SIMULTANEOUS REMOVAL OF COPPER AND LEAD FROM AN AQUEOUS SOLUTION USING LOW-COST ADSORBENTS

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

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APRIL 2010

I declare that this thesis entitled "*Simultaneous Removal of Copper and Lead from an Aqueous Solution using Low-cost Adsorbents*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature	:	……………………....
Name	:	Mohd Amin Bin Adrah
Date	:	……………………....

To my beloved parents and siblings

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ABSTRACT

The presence of heavy metals in waste water is known to cause severe damage to aquatic life, beside the fact that these metals kill microorganism during biological treatment of waste water with a consequent time delay of the treatment process. Hence this paper aims to ascertain the feasibility of using low-cost adsorbents for simultaneous removal of copper and lead from an aqueous solution. In this work, sawdust and peanut shells were used as low-cost adsorbents, which are abundant in nature, or are a by-product or waste material from another industry. The experiments were conducted using batch adsorption method. The effect of contact time, agitation rate and pH on the simultaneous removal of copper and lead were studied. The experimental results concluded that the better adsorption efficiency was obtained only after 60 minutes of contact time for both the adsorbents. Furthermore, the adsorption of copper and lead increased with increase in agitation rate for sawdust, but for peanut shells the effect of agitation rate was negligible. The maximum removal of copper and lead on sawdust was achieved when pH is 6, whereas for peanut shells when pH is 3.

ABSTRAK

Kewujudan logam berat dalam air sisa diketahui menyebabkan kerosakan teruk kepada kehidupan air, di samping fakta tersebut logam berat turut membunuh mikroorganisma semasa rawatan biologi air sisa yang menyebabkan penangguhan masa proses rawatan. Oleh sebab itu, makalah ini bertujuan untuk memastikan kesesuaian menggunakan penjerap kos rendah untuk menjerap secara serentak kuprum dan plumbum dari larutan akueus. Dalam kajian ini, serbuk kayu dan kulit kacang digunakan sebagai penjerap kos rendah, yang mempunyai sumber yang amat banyak di alam semula jadi, atau produk sampingan atau bahan sisa dari industri lain. Eksperimen ini dilakukan dengan menggunakan kaedah jerapan kelompok. Kesan jumlah masa berhubungan, kadar agitasi dan pH pada penjerapan serentak kuprum dan plumbum dikaji. Keputusan eksperimen menyimpulkan bahawa kecekapan jerapan yang lebih baik diperolehi hanya selepas 60 minit masa pertemuan untuk kedua-dua adsorben. Tambahan lagi, jerapan kuprum dan plumbum meningkat dengan meningkatnya kadar agitasi untuk serbuk kayu, tetapi untuk kulit kacang pengaruh kadar agitasi boleh diabaikan. Penjerapan maksimum kuprum dan plumbum untuk serbuk kayu adalah pada ketika pH 6, sedangkan untuk kulit kacang apabila pH 3.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Heavy metal removal from industrial wastewater is currently an important environmental concern. The removal of heavy metal ions from wastewater is a real challenge due to their toxic effect even at very low concentrations. The presence of these metals such as copper, lead, cadmium, nickel and chromium in improperly treated waste is of serious concerns to the aquatic life and ecological systems. Potential sources of these are mining industries, metal plating and fabrication, fertilizer, illegal landfills, and abandoned waste disposal sites (Wan *et al.*, 2010).

Currently, several technologies have been employed for the removal of heavy metals from aqueous solution such as adsorption, ion exchanges, chemical precipitation, and membrane separation (Wang *et al.*, 2008). Among these, adsorption processes have been shown to be effective and economical treatment process, thus many low-cost adsorbents have been investigated (Wan *et al.*, 2010).

The process of adsorption implies the presence of an adsorbent. Adsorbent is a solid that efficiently binds molecules by means of physical attractive forces, ion exchange or chemical binding (Larous *et al.*, 2005) whereas low-cost adsorbent is defined as adsorbent which is abundant in nature, or is a by-product or waste material from another industry (Aydin *et al.*, 2008). At present, a wide variety of materials such as tea waste (Amarasinghe and Williams, 2007), fly ash (Papandreou *et al.*, 2007; Cho *et al.*, 2005), sawdust (Yasemin and Zaki, 2007; Larous *et al.*, 2005; Yu *et al.*, 2000) and peanut shells (Vazquez *et al.*, 2009; Oliveira *et al.*, 2009; Imamoglu and Tekir, 2008) are being used as low-cost alternatives to expensive adsorbents like commercial activated carbon.

1.2 Problem Statement

Copper and lead, in particular, are natural elements predominantly used in chemical process industries (CPI). Copper is a very common substance that occurs intrinsically in the environment and spreads through natural phenomena, which is extensively utilized by electrical industries, in fungicides, and in antifouling paints. There is evidence to suggest that copper may be carcinogenic to humans and can cause damage to a variety of aquatic life (Ng *et al.*, 2002). Among the ionic species of copper, Cu(II) ions can have alarming effects in aqueous solution, attaching easily to organic and inorganic matter based on solution pH (Wan *et al.*, 2010).

Lead ion is introduced into natural waters through various industrial applications such as those from the insecticide, storage battery, and metal plating/finishing industries (Ofomaja *et al.*, 2010). It is a highly toxic cumulative element, causing variety of negative effects on humans and also for aquatic life, even at low dosages. Lead can contaminate the environment from anthropogenic sources as well as by natural geochemical processes (Wan *et al.*, 2010).

The fact that industrial effluents contain several pollutants simultaneously cannot be neglected. But, only little attention has been given to concurrent adsorption of different metals from wastewater. Also, the commercially available activated carbon is expensive (Crini, 2006). Furthermore, there is yet little literature containing a full study of comparisons between adsorbents. This is important because the comparison of adsorption performance depends not only the parameters related to the experimental conditions and the effluent, but also on the analytical method used for the adsorption experiments. Hence, this research aimed to study the simultaneous removal of copper and lead from an aqueous solution by using low-cost adsorbents. In addition, the effect of contact time, agitation rate and pH on the simultaneous removal of copper and lead using sawdust and peanut shells will be studied.

1.3 Objective

The objectives of this research are as follows:

(a) To determine the percentage adsorption of copper and lead from an aqueous solution using low-cost adsorbents namely sawdust and peanut shells.

(b) To study the effect of contact time, agitation rate and pH on the simultaneous removal of copper and lead using sawdust and peanut shells.

1.4 Scope of Research

In order to achieve the objectives of this research, the following scope of research has been identified:

(a) The effect of contact time between the low-cost adsorbent and heavy metals on the percentage adsorption.

(b) The effect of agitation rate on the percentage adsorption.

(c) The effect of pH on the percentage adsorption.

(d) Comparison on the adsorption capacity for two different low-cost adsorbents.

CHAPTER 2

LITERATURE REVIEW

In this chapter, journals and books related to the research topic are reviewed to gain knowledge and idea about the research.

2.1 Heavy Metal

Heavy metals such as chromium, cadmium, zinc, and copper in wastewater are hazardous to the environments (Khazali *et al.*, 2007). The term "heavy metal" does not have a rigorous scientific basis or chemical definition. In observation, this group should preferably have been referred to as "toxic elements". Table 2.1 shows the source of eight most common heavy metals that contribute to their appearance in water ways.

Table 2.1: Source of eight most common heavy metals (Mishra, 2008)

No.	***Source***	***As***	***Cd***	***Cr***	***Cu***	***Pb***	***Hg***	***Ni***	***Zn***
1.	Mining and ore processing	X	X			X	X		X
2.	Metallurgy	X	X	X	X	X	X	X	X
3.	Chemical Industry	X	X	X	X	X	X		X
4.	Alloys					X			
5.	Paint		X	X		X			X
6.	Glass	X				X	X		
7.	Pulp and paper mills			X	X	X	X	X	
8.	Leather	X		X			X		X
9.	Textiles	X	X		X	X	X	X	X
10.	Fertilizers	X	X	X	X	X	X	X	X
11.	Petroleum refining	X	X	X	X	X	X		X
12.	Coal burning	X	X	X	X	X	X	X	

Among the eight heavy metal mentioned in the above table, copper and lead are most commonly found in wastewater from electroplating and metal finishing industries, and have toxic effects on human and environment (Aydin *et al.*, 2008).

2.1.1 Copper

Copper, a chemical element, is the twentieth most abundant element present in the Earth’s crust. Although some pure copper metal is present in nature, commercial copper is obtained by reduction of the copper compounds in ores followed by electrolytic refining (Licker, 2004). Other than that, it is one of the major contaminants emanating from electrical, electroplating and metal finishing industries (Chakravarty *et al*., 2008).

Furthermore, long term exposure to copper may cause mutations in humans. May damage the testes and decrease fertility in both males and females. Repeated exposure can cause shrinking of the lining of the inner nose with watery discharge; liver damage (Pohanish, 2008).

