

**The Effectiveness of Activated Surface Kapok on Removal
of Heavy Metal in Wastewater**

by

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15568

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

SEPTEMBER 2015

Universiti Teknologi PETRONAS,
32610, Bandar Seri Iskandar,
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

(AP Ir. Abdul Aziz Omar)

UNIVERSITI TEKNOLOGI PETRONAS
BANDAR SERI ISKANDAR, PERAK

September 2015

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.

NUR ADILAH RAMLI

ABSTRACT

Kapok (*Ceiba pentandra*) fiber is a natural hollow fiber with thin shell and large cavity. It is seldom used as adsorbent for heavy metal ions. In this paper, kapok fibers is chemically modified using Diammonium Hydrogen Phosphate, $(\text{NH}_4)_2\text{HPO}_4$ by impregnation treatment step after the pre-oxidation step to produce an activated surface kapok. Activated surface kapok has greater porosity that will increase the adsorption rate. Lead (Pb^{2+}) ionic solution is used as the test material. The effectiveness of the low cost activated carbon prepared from kapok fibers, for the removal of lead from aqueous solution is investigated using experimental parameters including solution pH, contact time, adsorbent dose and initial metal ions concentration. The amount of metal residue in the solution is analyze using Atomic Absorption Spectroscopy method. Results show that heavy metal removal is optimum at pH 6.0, equilibrium contact time of 1 hour, kapok dose of 0.3 mg and initial concentration of 40 – 50 mg/L. Activated surface kapok is proven to be effective for removing heavy metals in wastewater.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

1.1.1 Heavy Metals

Over this past few decades, the tremendous increase in the use of the heavy metals in industrialization has inevitably resulted in a globally major problem. Heavy metal contamination in water is a crucial issue in water pollution. Compared to organic pollutants which majority are prone to biological degradation, heavy metal ions do not decompose into unhazardous end products (Hegazi, 2013; Gupta, Gupta and Sharma, 2001). Heavy metal contamination always occurs in aqueous waste of various industries, such as alloy industries, mining operations, paper and pulp, chloro-alkali, radiator manufacturing, storage batteries, metal plating, tanneries and smelting industries (Hegazi, 2013; Rao, Rao, Seshaiyah, Choudary and Wang, 2008; Kadirvelu, Thamaraiselvi and Namasivayam, 2001).

Other than that, landfill is also a source of heavy metal pollution. A lot of materials that thrown out as waste contain toxic substances which over time these toxins will seep into the soil, thus increasing the heavy metal flux in the land. Electronic waste such as mobile phones, televisions, computers and other electronic appliances contain of several dangerous toxic substances (Mohd Ali, 2014) that may cause land and water pollution. To date, about 20 metals are classified as toxic which if emitted to the environment in huge amounts will endanger the living beings through food chain mainly. Heavy metal contamination does not only affect the quality of water but also influence the crops and aquatic products such as wheat and fish.

1.1.2 Adsorption

Consequently this issue led to a pressing need to remove heavy metals from wastewater before it is being transported and cycled into the surroundings which also instigated human to find efficient solution to combat this wastewater pollution issue. The conventional chemical approaches for removing heavy metal ions from aqueous solution and wastewater have been carried out, such as oxidation, reduction, chemical precipitation-neutralization, membrane filtration technology, reverse osmosis (RO), electrochemical process and adsorption (Duan, Zhao, Yu, Zhang and Xu, 2013; Rao et al., 2008; Amuda, Giwa and Bello, 2007).

Among these methods, adsorption is an effective and economical method that was extensively used for the removal of heavy metals in wastewater (Yin, Ju, Zhang, Wang and Yang, 2008). Activated carbon is one of the most commonly utilized adsorbents other than clay (Ozdes, Duran and Senturk, 2011) and oxides (Shi, Tao, Yu, Wang and Ma, 2011) type adsorbents. However, most of these adsorbents especially activated carbon is non-biodegradable and may cause secondary pollution (Duan et al., 2013). Besides, since activated carbon is an expensive material, thus limiting its use as an adsorbent in developing countries.

