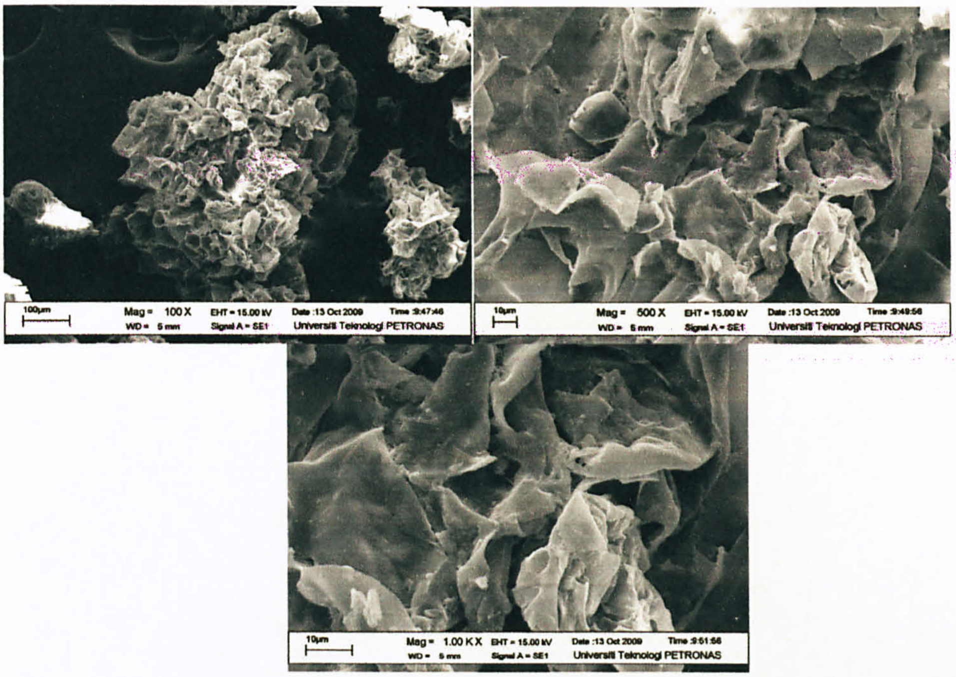


Figure 4.7: Images of small dust of raw coconut coir

Figure 4.6 and Figure 4.7 are the images of raw coconut coir that have not undergone any chemical reaction. There are 2 parts of the coir that had been analyzed. Those parts are from the long pieces of the coir and the small dust of the coir. The images had been magnified for 100, 500 and 1000 times. The images shows that there are quite a number of small pores that is important for adsorption. In Figure 4.6, there are small pores but very little in number. Meanwhile, Figure 4.7 shows the structure of the dusts that are not uniform. There are also a lot of small pores or spaces inside the structure.