Copper may be toxic if ingested in large amounts and it also participates in vegetable metabolism via two ways: in the synthesis of chlorophyll and in the activity of other enzymes. Even though copper does not exist in chlorophyll, it is indispensable for vegetables and its lack may result in photosynthesis deficiencies and incapacity to produce seeds. It is also a constituent of enzymes responsible for the catalysis of oxidation-reduction reactions. The lack of copper in animal diet may lead to anemia, diarrhea, and nervous disturbances. On the other hand, the excessive ingestion might cause in vomit, cramps, convulsion, and even death (Paulino *et al.*, 2008).

2.1.2 Lead

Lead, a heavy metal, is toxic and has resulted in poisoning of workers from misuse and overexposure. The largest single use of lead is for the manufacture of storage batteries (Licker, 2004).

Long term exposures to lower levels of lead can result in a buildup of lead in the body. Prolonged exposure to high enough levels may result in serious, permanent kidney and brain damage. If the nervous system is affected, usually due to very high exposures, the resulting effects include severe headache, mood and personality changes. Lead is a probable teratogen in humans. Continuous exposure can result in decreased fertility in males and females. Elevated lead exposure of either parent before pregnancy can increase the chances of miscarriage or birth defects (Pohanish, 2008).

On the other hand, lead also classified as a persistent environmental toxic substance and is toxic in even very small amounts, with a toxicity limit is less than 0.05 μg/mL (Paulino *et al.*, 2008).

2.2 Technologies Available for Heavy Metal Removal

Due to the fact that the existence of heavy metal in the wastewater can bring negative effect to human life, it is important to remove this heavy metal from wastewater. Currently, there are several reported methods for the removal of heavy metal from wastewater. These methods include adsorption, ion-exchange, membrane separations, and chemical precipitation. Figure 2.3 shows the process of biosorption summarized by Sud *et al.* (2008).

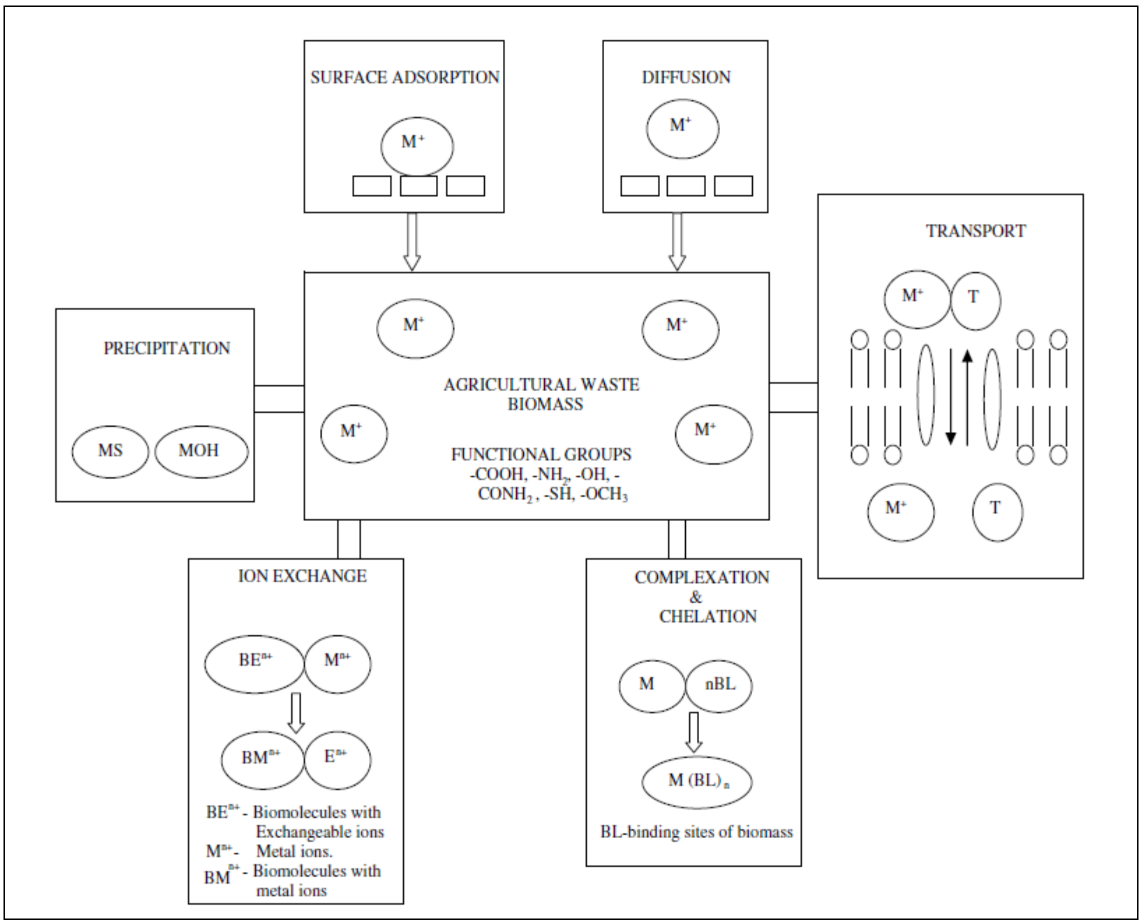

Figure 2.1: Plausible mechanism of biosorption (Sud *et al.*, 2008)

2.2.1 Adsorption

Adsorption can be defined as the binding of molecules on the surface of solid material. This process should not be confused with absorption which is the taking up, usually, of a liquid or gas into the body another material (Porteous, 2000). The solid material is called the adsorbent whereas the molecule that binds on the adsorbent is referred as the adsorbate. The specific locations where the solute usually binds on the surface of the adsorbent are referred as the binding sites or ligands (Ghosh, 2006).

According to Wan Ngah *et al.* (2004), adsorption is the promising alternatives to remove heavy metals ions in trace quantities from an aqueous solution. Furthermore, it is usually the simplest and most cost-effective technique for removal of heavy metals (Wang *et al.*, 2008).

2.2.2 Ion Exchange

Ion exchange is a phenomenon that is reversibly exchanged between counter ion on bead surface and ion in the solution by the difference of electrostatic force. This technology has been reported that it can treat a large volume of solution at once (Eom *et al.*, 2005). Moreover, this process has proven to be very promising in the removal and recovery of heavy metals (Cavaco *et al.*, 2007).

Currently, there are a variety of different types of exchange materials used in this method which may be mineral in nature or synthetic. Such as aluminas, carbons, silicates and synthetic zeolites (Fernandez *et al.*, 2005).

2.2.3 Membrane Separation

Membrane separation processes have been used for the treatment of inorganic effluent to decrease the amount of wastewater produced and to improve the quality of the treated effluent (Barakat and Schmidt, 2010).

Moreover, reuse of water from industrial processes is an important business goal especially when the industry is using large amounts of water. It can be achieved through industrial wastewater treatment by membrane processes (Qdais and Moussa, 2004). Other than that, membrane processes also provide a viable alternative for heavy metal recovery (Landaburu-Aguirre *et al.*, 2006).

2.2.4 Chemical Precipitation

Chemical precipitation is an approach that converted the dissolved metal ions to the insoluble solid phases via a chemical reaction with a precipitant, for example, alkali or sulfide. Then, the resultant precipitates were separated from the water by sedimentation and/or filtration or flotation (Chen *et al.*, 2009).

Chemical precipitation is widely used for wastewater treatment due to its simplicity. But, this process have disadvantages, it requires a large amount of treatment chemicals to decrease the heavy metals to levels imposed by the regulations. Additionally, the sludge produced from the precipitation process has to be subjected to dewatering and disposal into landfills, which adds an additional cost to the treatment process (Qdais and Moussa, 2004).

Among the numerous technologies have been discussed previously, adsorption processes have been shown to be effective and economical treatment for the removal of heavy metal from wastewater due to the presence of low-cost adsorbents compared to other technologies which are generally expensive (Aydin *et al.*, 2008).

2.3 Low-cost Adsorbents

Most commonly adsorption processes currently use activated carbon as adsorbent to remove heavy metal in wastewater because of its excellent adsorption ability. Although it is a preferred sorbent, its widespread use is restricted due to high cost. In order to decrease the cost of treatment, attempts have been made to find inexpensive alternative adsorbents also known as low-cost adsorbents. A low-cost adsorbent is defined as one which is abundant in nature, or is a by-product or waste material from another industry (Aydin *et al.*, 2008). Table 2.2 listed examples of low-cost adsorbent used in wastewater treatment.

Table 2.2: Recent reported adsorption capacities for low-cost adsorbents

Low-Cost Adsorbent	Heavy Metal	Adsorption Capacities (mg/g)	Sources
Mansonia sawdust	Copper Lead	20.20 39.50	Ofomaja *et al.* (2010)
Chitosan-coated sand	Copper Lead	8.18 12.32	Wan *et al.* (2010)
Lentil shell Wheat shell Rice shell	Copper	8.98 7.39 1.85	Aydin *et al.* (2008)
Fly ash	Copper Lead	7.00 18.00	Wang *et al.* (2008)
Hazelnut husks	Copper Lead	6.65 13.05	Imamoglu and Tekir (2008)

2.3.1 Sawdust

Raw agricultural solid wastes and waste materials from forest industries such as sawdust have been used as adsorbents. These materials are available in large quantities and are often present a disposal problem (Yu *et al.,* 2000). It may have potential as sorbents due to their physico-chemical characteristics and low-cost. This will open a new market for the sawdust.