In the recent years, numerous natural materials research topics has intensified as the alternative adsorbents for heavy metal removal from wastewater. These type of adsorption is known as biosorption method which has many advantages such as having adsorbent which is renewable, biodegradable, cheap and abundant in resource. Few researchers have used modified activated carbons prepared from biomaterials such as oil palm shells, sugarcane bagasse and cellulose (Gurgel, Júnior, Gil and Gil, 2008), rice husk (Aliyah, 2012), coir pith carbon (Kadirvelu and Namasivayam, 2000), peanut shells (Liu, Sun and Li, 2010), olive pulp, banana peel to extract lead (Pb) and copper (Cu) ions (Castro, Caetano, Ferreira, Padilha, Saeki, Zara, ... and Castro, 2011) and charcoal (Mishra and Chaudhary, 1994) for the removal of heavy metal ions from wastewater.

1.2 Problem Statement

The availability of high percentage of heavy metal ions in wastewater has become one of the serious issues of water pollution. This is due to their high solubility in the aquatic environments, heavy metals can be absorbed by living organisms. Once the heavy metal ions enter the food chain, it will result in accumulation of high concentrations of heavy metals in human body. If the metals are ingested more than the tolerable concentration, it can cause serious health disease (Babel and Kurniawan, 2004). Thus, it is absolutely essential to treat heavy metal contaminated wastewater prior to its release to the environment using the most efficient method, adsorption.

Various researches about heavy metals is done and the researchers discovered that adsorption is the most effective technique to remove heavy metals. The common adsorbent that has been used before to remove heavy metals is the activated carbon which is costly. Therefore, this research is aiming to solve the problem by using a low cost material from agricultural source as an effective adsorbent to remove heavy metal ion from aqueous solution.

Kapok fibre is a natural fibre consisting of a large hollow structure and its porosity volume is more than 90% (Hori, Flavier, Kuga, Lam, Iiyama, 2000). Moreover, it also exhibited good water repellency, high oil adsorption capability, and well reusable characteristic which is extremely useful for oil absorption mechanism (Mohd Ali, 2014) and a lot of analysis on kapok as an adsorbent has been focusing on oil spill issue but kapok fibres are seldom used as adsorbent for heavy metal.

This study will utilize the activated kapok fibres for the adsorption of heavy metal ions from aqueous solutions containing lead (Pb) metals. However, it is still questionable whether the heavy metal absorbed by the kapok fibre will contain any traces amount of water and if the role of huge lumen and its hydrophobic-oleophilic characteristic on the heavy metal removal still cannot be generally distinguished.

1.3 Objectives

The objectives of the study are:

- i. To investigate the effectiveness of activated surface kapok to remove lead under different experimental parameters;
- ii. To study the effect of contact time in removing lead;
- iii. To examine the effect of lead concentration in the aqueous solution on the adsorption;
- iv. To analyze the effect of amount of activated kapok used; and
- v. To observe the effect of pH on the adsorption of lead using activated surface kapok.

1.4 Scope of Study

The scope of the study involves preparation of activated kapok fibre using chemicals and it will be subjected to test in terms of heavy metal absorbency and optimum time to be exposed in aqueous condition. The characteristic of kapok fibres after treatment of chemical is studied. The kapok fibres will be characterized after the preparation using Field-Emission Scanning Electron Microscope (FESEM). The comparative study on the kapok fibres adsorbent will be studied in heavy metal aqueous solution containing lead (Pb) with different concentration, contact time, adsorbent amount used and different pH. The analysis will be produced from using Atomic Absorption Spectroscopy (AAS) results.

CHAPTER 2

LITERATURE REVIEW

2.1 Heavy Metals in Industrial Wastewater

2.1.1 Definition and Toxicity

Heavy metals are metal or metalloid elements whose density greater than 5 gram per centimeter cubic (g/cm^3). Example of heavy metals are copper (Cu), zinc (Zn), cadmium (Cd), mercury (Hg), chromium (Cr), cobalt (Co), lead (Pb), arsenic (As), manganese (Mn), nickel (Ni), selenium (Se), silver (Ag), and antimony (Sb). Several elements fall in this class, but Table 2.1 shows the heavy metal which relevant to environmental framework (Barakat, 2011). MCL stands for Maximum Contaminant Level, as established by United States Environmental Protection Agency (USEPA).