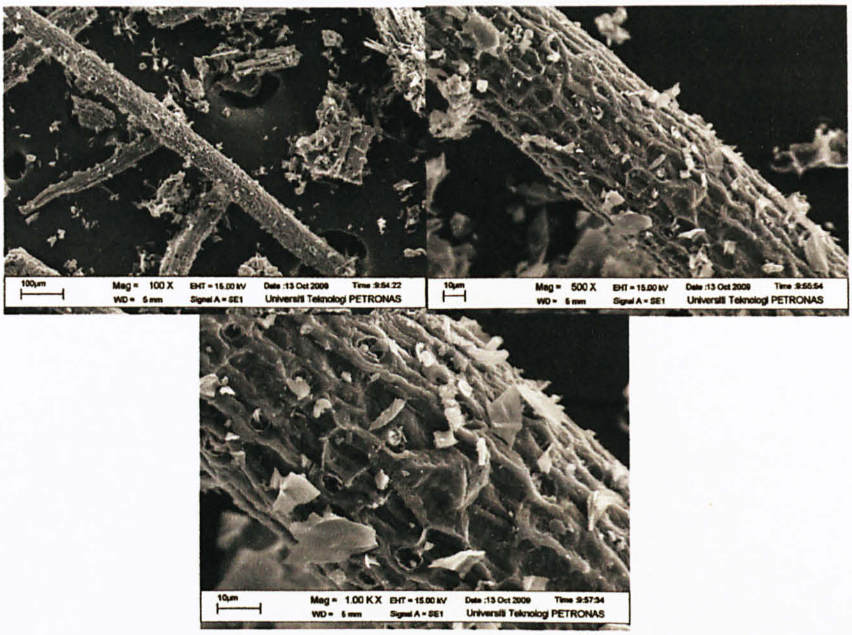


Figure 4.8: Images of long pieces of the activated carbon

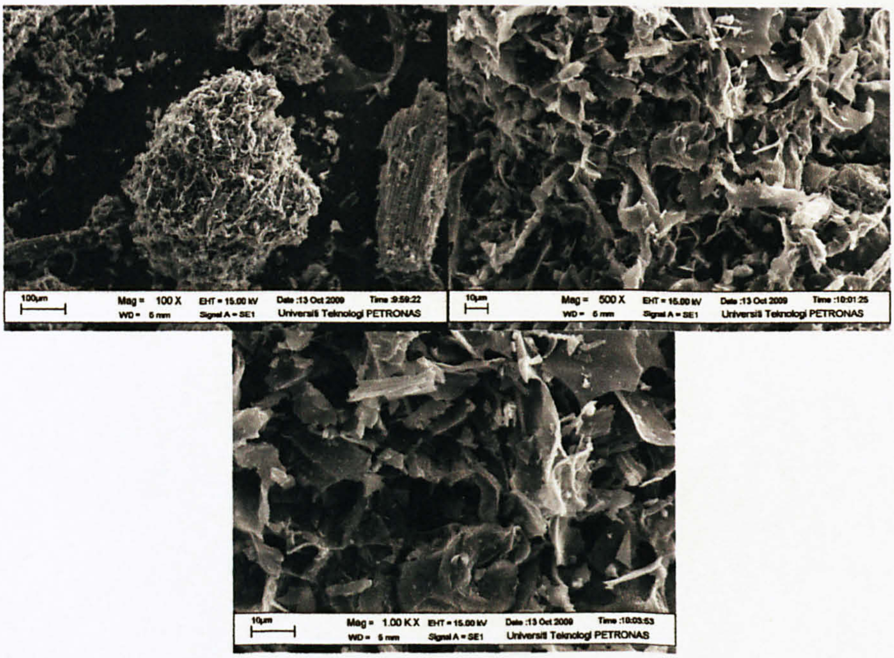


Figure 4.9: Images of small dust of the activated carbon

Figure 4.8 and Figure 4.9 show the structure of the coconut coir that had been converted into activated carbon. The number of pores of the coconut coir increased after the chemical and physical activation. From the images, they show that the ability of the coir to adsorb will be higher because of greater number of pores. The structures have become more complex. Much more "chamber" like structures had been formed. Thus it will increase the capacity of adsorption.

4.3 Adsorption Experiments

4.3.1 Isotherm Experiment

The adsorption isotherm for the removal of metal ion was studied using initial concentration of between 5 and 20 ppm at an adsorbent dosage level of 0.2g for Pb(II) 25 ∘C. The adsorption equilibrium data are conveniently represented by adsorption isotherms, which correspond to the relationship between the mass of the solute adsorbed per unit mass of adsorbent q_t and the solute concentration for the solution at equilibrium C_e.

Adsorption equilibrium data or isotherms are required in designing an adsorption system. The measured adsorption equilibrium data are fitted with Langmuir and Freundlich adsorption isotherm. These models are used to describe the adsorption of lead ions within the initial metal ion concentration range. Linear regression was frequently used to determine the most fitted isotherm (Tushar Kanti Sen and Meimoan Velyny Sarzali, 2008).

The Freundlich adsorption isotherm, which assumes that adsorption takes place on heterogeneous surfaces, can be expressed as

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

Where q_e is the amount of lead ions adsorbed at equilibrium time, C_e is equilibrium concentration of lead metal ions in solution. K_f and n are isotherm constants which indicate the capacity and the intensity of the adsorption, respectively and can be calculated from this plot (refer to Figure 4.10) are 3.294 and -0.445 respectively.

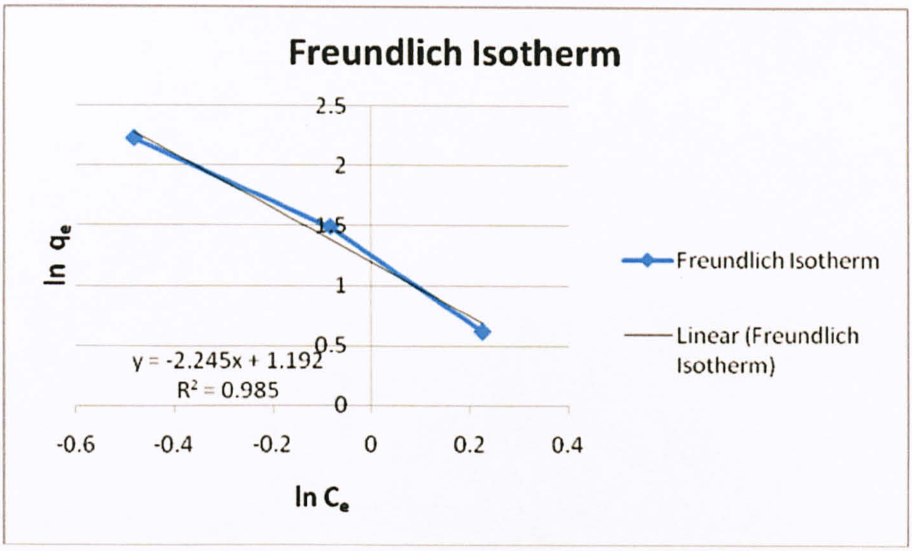


Figure 4.10: Freundlich isotherm plot for coconut coir AC as adsorbent (initial Pb^{2+}= 5, 10 and 20 ppm; amount of AC= 0.2g; shaker speed= 120 rpm)

Langmuir isotherm equation was tested with this same metal ion concentration range. The linearized form of Langmuir can be written as

$$\frac{1}{q_e} = \left(\frac{1}{K_a q_m}\right)\frac{1}{C_e} + \frac{1}{q_m}$$

The Langmuir constant, q_m (maximum adsorption capacity) and K_a (values for Langmuir are predicted from the plot between $1/q_e$ versus $1/C_e$) are shown in Figure 4.11. The maximum adsorption capacity of Pb^{2+}, q_m and constant related to the binding energy of the sorption system, K_a is calculated which are 1.168 mg/g and -1.769 respectively. Both calculated values for Freundlich and Isotherm gave undesirable values. This is may caused from the selection of initial metal ion concentrations and amount of adsorbent used in this experiment. The difference between those concentrations is quite small while the amount of adsorbent used is a big amount for all it means C_e and q_e can be close to each other. However, for this project, it should fit with the Freundlich isotherm since this type of isotherm is used for solid-liquid systems. 1/n value should also lie between 0 and 1 to indicate favorable adsorption. Besides, Freundlich isotherm is used most commonly to describe adsorption characteristics of the activated carbon used in water and waste water treatment.