Sawdust is an abundant by-product of the wood industry that is either used as cooking fuel or as packing material. Sawdust is easily available at zero or negligible price. It contains various organic compounds such as lignin, cellulose and hemicellulose. Sawdust has proven to be a promising effective material for the removal of heavy metal from wastewaters (Ofomaja *et al.*, 2010; Larous *et al.*, 2005; Yu *et al.*, 2000). The sorption mechanisms can be explained by the presence of several interactions, such as complexation, ion-exchange due to surface ionization, and hydrogen bonds. Table 2.3 shows reported research on heavy metal removal using sawdust as adsorbent.

Table 2.3: Reported research on heavy metal removal using sawdust as adsorbent

Journal Title	Adsorbent Preparation Method	Results	Source
The removal of heavy metals from aqueous solutions by sawdust adsorption – removal of lead and comparison of its adsorption with copper.	Sawdust was used directly without any treatment.	Optimum pH for lead removal is 5 whereas for copper is pH 7. Adsorption capacity is 1.79 mg Cu/g and 3.19 mg Pb/g.	Yu *et al.* (2001)
Competitive modeling for the biosorptive removal of copper and lead ions from aqueous solution by Mansonia wood sawdust.	Mansonia sawdust was washed several times with distilled water, and followed by drying at 100 °C for 24 hours. The sawdust was ground and sieved (150 – 400 µm).	Optimum pH for both copper and lead removal is 6. Adsorption capacity is 20.2 mg Cu/g and 39.5 mg Pb/g.	Ofomaja *et al.* (2010)

2.3.2 Peanut Shells

Peanut shells can be assumed to be low-cost adsorbents since they are abundant in nature, inexpensive, require little processing and are effective materials. These waste materials have little or no economic value and often present a disposal problem. Therefore, there is a need to valorize these low-cost by-products as a potential inexpensive alternative to the existing commercial adsorbent for wastewater treatment.

According to Wilson *et al.* (2006), peanut shells are low in density and high in volume, and are used in animal feed or burned for energy. Peanut shells have been reported to be promising effective material for the removal of heavy metal from wastewater (Imamoglu and Tekir, 2008; Oliviera *et al.*, 2009; Brown *et al.*, 2000). Table 2.4 shows reported research on heavy metal removal using peanut shells as adsorbent.

Table 2.4: Reported research on heavy metal removal using peanut shells as adsorbent

Journal Title	Adsorbent Preparation Method	Results	Source
Copper and lead removal by peanut hulls: Equilibrium and kinetic studies.	The peanut hulls were ground and sieved.	Optimum pH for copper and lead removal is in the range from 4 to 4.5. Adsorption capacity is 0.22 mmol Cu/g and 0.20 mmol Pb/g.	Oliveira *et al.* (2009)
Removal of copper (II) and lead (II) ions from aqueous solutions by adsorption on activated carbon from a new precursor hazelnut husks.	Activated carbon was prepared from hazelnut husks with zinc chloride activation at 973 K in nitrogen atmosphere.	Optimum pH for copper and lead removal is in the range from 6.7 to 7. Adsorption capacity is 6.65 mg Cu/g and 13.05 mg Pb/g.	Imamoglu and Tekir (2008)

CHAPTER 3

RESEARCH METHODOLOGY

Research methodology is a set of procedures or methods used to conduct research. This chapter describes the research methods used to study the simultaneous removal of copper and lead from an aqueous solution using low-cost adsorbents.

3.1 Overall Methodology

The overall methodology used in this research was shown in Figure 3.1. In order to start this research, literatures related to the research title are reviewed. From this literature review, the best low-cost adsorbents were selected. Subsequently, the adsorbents were prepared. Next, the aqueous solutions containing copper and lead were prepared. Then, the batch adsorption experiments were done by using the low-cost adsorbents and aqueous solution that has been prepared before. The effect of contact time, agitation rate and pH were studied in this research. After the experiments were done, the concentration of metal ions presence in the supernatant was analyzed by using Atomic Absorption Spectrophotometer. Then, the results obtained were discussed. Finally, the conclusion was made based on the results.

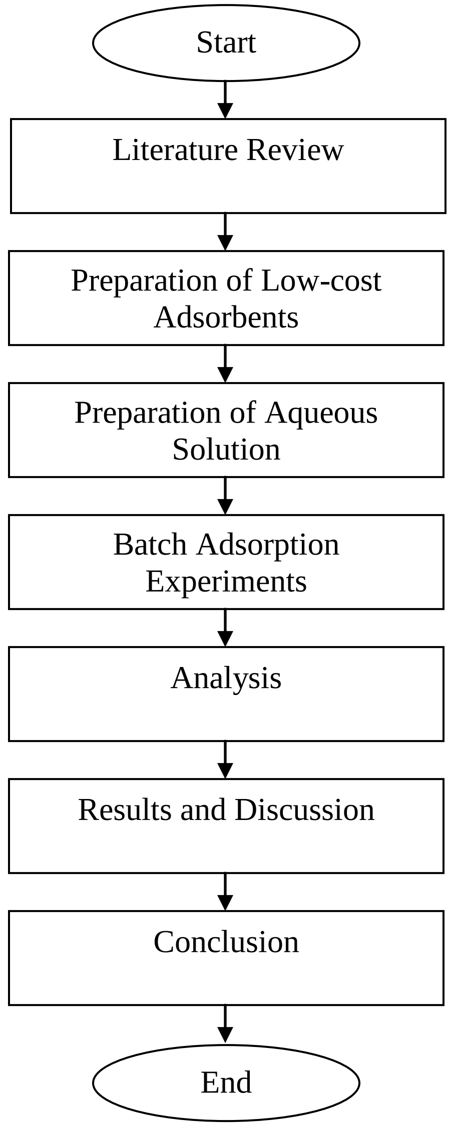

Figure 3.1: Overall methodology

3.2 Materials

Sodium hydroxide; hydrochloric acid, fuming 37%; AAS Standard solution, 1000 mg/L copper and lead were purchased from Merck (M) Sdn Bhd. Copper sulphate; and lead nitrate were purchased from Sigma-Aldrich (M) Sdn Bhd.

3.3 Instrumentations

Z-5000 Polarized Zeeman Atomic Absorption Spectrophotometer operating with an air-acetylene flame has been used for the determination of copper and lead concentrations in aqueous solutions. The specifications of the Z-5000 Polarized Zeeman Atomic Absorption Spectrophotometer are as follows:

Spectrophotometric section:

(a) Measurement mode:	Atomic absorption and flame
(b) Measurement method:	Double beam spectrophotometry
(c) Background correction:	Polarizing Zeeman-effect method
(d) Range of wavelength:	190 – 900 nm

Light source section:

(a) Numbers of installed lamp:	8 lamps
(b) Simultaneous lighting:	2 lamps
(c) Lamp position setting:	Automatic position setting

Flame section:

(a) Ignition system:	Pilot flame ignition (automatic)
(b) Burning condition setting:	Controlled by the mass flow controller

Fourier Transform Infrared Spectroscopy (FTIR) has been used to identify mainly organic materials exists in the sawdust and peanut shells. The specifications of the FTIR are as follows:

Model:	Thermo Nicolet, Avatar 370
Spectral range:	4000 – 400 cm^{-1}
Resolution:	0.9 cm^{-1}
DTGS detector:	7800 – 350 cm^{-1}
Mass of samples:	About 5 mg

The pH measurements have been performed with Metrohm 827 pH Lab meter. The meter was standardized using buffer solutions with pH values 4.00, 7.00 and 10.00. The technical specifications of the Metrohm 827 pH Lab meter are as follows:

Measuring ranges:	(a) pH	0 – 14
	(b) Potential	U ± 1200 mV
	(c) Temperature	-150.0 °C – 250.0 °C (Pt 1000)
		-5.0°C – 250.0 °C (NTC)
Resolution:	∆ pH 0.001	
	∆ U 0.1 mV	
	∆ T 0.1 °C	
Power:	100 V/ 115 V/ 220 V, 50 Hz/ 60 Hz	

All filtrations during this research have been carried out by the means of the glass microfiber filter 0.45 micron – Sartorius Stedim Biotech.

3.4 Preparation of Adsorbents

The low-cost adsorbents were prepared by referring to the simplest method used by other investigators in their research. The similar low-cost adsorbents were used throughout this research.

3.4.1 Sawdust

Sawdust obtained from Seng Peng Sawmills Sdn. Bhd., Kuantan was sieved to the desired size fractions, 200 microns, and it was used directly for adsorption experiments without any treatment.

3.4.2 Peanut Shells

Peanut shells obtained from a local market were ground and sieved to the desired size fractions, 200 microns, and it was used directly for adsorption experiments without any treatment.