TABLE 2.1 MCL Standard – Most Hazardous Heavy Metal (Babel and Kurniawan, 2003)

Heavy metal	Toxicities	MCL (mg/L)
Arsenic	Skin manifestations, visceral cancers, vascular disease	0.05
Cadmium	Kidney damage, renal disorder, human carcinogen	0.01
Chromium	Headache, diarrhea, nausea, vomiting, carcinogenic	0.05
Copper	Liver damage, Wilson disease, insomnia	0.25
Nickel	Dermatitis, nausea, chronic asthma, coughing, human carcinogen	0.2
Zinc	Depression, lethargy, neurological signs and increased thirst	0.8
Lead	Damage the fetal brain, diseases of the kidneys, circulatory system, and nervous system	0.006
Mercury	Rheumatoid arthritis, and diseases of the kidneys, circulatory system, and nervous system	0.00003

Heavy metals enter living organism such as humans, animals and plants system via inhalation, food chain and direct handling. Naturally, a few of heavy metals are already presents in the organism themselves but in an small amount for example trace concentrations of Zinc (Zn) are essential for the physiological functions of living tissue and regulation of various biochemical processes (Amuda et al., 2007).

Unfortunately, the heavy metals become toxic and hazardous when it is in high concentration. It can cause severe health problems, including stunted growth and development, organ damage, cancer, impairment of the nervous system, and in acute cases, death (Barakat, 2011). Moreover, exposure to mercury (Hg) and lead (Pb), may also cause development of auto-immunity which is explained as a person's immune system attacks its own cells. This circumstance could trigger a joint diseases such as rheumatoid arthritis, and kidneys disease, nervous system, circulatory system, and damaging of the fetal brain. Other than that, heavy metals can cause irreversible brain damage at greater doses.

The deadliness of mercury was tragically illustrated in the Minamata Bay area of Japan incident that happen during the period of 1953 – 1960. It was reported that a total of 111 cases of mercury poisoning and 43 deaths among people who had consumed seafood from the contaminated bay. The toxicological effects of mercury were neurological damage, such as insanity, blindness, irritability, paralysis, chromosome breakage and birth defects (Rein, 2005).

In addition, there was also “Itai-Itai” incident that caused by the pollution of cadmium in Jintsu river of Japan (Sud, Mahajan and Kaur, 2008). Alturkmani (n.d.) reported in his study that an acute cadmium poisoning in humans poses a critical effects to them. For instance, kidney damage, high blood pressure, destruction of testicular tissue and the destruction of red blood cells. Wastewater regulations were established to minimize human and environmental exposure to hazardous chemicals. This includes limits on the types and concentration of heavy metals that may be present in the discharged wastewater.

2.1.2 Industrial Wastewater Sources

Industrial wastewater streams containing heavy metals are produced from different industries. Electroplating and metal surface treatment processes produce significant quantities of wastewaters with heavy metals from various applications. For instance electroplating, anodizing-cleaning, electroless depositions, milling, conversion-coating and etching. Metal finishing and electroplating include the deposition of thin protective layers into a prepared surface of metal using electrochemical processes. In addition, other significant source of heavy metals pollution come from manufacture of the printed circuit board (PCB). Tin, nickel and lead solder plates are the most extensively used resistant over plates.

Other than that, petroleum refining also generates conversion catalysts contaminated with vanadium, nickel, and chromium; production of inorganic pigment producing pigments that contain cadmium and sulfide chromium compounds; and photographic processes producing film with high concentrations of ferro-cyanide and silver (Sörme and Lagerkvist, 2002). While in the nuclear plants, large amount of water is consumed for operation results in the nuclear effluent with heavy metals ions are discharged into surface water which could pollute the aquatic systems (Hagberg and Lofgren, 2007; Begum, Zaman, Mondol, Ismal and Hossain, 2011). Table 2.2 simplifies the type of heavy metals found in major industries.

TABLE 2.2 Heavy Metals Found in Major Industries (Alturkmani, n.d.)

Industry	As	Cd	Cr	Hg	Pb	Ni	Zn
Pulp and Paper Mills			X	X	X	X	X
Organic Chemicals	X	X	X	X	X		X
Alkalis, Chlorine	X	X	X	X	X		X
Fertilizers	X	X	X	X	X	X	X
Petroleum Refining	X	X	X		X	X	X
Steel Works	X	X	X	X	X	X	X
Aircraft Plating, Finishing		X	X	X		X	
Flat Glass, Cement			X				
Textile Mills			X				
Tanning			X				
Power Plants			X				

2.2 Lead

Lead is one of the most toxic heavy metals if it is highly accumulated in human. In natural water contents, lead are always less than 5 µg/L (Srisa-ard, 2002). Furthermore, in reference to USEPA (1986), the recent Environmental Protection Agency (EPA) standard for lead in drinking water and wastewater is 0.05 mg/L and 0.5 mg/L, respectively. According to the Substance Priority List compiled in 2011 by USEPA and the Agency for Toxic Substances and Disease Registry (ATSDR), the second hazardous heavy metals among the substances after arsenic (As) is lead (Pb). This is due to its' chronic effect to human health.