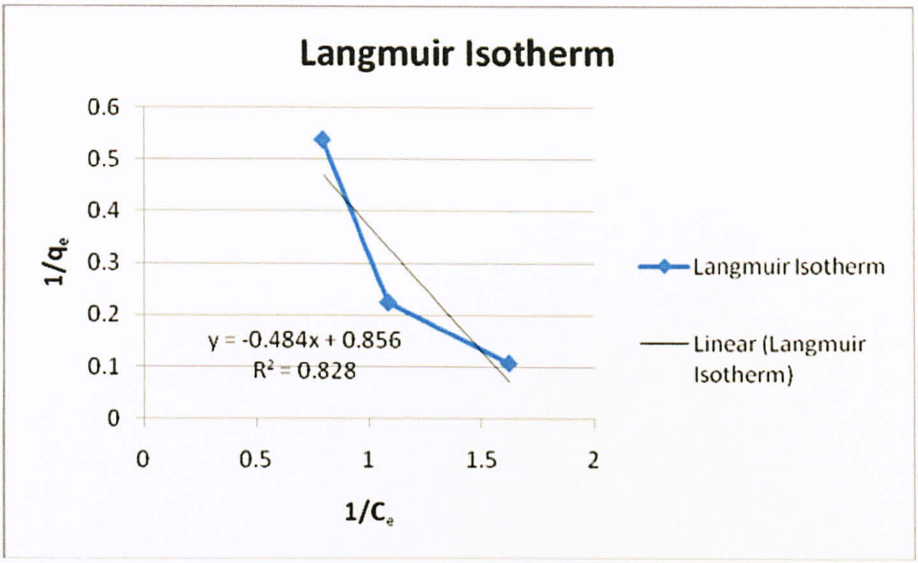


Figure 4.11: Langmuir isotherm plot for coconut coir AC as adsorbent (initial Pb^{2+}= 5, 10 and 20 ppm; amount of AC= 0.2g; shaker speed= 120 rpm)

Table 4.1: Langmuir and Freundlich isotherm constants for Pb onto coconut coir at room temperature (25 °C)

Freundlich constants			Langmuir constants		
k_f	1/n	R^2	q_t	k_a	R^2
3.294	-2.245	0.985	1.168	-1.769	0.828

4.3.2 Effect of Initial Metal Ion Concentration

From those plots, it is found that the amount of adsorption increases with increasing contact time at each initial metal ions concentration and equilibrium is attained within about 165 min for the systems. Further, it was observed that the amount of metal ion uptake, q_t is increased with increase in initial metal ion concentration. At lower concentration, lead ion in the solution would interact with the binding sites and thus facilitated almost 100% adsorption while at higher concentration, more lead are left un-adsorbed in the solution due to saturation of the binding sites.

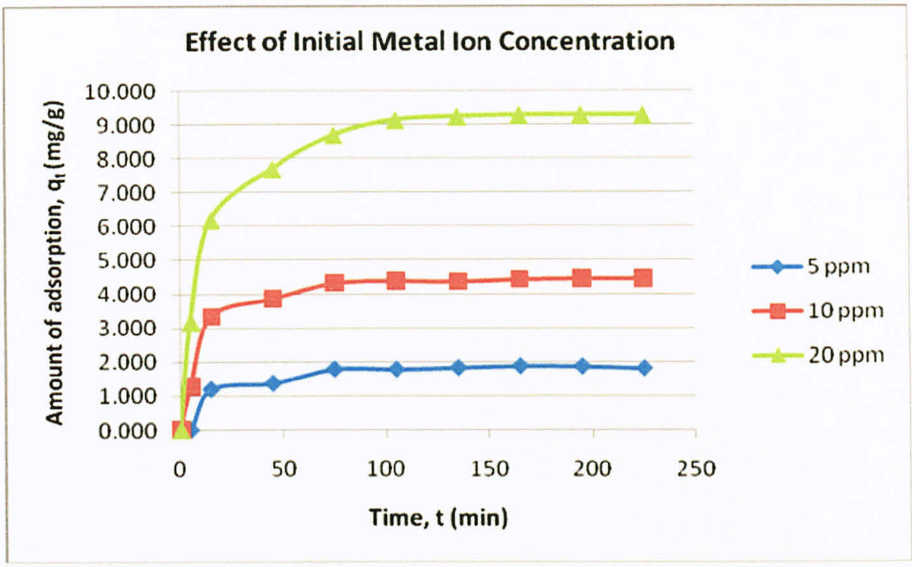


Figure 4.12: Effect of Initial Metal Ion Concentration

4.3.3 Effect of pH

The pH of aqueous solution is an important controlling parameter in the adsorption process. Figure 4.12 shows the effect of pH on amount of metal ion adsorbed, q_t (mg/g), where q_t was found from the mass balance equation which is given by

$$q_t = \frac{(C_o - C_t)V}{m}$$

where C_o (ppm) and C_t (ppm) are the concentration in the solution at time t=0 and at time t, V is the volume of the solution (L) and m is the amount of adsorbent (g) added. Figure 4.12 also shows that Pb adsorption reaches the highest point at pH of 6. It means that the removal of metal ions was found to increase when the solution pH was increased from 3.0 to 6.0 for the system. This phenomenon can be explained by the surface charge of the adsorbent and the H^+ ions present in the solution. At low pH, the cations compete with the H^+ ions in the solution for the active sites and therefore lower adsorption. The pH range was chosen as 3–6 in order to avoid metal hydroxides, which has been estimated to occur at pH> 6.5 for $Pb(OH)_2$ (Tarun Kumar Naiya, Ashim Kumar Bhattacharya, Sailendranath Mandal and Sudip Kumar Das, 2009). In addition when pH increases, there is a decrease in positive surface charge (since the deprotonation of the sorbent functional groups could be occur), which results in a lower electrostatic repulsion between the positively charged metal ion and the surface of activated carbon thus favoring adsorption.