3.5 Preparation of Aqueous Solution

Copper sulphate ($CuSO_4$) and Lead nitrate ($Pb(NO_3)_2$) salts were used in the preparation of the salt solutions. Stock solutions of 1000 ppm were prepared by dissolving the accurately weighed amounts of $CuSO_4$ and $Pb(NO_3)_2$ in 1000 ml ultrapure water. The aqueous solutions used in this research were prepared by diluting the stock solution with ultrapure water.

3.6 Batch Adsorption Experiments

The simultaneous removal of copper and lead from an aqueous solution by using low-cost adsorbent will be studied by batch technique. The effect of several parameters such as contact time, agitation rate and pH on the adsorption was studied.

3.6.1 Effect of Contact Time

Contacting time is inevitably a fundamental parameter in all transfer phenomena such as adsorption. It is important to study its effect on the capacity of retention of copper and lead by sawdust and peanut shells.

The batch adsorption experiments were carried out by adding 0.5 g of the sawdust with 100 ml of the binary solution which contain 10 ppm of copper and 10 ppm of lead in 250 ml Erlenmeyer flask. The suspension is agitated at 200 rpm in agitation period range 30 – 180 minutes. At the end of the experiments, the sawdust is removed by filtration and a sample of the filtrate is analyzed by means of the atomic absorption spectrophotometer. It should be noted that this experiments were done without any pH adjustment. The same experimental procedure will be repeated by replacing sawdust with peanut shells.

3.6.2 Effect of Agitation Rate

The agitation rate is an important parameter in any transfer phenomena, since it can promote a certain turbulence which insures an intimate contact between the phases, a fact which contributes to the improvement of mass transfer (Larous *et al.*, 2005).

The batch adsorption experiments were carried out by adding 0.5 g of the sawdust with 100 ml of the binary solution which contain 10 ppm of copper and 10 ppm of lead in 250 ml Erlenmeyer flask. The suspension is agitated for 60 minutes in agitation rate range 100 – 400 rpm. At the end of the experiments, the sawdust is removed by filtration and a sample of the filtrate is analyzed by means of the atomic absorption spectrophotometer. It should be noted that this experiments were done without any pH adjustment. The same experimental procedure will be repeated by replacing sawdust with peanut shells.

3.6.3 Effect of pH

The pH of the aqueous solution is an important controlling parameter in the adsorption process and thus, the effect of pH has been studied by varying it in the range of 2 – 6.

The batch adsorption experiments were carried out by adding 0.5 g of the sawdust with 100 ml of the binary solution which contain 10 ppm of copper and 10 ppm of lead in 250 ml Erlenmeyer flask. The suspension is agitated at 200 rpm for 60 minutes. The pH of the binary solution of copper and lead were adjusted to pH range 2 – 6 using either 0.1 M NaOH or 0.1 M HCl. At the end of the experiments, the sawdust is removed by filtration and a sample of the filtrate is analyzed by means of the atomic absorption spectrophotometer. The same experimental procedure will be repeated by replacing sawdust with peanut shells.

3.7 Percentage Adsorption

According to Wan *et al.* (2010), the amount of copper and lead ions adsorbed per unit mass of adsorbents was calculated using the equation:

$$Q = \frac{[C(o) - C(e)]v}{m} \qquad (3.1)$$

where *C(o)* and *C(e)* are the initial and equilibrium concentration of metal ion solution (mg/L) respectively; *v* is the volume of the solution in liter (L); and *m* is the amount of adsorbents in grams (g).

The percentage adsorption was determined using the equation:

$$\%\ Adsorption = \frac{C(o) - C(e)}{C(o)} \times 100 \qquad (3.2)$$

where *C(o)* and *C(e)* are the initial and equilibrium concentration of metal ion solution (mg/L).

CHAPTER 4

RESULTS AND DISCUSSION

This chapter will discuss the results obtained from this research. Furthermore, the results obtained are compared with the results of similar work by other investigators.

4.1 Fourier Transform Infrared Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy Analysis was done to identify material of the low-cost adsorbents which provides information about the chemical bonds and molecular structure (SEM Lab, 2010).

4.1.1 Sawdust

Figure 4.1 shows the FTIR spectrum for sawdust. It displays a number of absorption peaks, indicating the complex nature of the sawdust was examined. The absorption peak 3466.99 cm^{-1} indicates the existence of amine group. The peaks at 2852.81 cm^{-1}, 2218.25 cm^{-1}, and 1035.37 cm^{-1} show the presence of carboxylic acid, nitrile, and esters group respectively. The existence of various functional groups in the sawdust suggests that the acid groups are more than the basic groups and are likely to be the sites for adsorption (Ofomaja *et al.*, 2010).

Table 4.1: Functional groups of sawdust

Functional Groups	Wave Numbers (cm^{-1})
N-H	3466.99
COOH	2852.81
CΞN	2218.25
C-O	1035.37

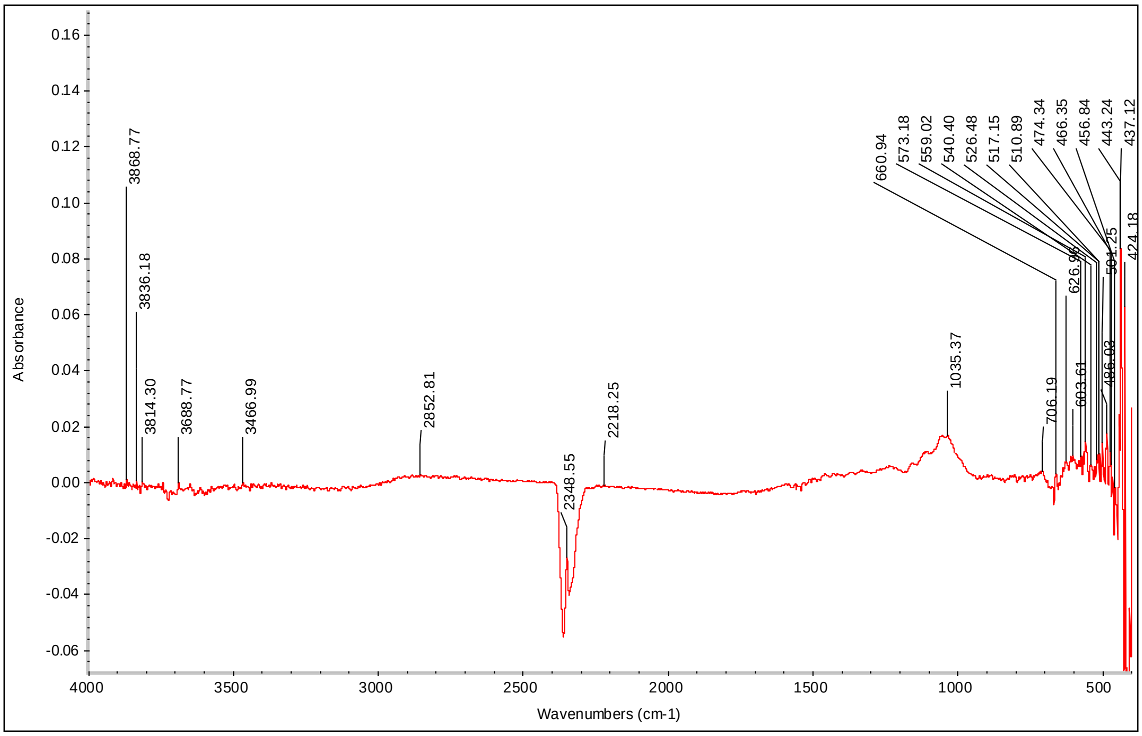

Figure 4.1: FTIR spectrum for sawdust

4.1.2 Peanut Shells

The FTIR spectrum shown in Figure 4.2 indicates the complex nature of the peanut shells that has been examined. It displays a number of absorption peaks such as peak at 3376.95 cm^{-1} which indicates the existence of carboxylic acid group. Moreover, the peaks at 2922.35 cm^{-1}, 1617.99 cm^{-1}, and 1247.26 cm^{-1} together with 1053.14 cm^{-1} show the presence of alkane, amine, and esters group respectively. The occurrence of various functional groups in the peanut shells suggests that the acid groups are more than the basic groups and are possible to be the sites for adsorption (Ofomaja *et al.*, 2010).