2.2.1 Lead Properties

The symbol Pb for lead is derived from Latin of Plumbum. Lead has a bluish-white color but in liquid phase the color turns chrome-silver. It is very soft, highly malleable and ductile, but it is a poor conductor of electricity. Table 2.3 shows the physical and chemical properties of lead based on the Material Safety Data Sheet (MSDS) and WHO (1989).

TABLE 2.3 Physical and Chemical Properties of Lead

Property Description	Value
Atomic number	82
Molecular Weight	207.2 g/mole
Density	11.34 g/cm ³ at 20°C
Melting point	327.43°C
Boiling point	1740°C

2.2.2 Source of Lead

Lead pollution in air was the highest during the previous century due to the lead attached to petrol. The lead salts formed by the burning of leaded petrol in car engine entered the environment through the car exhaust. The larger particles will drop to the ground and contaminate soil or water, but the smaller particles will travel via air and remain in the atmosphere (Lenntech, 2015). However, the lead pollution gradually decrease with the use of unleaded petrol as shown by a graph in Figure 2.1.

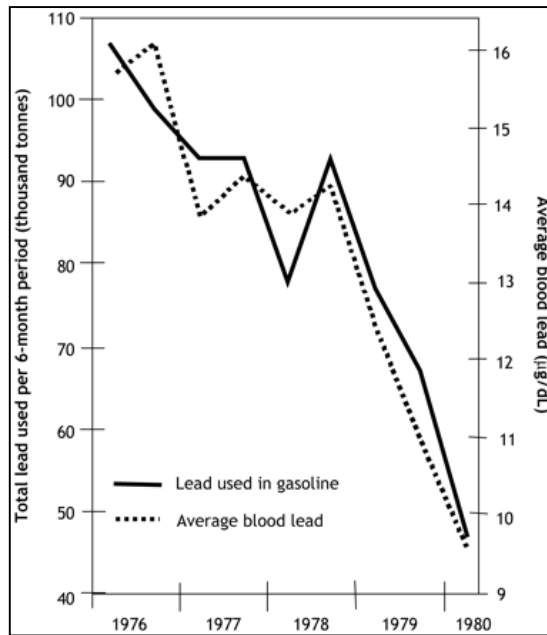


FIGURE 2.1 Lead Concentrations in Petrol and Children’s Blood (USA) (Järup, 2003)

According to Table 2.2, lead is mainly found in pulp and paper mills, organic chemicals, alkalis, chlorine, steel works, fertilizers and petroleum refining industries. Furthermore, it is used in the manufacturing of lead acid batteries, solder, alloys, cable sheathing, pigments, rust inhibitors, ammunition, glazes, and plastic stabilizers (Aliyah, 2012; WHO, 1989) as shown in Figure 2.2. Lead concentrations in wastewater from battery manufacturing, tailing pond, acid mine drainage, and steel production plants range from 0.5 to 25 mg/L.

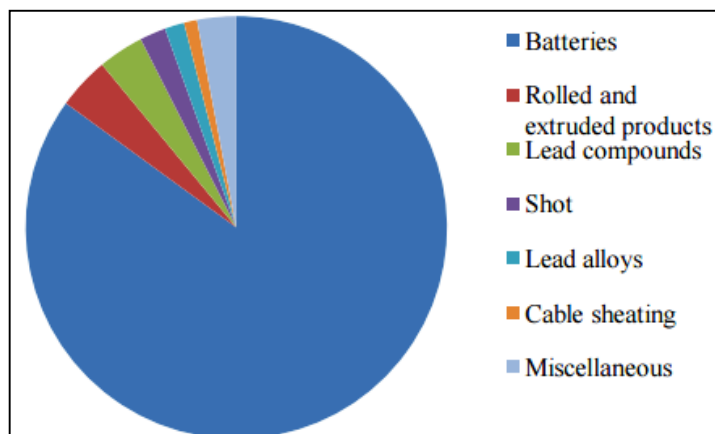


FIGURE 2.2 Percentage of Lead Usage

2.2.3 Health Effects

Lead is ubiquitous in the earth and is lethal at high concentration. It is a general metabolic poison and enzyme inhibitor and it enters the human body through the ingestion of food, water and inhalation of air containing lead more than allowed Maximum Contaminant Level (MCL). The detrimental effects of lead accumulation is most prone to pregnant women, children up to 6 years old, infants, and the fetus (Aliyah, 2012).