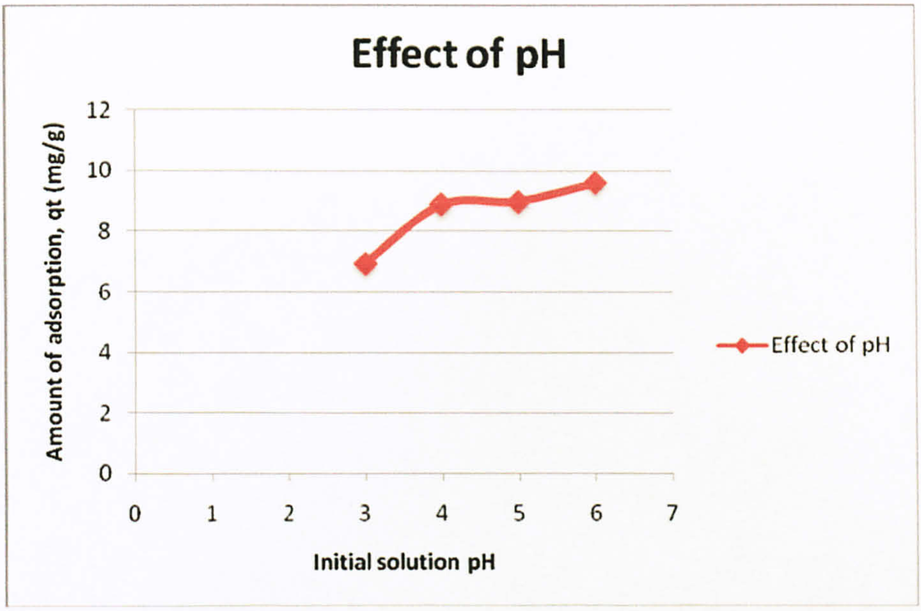


Figure 4.13: Effect of Initial Solution pH

4.3.4 Effect of Adsorbent Dose

The results of the kinetic experiments with varying adsorbent concentrations are presented in Figure 4.13. It shows that Pb^{2+} adsorbed per unit weight of adsorbent decrease as the adsorbent mass increases. This is due to the fact that at higher adsorbent dose the solution ion concentration drops to lower value and the system reaches equilibrium at lower value of q_t indicating the adsorption sites remain unsaturated. Similar observations are also reported by various researchers (M. Sekar, V. Sakthi, S. Rengaraj, 2004) (Tarun Kumar Naiya, Ashim Kumar Bhattacharya, Sailendranath Mandal and Sudip Kumar Das, 2009). From the experimental data, it shows that final concentration of metal solution is increased when the amount of adsorbent used is increased (refer to Appendix A.3). This can be explained because of the systematic error made during the experiment was conducted. The use of rotary mechanical shaker is not suitable for the project and that is the main reason for not getting the expected result. The disadvantage of using rotary mechanical shaker is the solution will not stir thoroughly. It will only stir at the bottom part of the bottle sample. Thus, result in poor data.