Table 4.2: Functional groups of peanut shells

Functional Groups	**Wave Numbers (cm^{-1})**
COOH	3376.95
H-C-H	2922.35
N-H	1617.99
C-O	1247.26, 1053.14

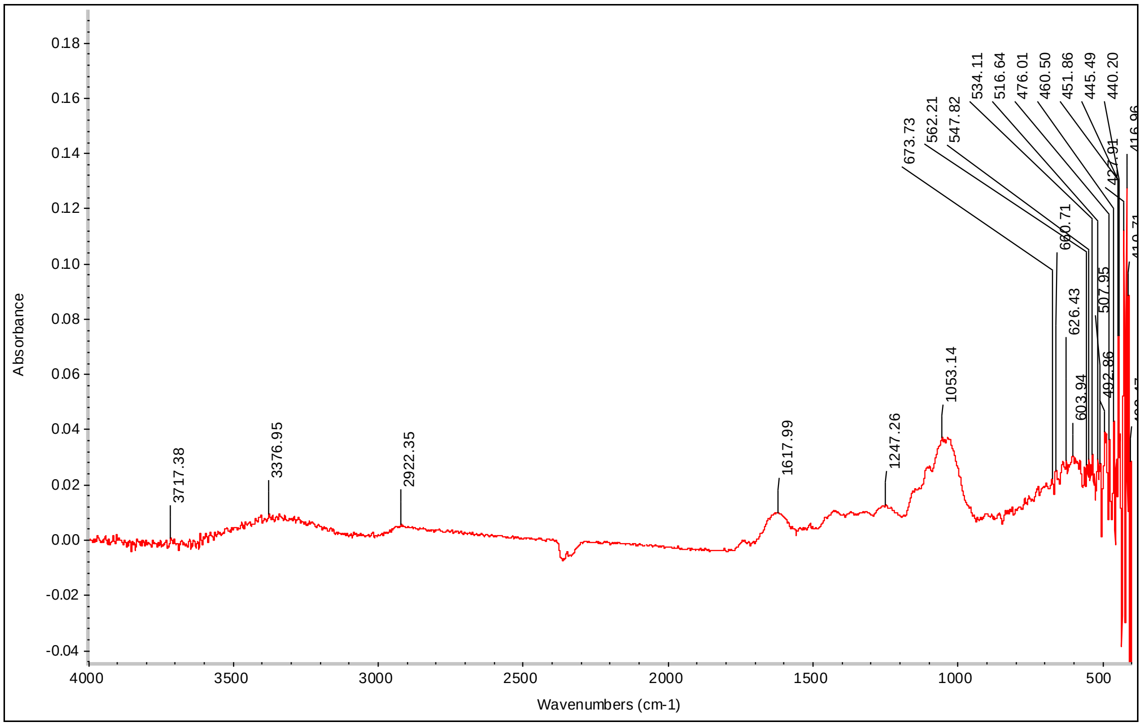

Figure 4.2: FTIR spectrum for peanut shells

4.2 Effect of Contact Time

Effect of contact time on the adsorption of copper and lead ions from an aqueous solution using low-cost adsorbents, sawdust and peanut shells, were discussed in this section.

4.2.1 Sawdust

The effect of contact time on adsorption percentage and adsorption capacity of copper and lead ions were shown in Figure 4.3 and Figure 4.4 respectively. Figure 4.3 and Figure 4.4 demonstrate the typical form of a saturation curve. It was clearly shown that higher adsorption rates were examined at the beginning and the adsorption equilibrium is achieved just after only 60 minutes. This phenomenon probably occurred due to the larger surface area of the sawdust being available at the beginning. But, as the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the metal ions are transported from the exterior to the interior sites of the sawdust (Yu *et al.*, 2000).

Figure 4.3 shows that the highest percentage of adsorption obtained for copper and lead ions were 27.7 % and 13.9 % respectively. In addition, 0.55 mg of copper and 0.28 mg of lead were adsorbed with every 1 g of sawdust used for the adsorption as illustrated in Figure 4.4. The disparity might be due to the molecular size of the metal ions. The larger ionic size of lead ions deterred the molecular array within the sawdust configuration thus eliciting lesser interaction of electrons (Wan *et al.*, 2010).

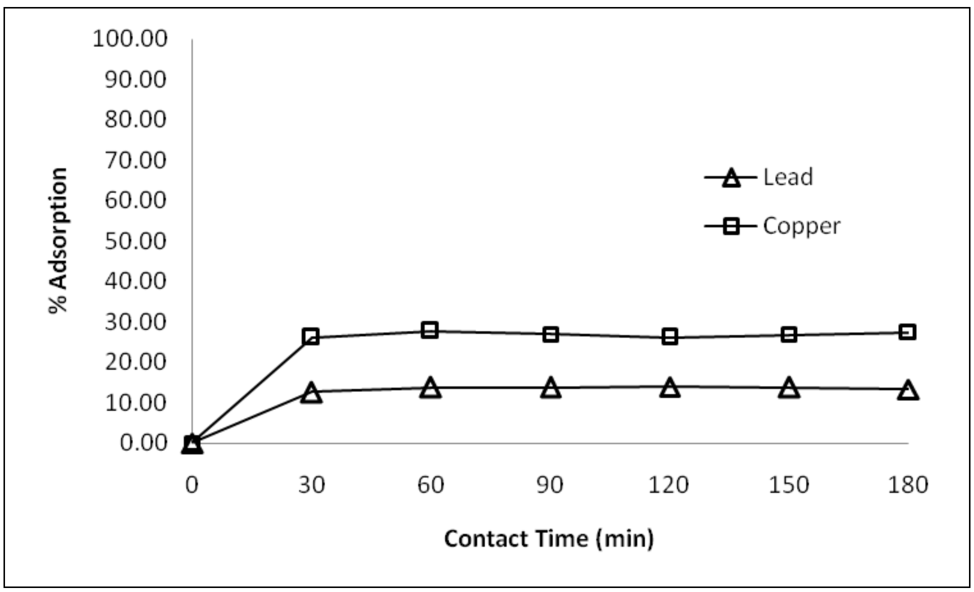

Figure 4.3: Effect of contact time on percentage adsorption using sawdust

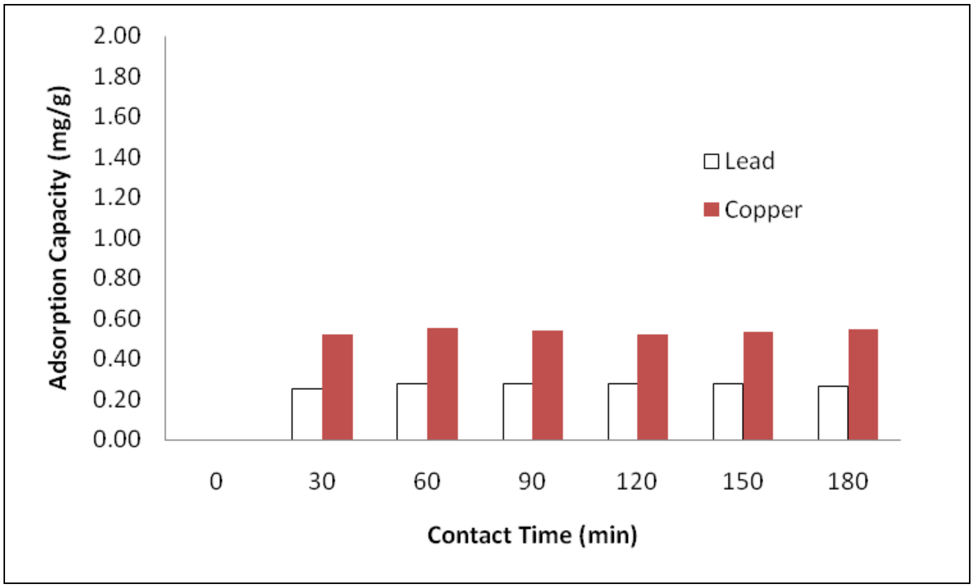

Figure 4.4: Effect of contact time on adsorption capacity using sawdust

4.2.2 Peanut Shells

Effect of contact time on adsorption percentage and adsorption capacity of copper and lead ions by using peanut shells as an adsorbent were shown in Figure 4.5 and 4.6 respectively. The graph shown has the typical form of a saturation curve. It clearly shows that higher adsorption rates were examined at the beginning and the adsorption equilibrium is attained just after only 60 minutes. This phenomenon probably occurred due to the larger surface area of the peanut shells being available at the beginning. But, as the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the metal ions are transported from the exterior to the interior sites of the peanut shells (Yu *et al.*, 2000).

Figure 4.5 demonstrated that the percentage of adsorption of copper ions was lower compare to lead ions which is 20 % for copper and 80 % for lead. Other than that, only 0.4 mg of copper were removed with every 1 g of peanut shells used compare to lead which achieved 1.6 mg removal. The difference might be due to the hardness of the metal ions. Hardness is inversely related to the ability of the molecule to share electrons. It follows that when the hardness is small, the number of shared electrons will be large and vice versa. A lead ion is larger but less hardness than a copper ion. Therefore, more numbers of electrons were shared, which resulted in stronger covalent interactions with the peanut shells (Wan *et al.*, 2010).