Fu and Wang (2011) said that it can cause central nervous system mutilation and also damage the liver, kidney, basic cellular processes, reproductive system and brain functions. In addition, lead poisoning may cause anemia, insomnia, dizziness, headache, loss of appetite, constipation, irritability, poor muscle coordination, hallucination and kidney failure (Environmental Protection Agency, 2013; Naseem and Tahir, 2001).

2.3 Kapok

2.3.1 Kapok Tree

Kapok tree or its scientific name *Ceiba pentandra (L.) Gaertn.* is a tropical tree which belong to the family of Bombaceae and grow in Asia, Africa and South America. Whereas, Malaysian Kapok is commonly found in northern parts of peninsular Malaysia (Mohd Ali, 2014). Kapok is mostly cultivated for the seed fiber.

2.3.2 Common Use

The fruits of this tree are in the form of capsules containing- a floss in which a number of dark brown seeds are embedded. The floss has been used for centuries to stuff pillows, soft toy and mattresses. The seeds are usually discarded. In rural areas, however, the seeds are roasted and consumed after removing the husk. Apparently they often upset the stomach and hence they are consumed only in small quantities.

Rao et al. stated that except for the fibers, the plant parts such as bark, gum, root and leaf have high medicinal applications. Georgi (1922) described that the oil content of Kapok seeds from different area in Malaysia, is in the range of 20% to 25% (Berry, 1979). On the other hand, kapok husk fibers are of no economic commercial and are often discarded as an agricultural waste (Rao et al., 2007).

2.3.3 Kapok Fiber and as an Adsorbent

Kapok is a silky fiber that encloses the seeds of *Ceiba pentandra* trees as shown in Figure 2.3 and has a yellowish or light-brown color with a silk-like luster. Kapok fiber is a highly lignified organic seed fiber mainly made up of the cellulose (Zheng, Wang, Zhu and Wang, 2014) and shown in Figure 2.4. Its' crystallization degree is 35.90% with bulk density of 0.30 g/cm³ (Xiao, Yu and Shi, 2005). From an investigation of the physicochemical and sorption characteristic of Malaysian kapok, its microstructure exhibit hollow tubular structure with an average external diameter of $2.15 \pm 6.5\mu\text{m}$. (Ismail, 2013).



FIGURE 2.3 Kapok Seeds within Fibres in Kolkata, West Bengal, India

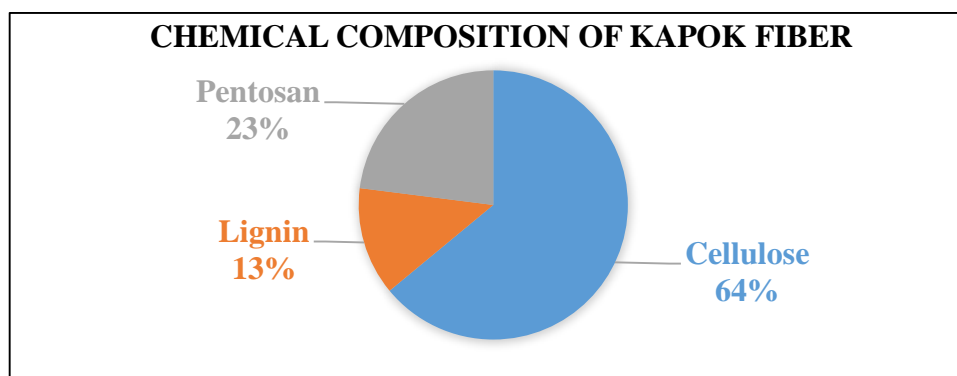


FIGURE 2.4 Chemical Composition of Kapok Fibre

Kapok has the advantages over other absorbing materials: inexpensive, biodegradable, intrinsic hydrophobic characteristic and great sorption capacity and huge lumen. Several studies on kapok stated that kapok fiber exhibited good water repellency, high oil adsorption capability, and well reusable features, which is a good potential for application in oil pollution control.

A studies from Rao et al. in 2006 revealed that activated carbon prepared from the kapok fibers has good capacity for the cadmium and copper adsorption. In their 2008 investigation, the effectiveness of removing lead and zinc from aqueous solutions using the activated carbon prepared from kapok fibers was researched. From the 2008 journal, they concluded that the adsorption of lead and zinc using activated carbon is dependent on contact time, initial concentration, pH and dose of the adsorbent as shown in Figure 2.5 below. Besides, not only it is effective but kapok activated carbon is an economically feasible adsorbent.