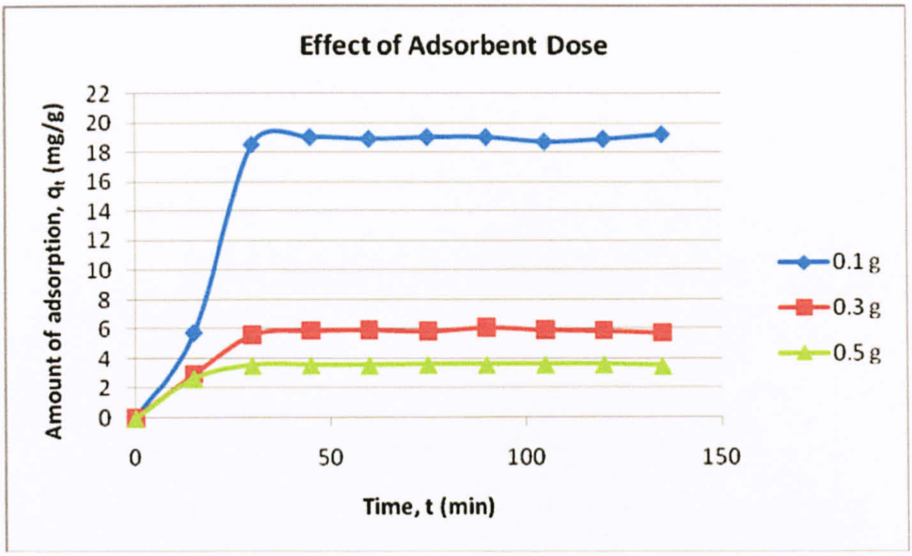


Figure 4.14: Effect of Adsorbent Dose

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The adsorbent used for this project which is activated carbon prepared from coconut coir has potential to remove heavy metal (i.e. Pb(II) ion) in polluted water. The results which had already obtained in this study clearly shows that coconut coir can be considered as a low-cost potential good adsorbent for the removal of lead (Pb^{2+}) ions form aqueous solution. From the characterization of the activated carbon, it is proved that the activated carbon prepared from coconut coir has high adsorption capacity. Batch adsorption kinetic studies show the adsorption of lead metal ions (Pb^{2+}) is strongly affected by initial solution pH, initial metal ion concentration and adsorbent doses. The amount of metal ion (Pb^{2+}) adsorption on activated carbon prepared from coconut coir increases with initial metal ion concentration, contact time and solution pH butt decrease with the amount of adsorbent. It has also found that the amount of adsorption increases with increasing contact time at all initial metal ion concentrations. pH is one of the important parameters for metal ion adsorption on coconut coir AC and it has found that lead adsorption increases with increasing pH of the solution. Thus, the objectives of this project are achieved.

5.2 Recommendation for Future Works

This project can be improved for further study. One of them is to vary the chemical used for activation agent of activated carbon at different temperature. These methods can affect the sorption capacity of activated carbon produced. In addition, types of activation for activated carbon can be also changed by comparing the thermal activation, chemical activation or both. The use of rotary mechanical shaker can be replaced with magnetic stirrer to have a better mixture through out the experiments. Isotherm experiment can be conducted by varying the temperature of the metal solution. Then, to verify the work, parallel experiment by using commercial activated carbon should be done in order to compare the validity and capability of the prepared activated carbon for adsorption process. Therefore, those improvements should be carried out for future work to get a better result.

CHAPTER 6

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CHAPTER 7

APPENDICES

APPENDIX A: CALCULATION OF ADSORPTION AMOUNT

$$q_t = \frac{(C_o - C_t)V}{m}$$

Where C_o (mg/L) and C_t are the concentration in the solution at time t=0 and at time, t, V is the volume of solution (L) and m is the amount of adsorbent (g) added

A.1 EFFECT OF INITIAL METAL ION CONCENTRATION

Volume of solution, V = 100 mL = 0.1 L
Amount of adsorbent, m = 0.2 g

Table A1: Effect of Initial Metal Ion Concentration

Time (min)	Initial concentration (ppm)	Sample ID	Solution concentration (ppm)			q_t Adsorption Amount
			C_o	C_t	C_o-C_t	
0	5	1	4.970	4.970	0.000	0.000
5	5	2	4.970	1.577	3.393	1.696
15	5	3	4.970	1.434	3.536	1.768
45	5	4	4.970	1.410	3.560	1.780
75	5	5	4.970	1.422	3.548	1.774
105	5	6	4.970	1.326	3.644	1.822
135	5	7	4.970	1.231	3.739	1.870
165	5	8	4.970	1.255	3.715	1.858
195	5	9	4.970	1.362	3.608	1.804
225	5	10	4.970	1.362	3.608	1.804
0	10	11	9.810	9.810	0.000	0.000
5	10	12	9.810	1.243	8.567	4.284
15	10	13	9.810	1.159	8.651	4.326

Time (min)	Initial concentration (ppm)	Sample ID	Solution concentration (ppm)			q_t Adsorption Amount (mg/g)
			C_o	C_t	C_o-C_t	
45	10	14	9.810	1.099	8.711	4.355
75	10	15	9.810	1.087	8.723	4.361
105	10	16	9.810	1.063	8.747	4.373
135	10	17	9.810	1.099	8.711	4.355
165	10	18	9.810	0.968	8.842	4.421
195	10	19	9.810	0.920	8.890	4.445
225	10	20	9.810	0.920	8.890	4.445
0	20	21	19.170	19.170	0.000	0.000
5	20	22	19.170	12.836	6.334	3.167
15	20	23	19.170	6.825	12.346	6.173
45	20	24	19.170	3.813	15.358	7.679
75	20	25	19.170	1.813	17.369	8.685
105	20	26	19.170	0.917	18.253	9.127
135	20	27	19.170	0.708	18.462	9.231
165	20	28	19.170	0.629	18.541	9.271
195	20	29	19.170	0.617	18.553	9.277
225	20	30	19.170	0.629	18.541	9.271

A.2 EFFECT OF PH

Volume of solution, V = 100 mL = 0.1 L
Amount of adsorbent, m = 0.2 g
Initial Metal Ion Concentration = 20 ppm

Table A2: Effect of pH

pH	Solution concentration (ppm)			q_t Adsorption Amount (mg/g)
	C_o	C_t	C_o-C_t	
3	19.87	5.9896	13.8804	6.9402
4	19.56	1.7858	18.8842	8.8871
5	19.64	1.6368	18.0032	9.0016
6	19.91	0.7158	19.1942	9.5971