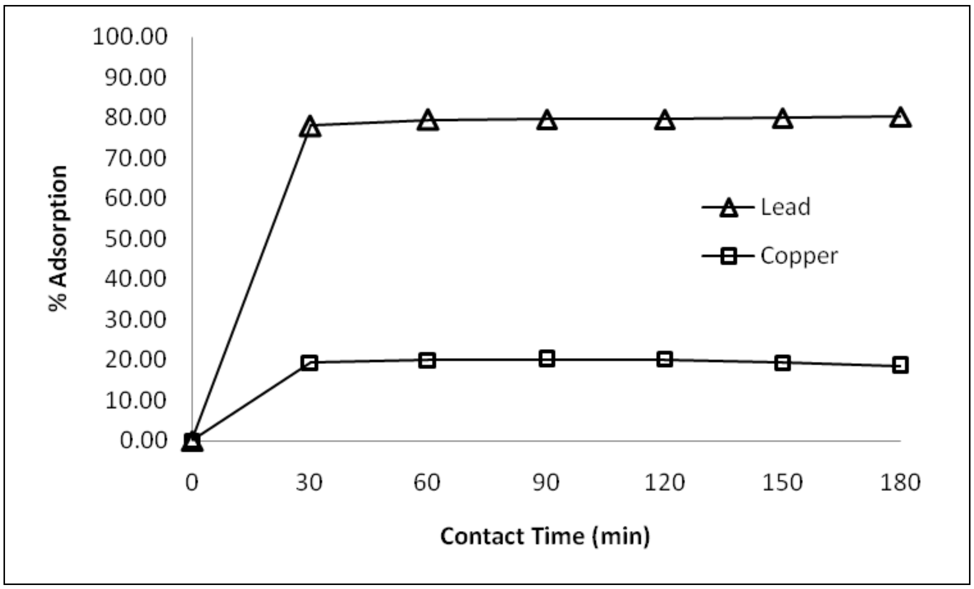

Figure 4.5: Effect of contact time on percentage adsorption using peanut shells

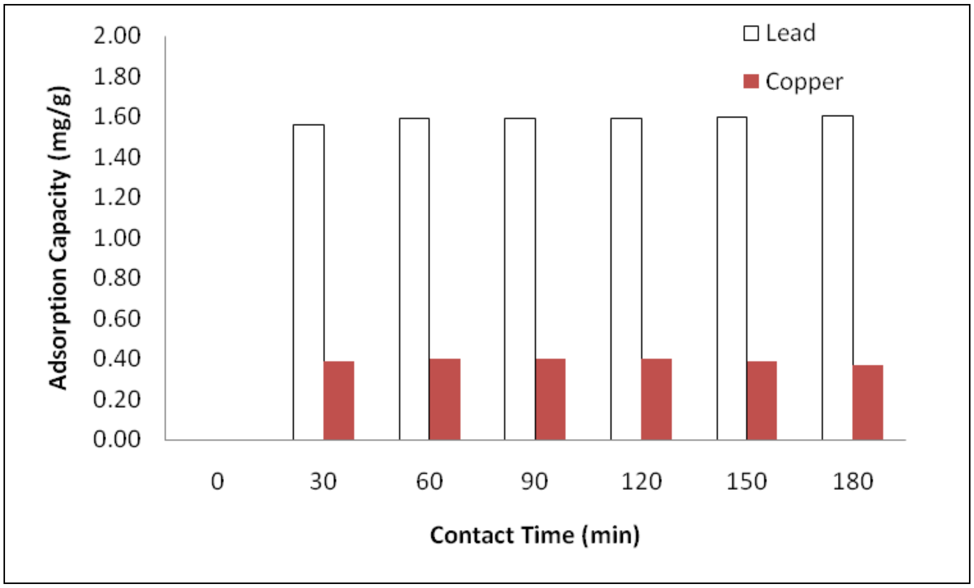

Figure 4.6: Effect of contact time on adsorption capacity using peanut shells

4.3 Effect of Agitation Rate

The effect of agitation rate on the adsorption of copper and lead ions by using low-cost adsorbent, sawdust and peanut shells, were discuss in this section.

4.3.1 Sawdust

Effect of agitation rate on the adsorption of copper and lead ions by using sawdust as a low-cost adsorbent were shown in Figure 4.7 and 4.8. The graph illustrated that the adsorption of copper and lead ions increases with agitation rate. It can be noted that the increase in agitation speed encourages a better transfer of species between the sawdust and the metal ions.

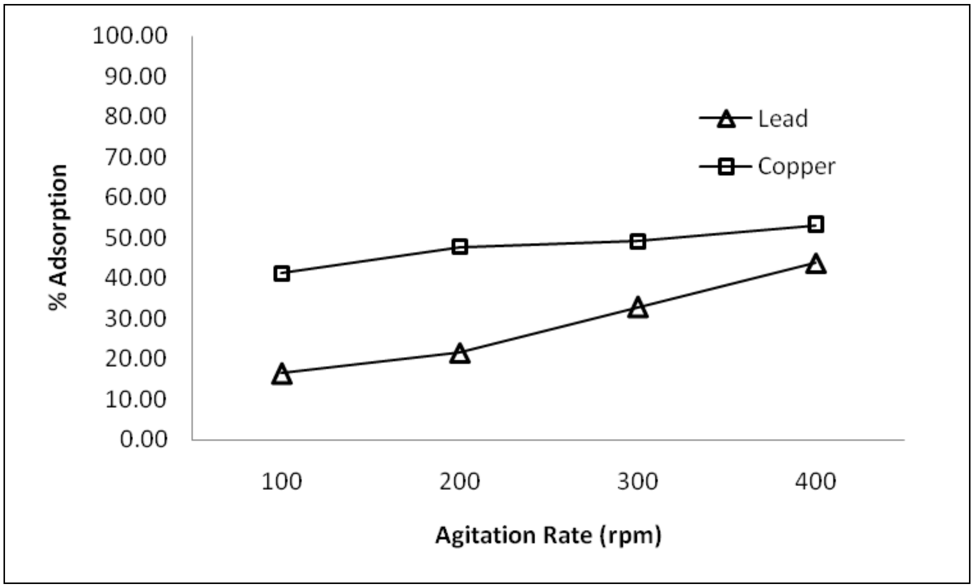

Figure 4.7: Effect of agitation rate percentage adsorption using sawdust

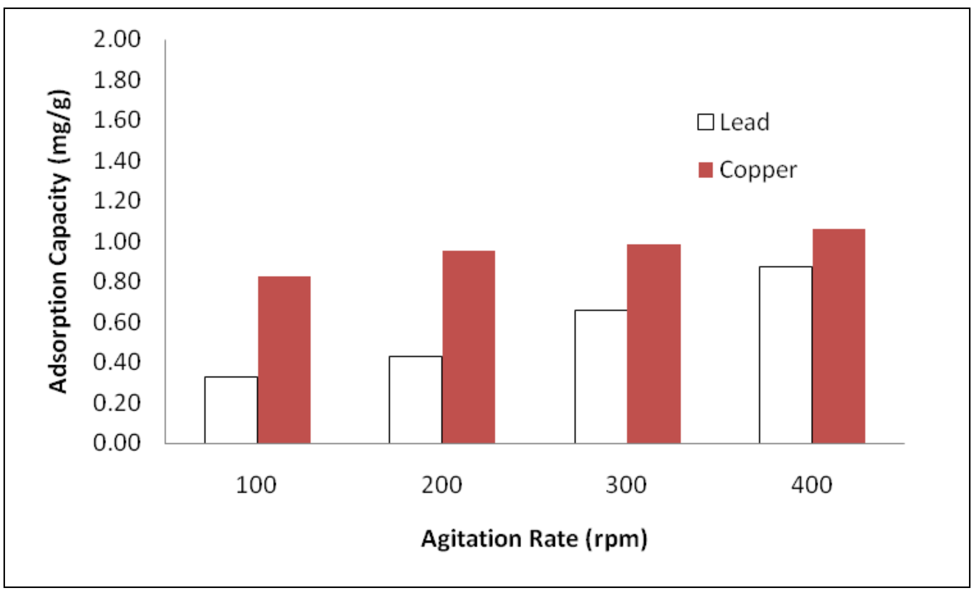

Figure 4.8: Effect of agitation rate on adsorption capacity using sawdust

4.3.2 Peanut Shells

Effect of agitation rate on the adsorption of copper and lead ions by using peanut shells as an adsorbent was shown in Figure 4.9 and 4.10. The graph demonstrated that the agitation rate has little effect on the adsorption of copper and lead ions when peanut shell was used as adsorbent. It was observed that the adsorption of copper and lead ions almost constant, implying equilibrium has been reached. This phenomenon was also obtained by Larous *et al.* (2005) which study the effect of a few agitation rate, 80 rpm, 300 rpm and 500 rpm, on contact time. The result shown that after 20 minutes the adsorption of copper ion for every agitation rate achieved equilibrium, and the adsorption capacity for each agitation rate was around 1.5 mg/g. Thus, can be concluded that after adsorption equilibrium was achieved, the agitation speed have only little effect on the adsorption capacity.