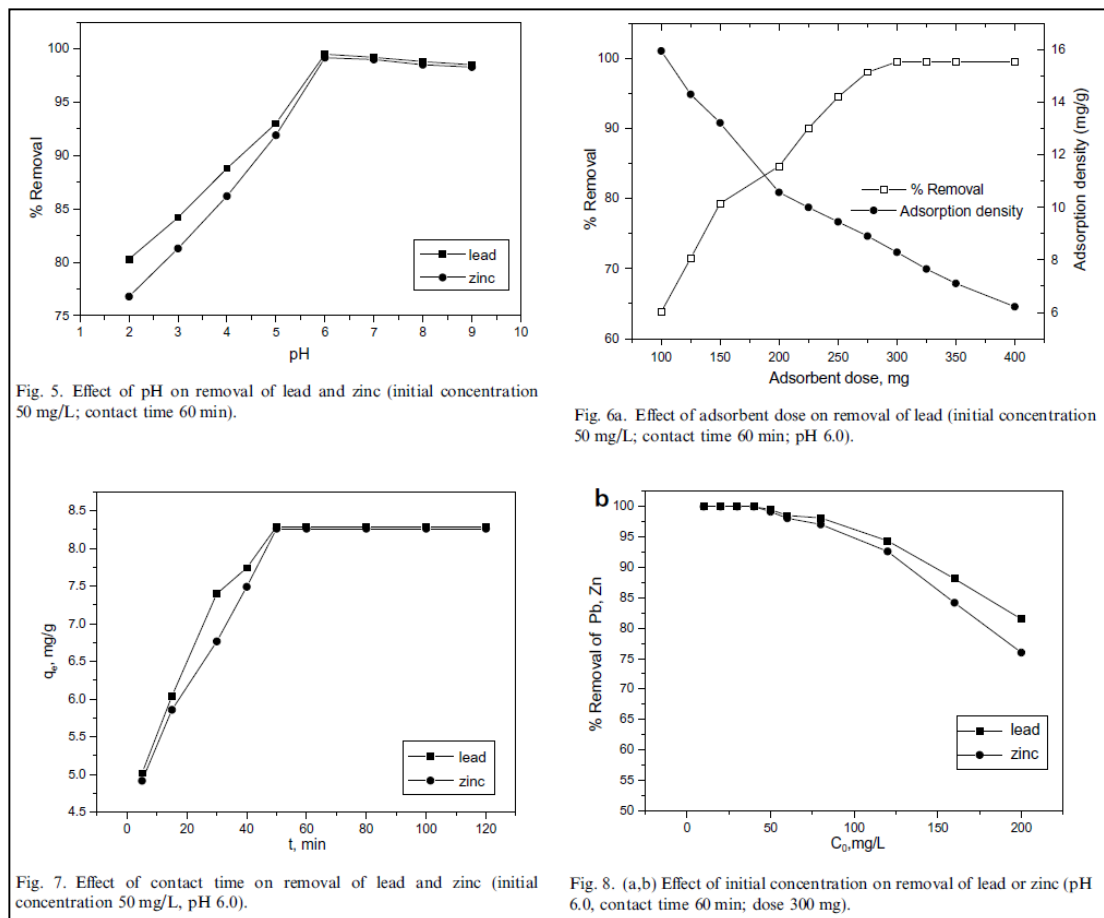


FIGURE 2.5 Results from Rao et al. (2008) Research

2.4 Activated Surface Kapok

Activated carbon (AC) is widely used as an effective adsorbent especially wastewater treatment. According to a journal by Wang and Zhou (2008), activated carbon fiber (ACF) from kapok was prepared by washing it with 2wt% NaOH to eliminate wax, ash and other small molecules. Next, it will undergo three different pre-treatment methods which are:

- i. Single-step pre-treatment: The kapok fibre was impregnated with $(\text{NH}_4)_2\text{HPO}_4$
- ii. Two-step pre-treatment (impregnation – pre-oxidation)
- iii. Two-step pre-treatment (pre-oxidation – impregnation)

The impregnation treatment is performed by impregnating the kapok fiber with 30wt% $(\text{NH}_4)_2\text{HPO}_4$ for 1 day. While, the pre-oxidation treatments is carried out by heating the materials at 200°C in air for 2 hours. Then all the pretreated samples is heated in the oven under a nitrogen flow at 923 K for 70 minutes.