A.3 EFFECT OF ADSORBENT DOSE

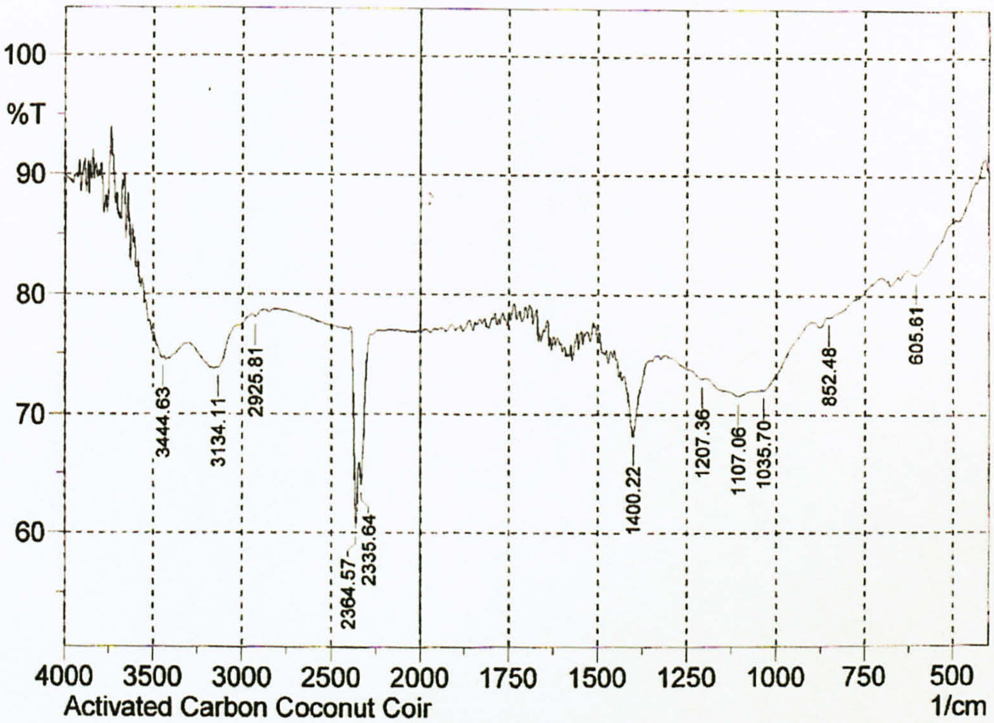
Volume of solution, V = 100 mL = 0.1 L
Initial Metal Ion Concentration = 20 ppm

Table A3: Effect of Adsorbent Dose

Adsorbent Amount (g)	Time (min)	Sample ID	Solution concentration (ppm)			q_t Adsorption Amount (mg/g)
			C_o	C_t	C_o-C_t	
0.1	0	1	19.7675	19.7675	0	0
	15	2	19.7675	13.9913	5.7762	5.7762
	30	3	19.7675	1.2382	18.5293	18.5293
	45	4	19.7675	0.7429	19.0246	19.0246
	60	5	19.7675	0.8667	18.9008	18.9008
	75	6	19.7675	0.7429	19.0246	19.0246
	90	7	19.7675	0.7429	19.0246	19.0246
	105	8	19.7675	1.0524	18.7151	18.7151
	120	9	19.7675	0.8667	18.9008	18.9008
	135	10	19.7675	0.5572	19.2103	19.2103
0.3	0	11	19.2534	19.2534	0	0
	15	12	19.2534	4.3336	14.9198	4.9733
	30	13	19.2534	2.4144	16.8390	5.6130
	45	14	19.2534	1.6096	17.6438	5.8813
	60	15	19.2534	1.4858	17.7676	5.9225
	75	16	19.2534	1.7334	17.5202	5.8400
	90	17	19.2534	0.9905	18.2629	6.0876
	105	18	19.2534	1.4858	17.7676	5.9225
	120	19	19.2534	1.6715	17.5819	5.8606
	135	20	19.2534	2.1049	17.1285	5.7162
0.5	0	21	19.8753	19.8753	0	0
	15	22	19.8753	2.5382	17.3371	3.4674
	30	23	19.8753	2.1049	17.7704	3.5541
	45	24	19.8753	2.0430	17.8323	3.5665
	60	25	19.8753	2.1049	17.7704	3.5541
	75	26	19.8753	1.8572	18.0181	3.6036
	90	27	19.8753	1.8572	18.0181	3.6036
	105	28	19.8753	1.8572	18.0181	3.6036
	120	29	19.8753	1.7953	18.0800	3.6160
	135	30	19.8753	2.2906	17.5847	3.5169

APPENDIX B: CHARACTERIZATION OF ACTIVATED CARBON

B.1 INTERPRETATION OF FTIR DATA



No.	Peak	Height	Corr. Height	Base (H)	Base (L)	Area	Corr. Area	
1	605.61	18.2653	1.1546	628.75	491.81	10.6209	0.471	
2	852.48	21.8088	0.0554	854.41	705.9	14.6326	0.1432	
3	1035.7	27.9647	0.3887	1045.35	898.77	18.4717	0.1715	
4	1107.06	28.4795	0.4275	1130.21	1070.42	8.6219	0.0762	
5	1207.36	27.029	0.2599	1305.72	1197.71	14.2195	0.0875	
6	1400.22	31.8527	5.4843	1423.37	1361.65	9.0511	0.9066	
7	2335.64	35.992	3.6014	2345.28	2281.63	9.5298	0.4022	
8	2364.57	39.666	10.0417	2391.57	2347.21	7.7443	1.1949	
9	2925.81	21.9325	0.3036	2943.17	2877.6	6.9424	0.0409	
10	3134.11	26.1909	0.2178	3139.9	3033.82	12.8844	0.0864	
11	3444.63	25.3896	0.4433	3496.7	3436.91	7.372	0.1597	