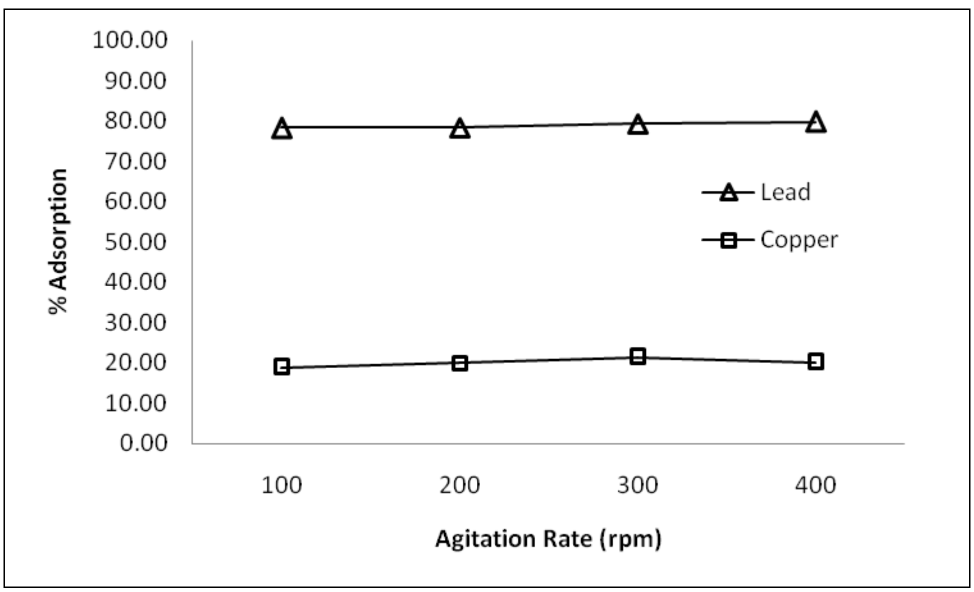

Figure 4.9: Effect of agitation rate on percentage adsorption using peanut shells

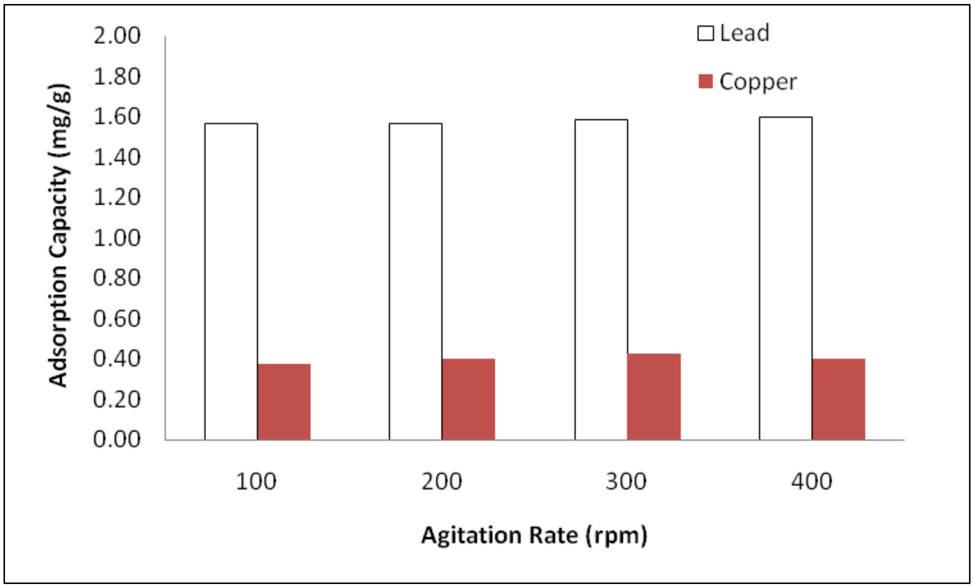

Figure 4.10: Effect of agitation rate on adsorption capacity using peanut shells

4.4 Effect of pH

The effect of pH in the range of 2 to 6 on the adsorption of copper and lead ions by using low-cost adsorbent, sawdust and peanut shells, were discuss in this section. Solution pH greater than 6 was not examined in order to avoid metal ion precipitation (Ofomaja *et al.*, 2010), and ensure the solubility of metal ion. At high pH, precipitation usually occurred with the metallic ions attached to hydroxide ions forming $Cu(OH)_2$ at pH greater than 6 and $Pb(OH)_2$ at pH greater than 8 (Wan *et al.*, 2010).

4.4.1 Sawdust

The effect of pH on the adsorption of copper and lead ions by using sawdust as an adsorbent was shown in Figure 4.11 and 4.12. The graph shown in Figure 4.11 and 4.12 illustrated that at low solution pH, the amount copper and lead ions adsorbed were quite low. The existence of positively charged sites on the sawdust surface may be responsible for the low metal ion uptake at low solution pH. As solution pH increased, the amount of both metal ions adsorbed increased; suggesting hydrogen ion competition with the metal ions for adsorption sites on the surface of the adsorbent (Ofomaja *et al.*, 2010).

At lower pH values (pH = 2), adsorption of copper ions was higher compared to lead ions. Although copper, lead and hydrogen ions were present at this pH value, copper ions were able to compete well for the active sites. This behavior can be explained by two possible mechanisms: (1) the bridging ligand effect, and (2) the adsorption of anionic species (Wan *et al.*, 2010).

In the bridging ligand effect mechanism, there was greater complexion due to the existence of excess chlorine ions produced by the addition of hydrochloric acid when the pH was adjusted, which can be explained by the dominant presence of chlorine ions at pH 2 rather than pH 3. It is known that depending on the chlorine ions concentration, copper can form a different type of complexes using chloride as a bridge. Even though lead ions has similar characteristic, the ionic size of copper ion is smaller than lead ion, which produce the reduction of steric hindrance effect. The presence of the mentioned complexes can generate retention since more than one metal ion occupies each active sites for adsorption (Wan *et al.*, 2010).

Relative to the second mechanism, the adsorption of anionic species emerged at lower pH, since there was also formation of stable sulfates and nitrates from salts of copper and lead ions, correspondingly. The higher charge of sulfates than nitrates may be more effective in charge compensation and ionic bonding. In addition, the chelation of the metal ions was sustained by the stronger binding of sulfates, which resulted in higher adsorption efficiency of copper ions compared to lead ions. In this pH range studied, the stability of the metal complexes associated well with the pH insentivity of the copper ions equilibrium (Wan *et al.*, 2010).

Conversely, the amount of lead ion adsorbed is higher than that of copper ion while the solution pH increased after pH = 2. According to Wan *et al.* (2010), low pH allows high numbers of hydrogen ions to favor the protonation of the amino sites. Thus, the electrostatic repulsion between the lead ions and the protonated amino group at pH = 2 might cause more lead ions to be remove compared to copper ions.

Between solution pH of 5 and 6, the rate of increase in metal ion uptake reduces for both copper and lead ions, therefore solution pH = 6 were chosen as the optimum pH value.