However, a report by Rao et al. (2008) also mentioned about preparation of kapok ACF, the kapok fibers were cut into small pieces, afterwards they were washed several times with deionized doubly distilled (DDD) water and left to dry. The carbonization of fibers was performed in a muffle furnace at 200°C for 2 hours. Steam activation (SA) of the carbon was carried out afterwards.

Other than these 2 methods to prepare ACF, a journal reported by Chung, Hwang, Shim, Kim, Park, Choi and Lee (2012) described the preparation of ACF from kapok fiber with different method from the method applied by Wang and Zhou (2008). The kapok fibers were air-dried and afterwards oven-dried at 100°C for 2 days to remove the moisture content. After that, the samples were carbonized at 700°C for 1 hour under nitrogen flow. Then, they were carbonized again under the same parameter but different times (10 – 50 minutes) to determine the optimum activation time. Lastly, the samples were washed with hot water to neutralize the pH and then dried at 120°C for 1 day in a vacuum dryer.

The ACF samples from Chung et al. then were analyzed by field-emission scanning electron microscopy (FESEM) to analyze the morphology of the activated kapok fiber. Next, an aqueous solution of preferred heavy metals is prepared and the kapok fiber ACF is tested and then analyzed using the Atomic Absorption Spectroscopy (AAS) to observe the residual amount heavy metal, lead (Pb) in the solution.

Pre-oxidation step is proven to be able to increase the surface area of kapok especially if then impregnated with $(\text{NH}_4)_2\text{HPO}_4$ (Wang and Zhou, 2008). A study from Chung (2008) reported that oxidized kapok fibers enhanced the adsorption of heavy metals. While, carbonization technique (Chung et al., 2012) concluded that the process stimulates the development of porosity on the kapok fibers' surface as shown in Figure 2.6.

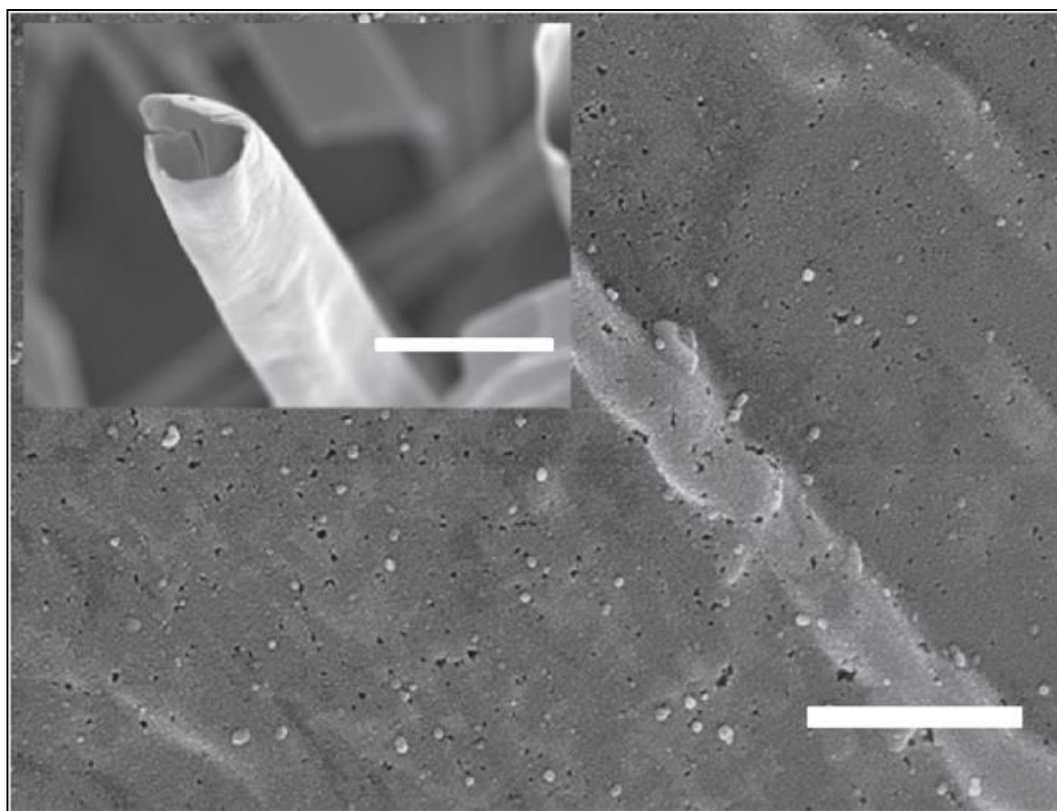


FIGURE 2.6 FESEM Image of Kapok-Derived Activated Hollow Carbon Fibres (KAHCF)