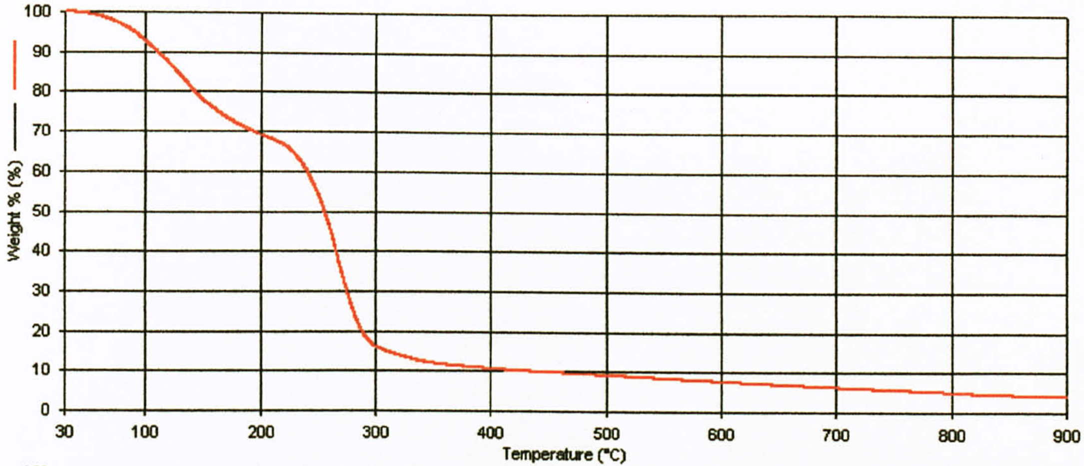
Table B1: Interpretation of FTIR

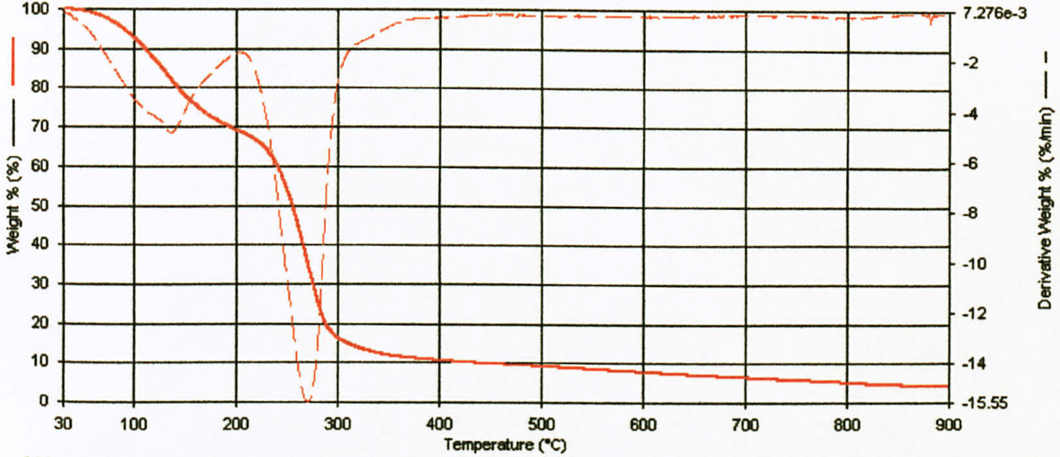
CHARACTERISTIC INFRARED ABSORPTION FREQUENCIES		
Bond	**Compound Type**	**Frequency range, cm^{-1}**
C-H	Alkanes	2960-2850(s) stretch
		1470-1350(v) scissoring and bending
	CH_3 Umbrella Deformation	1380(m-w) - Doublet - isopropyl, *t*-butyl
C-H	Alkenes	3080-3020(m) stretch
		1000-675(s) bend
C-H	Aromatic Rings	3100-3000(m) stretch
	Phenyl Ring Substitution Bands	870-675(s) bend
	Phenyl Ring Substitution Overtones	2000-1600(w) - fingerprint region
C-H	Alkynes	3333-3267(s) stretch
		700-610(b) bend
C=C	Alkenes	1680-1640(m,w)) stretch
C≡C	Alkynes	2260-2100(w,sh) stretch
C=C	Aromatic Rings	1600, 1500(w) stretch
C-O	Alcohols, Ethers, Carboxylic acids, Esters	1260-1000(s) stretch
C=O	Aldehydes, Ketones, Carboxylic acids, Esters	1760-1670(s) stretch
O-H	Monomeric -- Alcohols, Phenols	3640-3160(s,br) stretch
	Hydrogen-bonded -- Alcohols, Phenols	3600-3200(b) stretch
	Carboxylic acids	3000-2500(b) stretch
N-H	Amines	3500-3300(m) stretch
		1650-1580 (m) bend
C-N	Amines	1340-1020(m) stretch
C≡N	Nitriles	2260-2220(v) stretch
NO_2	Nitro Compounds	1660-1500(s) asymmetrical stretch
		1390-1260(s) symmetrical stretch