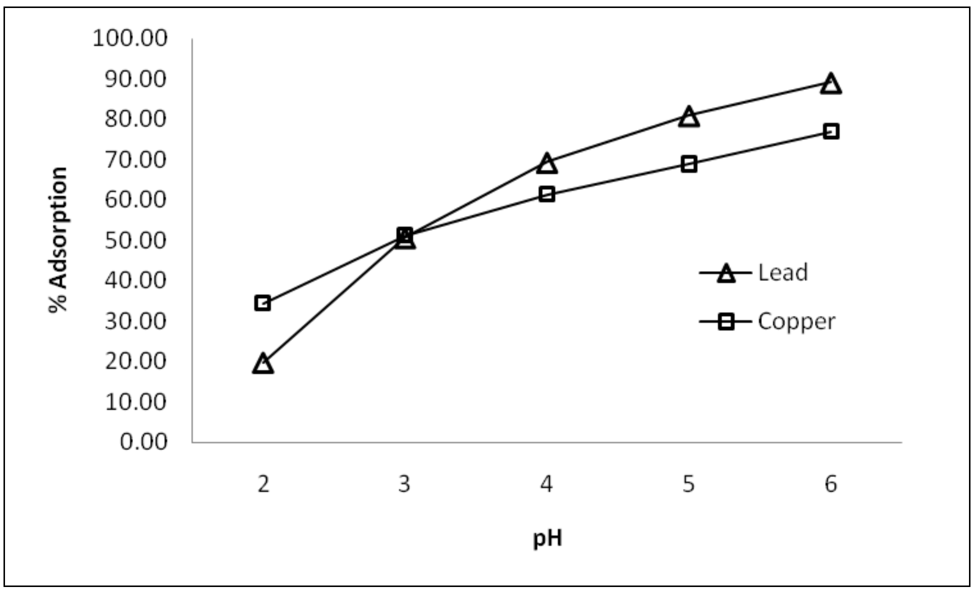

Figure 4.11: Effect of pH on percentage adsorption using sawdust

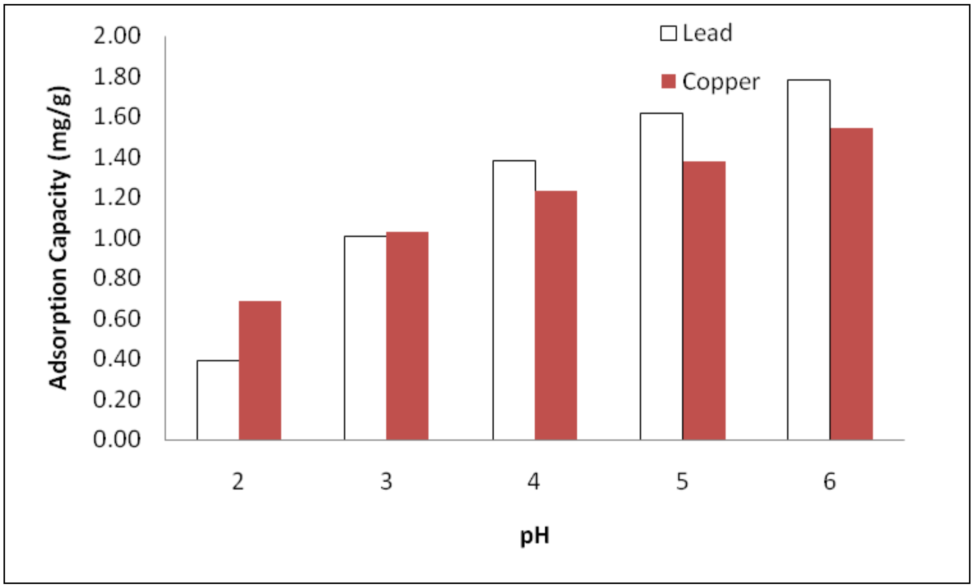

Figure 4.12: Effect of pH on adsorption capacity using sawdust

4.4.2 Peanut Shells

Figure 4.13 and 4.14 show the effect of pH on the adsorption of heavy metal ions by using peanut shells, it can be observed that at pH = 2 the amount of copper and lead ions adsorbed were quite low. The presence of positively charged sites on the peanut shells surface may also be responsible for the low metal ion uptake at low solution pH. The amount of copper and lead ions adsorbed increased between pH 2 and pH 3, after that the rate of adsorption decreases slightly between pH 4 to 6.

This behavior might be explained by two possible reasons, first, the electrostatic attraction seems to be an important mechanism, the presence of sodium chloride on the peanut shells can help to render the surface of peanut shells not easily reachable to copper and lead ions and hence decreasing the adsorption rate due to increase in ionic strength. In fact, when solid adsorbent is in contact with sorbate species in solution, they are bound to be surrounded by an electrical diffused double layer, the thickness of which is significantly expanded by the existence of electrolyte. Such expansion inhibits the adsorbents particles and heavy metal ions from approaching. Second, the relative competition between sodium and heavy metal ions for the active sites of peanut shells, can also be an explaining factor (Larous *et al.*, 2010).

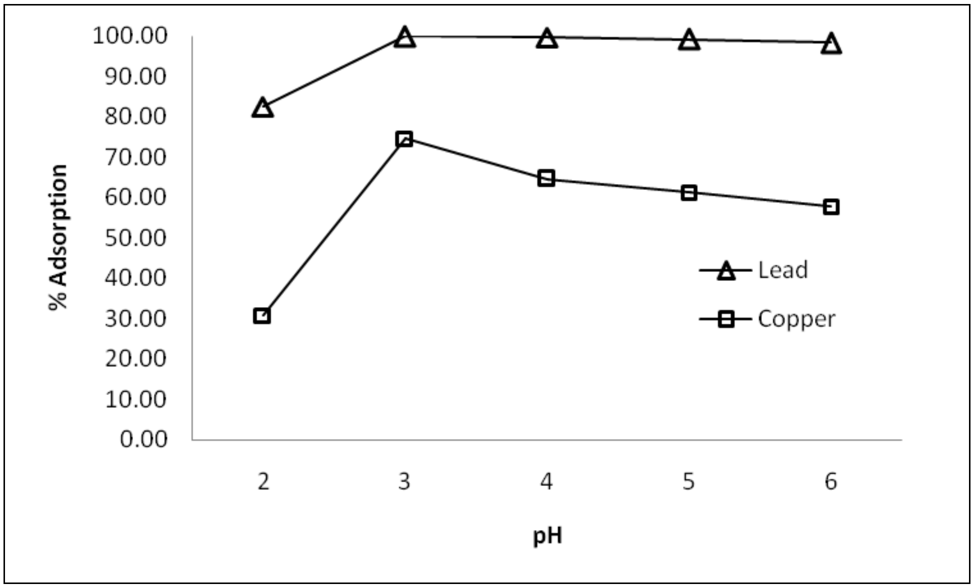

Figure 4.13: Effect of pH on percentage adsorption using peanut shells

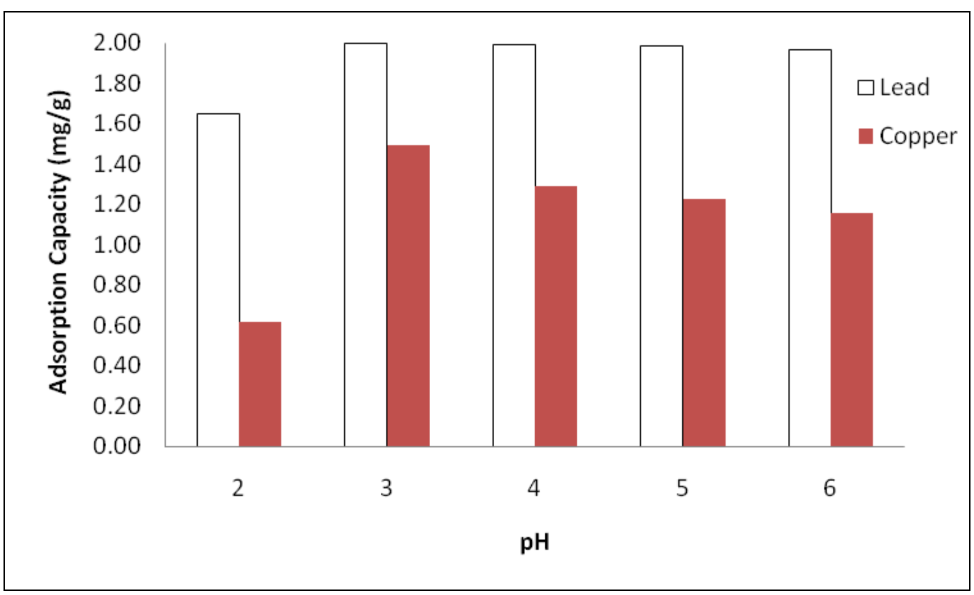

Figure 4.14: Effect of pH on adsorption capacity using peanut shells

4.5 Comparison on Adsorption Capacity

To illustrate the performance of low-cost adsorbents, sawdust and peanut shells, comparison on adsorption capacity is presented in Table 4.3. The result shows that peanut shells adsorbed more lead ion compared to sawdust. For copper ion, sawdust adsorbed more compared to peanut shells but the adsorption capacity is only slightly different. Therefore, peanut shells are a better low-cost adsorbent for the simultaneous removal of copper and lead from an aqueous solution compared to sawdust.

Table 4.3: Comparison on adsorption capacity between sawdust and peanut shells

Adsorbent	**Adsorption Capacity**	
	Copper (mg/g)	**Lead (mg/g)**
Sawdust	1.54	1.78
Peanut Shells	1.49	2.00

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

This chapter will conclude the research that has been done. In addition, recommendation to improve this research in the future also was being discussed.

5.1 Conclusions

Two low-cost adsorbents namely sawdust and peanut shells were used in this research to study the simultaneous removal of copper and lead from an aqueous solution.

The optimum contact time for both low-cost adsorbents for the simultaneous removal of copper and lead from an aqueous solution are achieved only after 60 minutes. Furthermore, the effect of pH on the simultaneous removal of copper and lead from an aqueous solution was studied. The results shown maximum removal of copper and lead on sawdust are at pH = 6 whereas for peanut shells are at pH = 3.

Moreover, the adsorption of copper and lead increased with increase in agitation rate when sawdust was used as a low-cost adsorbent. But, for peanut shells, the agitation rate has little effect on the simultaneous removal of copper and lead from an aqueous solution.

From the results obtained through this research, it can be conclude that sawdust and peanut shells appears to be a promising adsorbent for the simultaneous removal of copper and lead from an aqueous solution.

5.2 Recommendations

Physical and chemical processes such as drying, autoclaving, cross-linking reactions or contacting with organic or inorganic chemicals are proposed to improve the sorption capacity and the selectivity of the low-cost adsorbents.

Various types of low-cost adsorbents such as peat, zeolites, biomass and chitosan can be used for the future research. This is because performance of adsorption process is dependent on the type of material used. Moreover, the applicability of low-cost adsorbents also depends strongly on their origin.

Desorption studies can also be done to illustrate the stability and potential recovery between the adsorbate and adsorbent. Recovery of adsorbed material is important for metal ion recycling processes.

The adsorption isotherms also need to be study in the future research. The design and efficient operation of adsorption processes requires equilibrium adsorption data for use in kinetic and mass transfer models. These models play an important role in predictive modeling for analysis and design of adsorption systems (Crini, 2006).

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