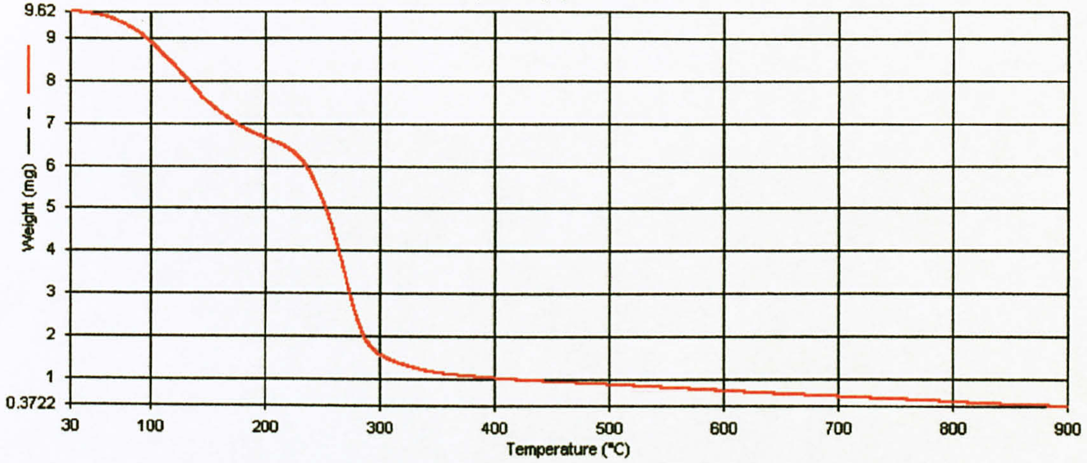
v - variable, m - medium, s - strong, br - broad, w - weak

B.2 INTERPRETATION OF TGA DATA

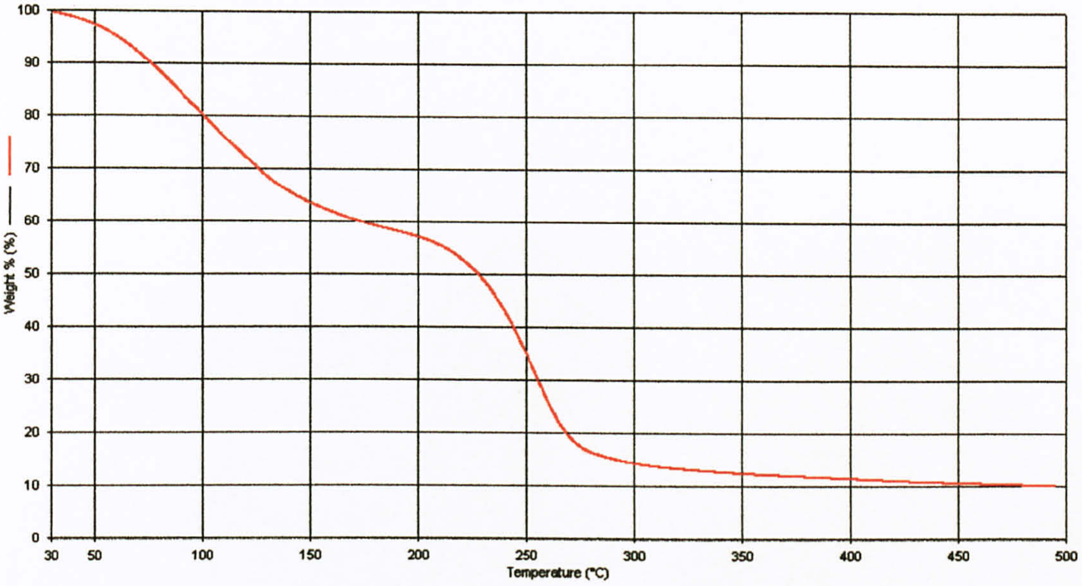
B.2.1 Heating rate: 15 ^{o}C

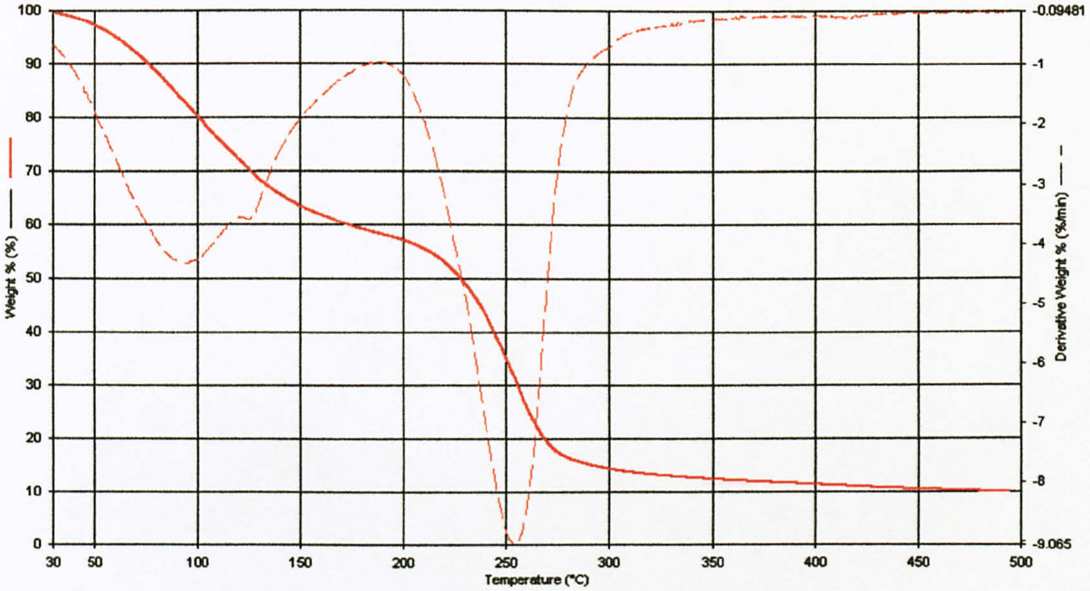






B.2.2 Heating rate: 10 °C





B.2.3 Heating rate: 5 °C