Rao et al. (2008) investigated on the four parameters; contact time, pH, kapok dosage and initial concentration however, the method of preparing activated kapok used by Wang and Zhou (2008) and Chung et al. (2012) is more clearly explained and the activated kapok produced might be better than Rao et al. activated kapok. However, Wang and Zhou (2008) and Chung et al. (2012) experimented the activated kapok on removal of methylene blue (MB). The combination of these 2 research on method of preparing activated kapok and using the analyzation procedure by Rao et al. to observe the effectiveness of this activated kapok on removal of lead by testing it based on the parameters.

From Figure 2.5, their results showed that lead removal optimum at pH 6.0, 300 mg adsorbent dose, 50 minutes of contact time and 0 – 50 mg/L of lead initial concentration. Thus, my project will be studying these four parameters but with different method of preparing activated surface kapok to see the effectiveness of this type of activated kapok.

CHAPTER 3

METHODOLOGY

3.1 Project Methodology

The methodology of this research is based on experimental work where the activated carbon fibre (ACF) from kapok fibre is prepared using impregnation with Diammonium Hydrogen Phosphate method. For the performance study, these activated kapok fibres will be used and the measurement as well as evaluation on the sorption capacity and efficiency will be conducted. The parameters used in the performance study will be according to the objectives which is the pH, contact time, initial concentration of lead and the dose of kapok adsorbent.

Field–Emission Scanning Electron Microscope (FESEM) would be used for the characterization to study the structural and composition of the activated surface kapok fibres. Two type of kapok fibre were observed using FESEM, the raw kapok and the activated surface kapok. Atomic Absorption Spectroscopy (AAS) is also used to analyze the aqueous solution for its metal content which in this study, to determine the amount of lead metal residue in the aqueous solution. Calculation was done to obtain the percent of lead removed in each solution sampled by the AAS. The experimental work was executed in 14 weeks.

3.1.1 Materials and Apparatus

TABLE 3.1 The Materials, Apparatus, Chemicals Used and Its Function

No	Materials/Apparatus/Chemicals	Uses
1	Kapok Fiber	To be modified as an adsorbent
2	Diammonium Hydrogen Phosphate, $(\text{NH}_4)_2\text{HPO}_4$	Chemical used to soak kapok to change its structure
3	Lead Nitrate, $\text{Pb}(\text{NO}_3)_2$ Solution	Represents wastewater, solution to be treated
4	Sodium Hydroxide, NaOH	To remove wax from kapok, to neutralize the $\text{Pb}(\text{NO}_3)_2$ solution
5	FESEM	To get the micrograph of kapok fiber
6	AAS	To analyze the aqueous solution for its metal content
7	Nitrogen Gas, N_2	To carbonize the kapok
8	Oven	To dry the kapok fiber
9	Vacuum Dryer	To dry the kapok fiber
10	Thermometer	To measure the temperature
11	Measuring Cylinder	To measure volume of chemical
12	Electronic Weighing Machine	To weigh any material
13	Filter Paper	To filter the kapok adsorbent from the $\text{Pb}(\text{NO}_3)_2$ solution
14	Flask	To contain the chemical
15	Water	To wash kapok fiber, to dilute the $\text{Pb}(\text{NO}_3)_2$ solution

3.1.2 Procedure

The procedure was divided into two parts where the first part is the preparation of the activated surface kapok. After the first part is completed, part two was carried out where the activated kapok prepared was investigated for its effectiveness in removing heavy metal lead (Pb) from its aqueous solution. The second part was split into four different experiments, Experiment A, B, C and D. Each experiment will test the pH, contact time, initial concentration of lead and the dose of kapok adsorbent.

3.1.2.1 Preparation of Activated Surface Kapok

- 1) Kapok fiber was washed with 2wt% NaOH to eliminate wax, ash and other small molecules.
- 2) Kapok fiber was air-dried and afterwards oven-dried at 100°C for 2 days to remove the moisture content.
- 3) Kapok fiber was impregnated with 30wt% $(\text{NH}_4)_2\text{HPO}_4$ for 1 day.
- 4) After that pre-oxidation treatment was carried out by heating the materials at 200°C in air for 2 hours for the pre-treatment step.
- 5) The samples of the pre-treatment were then carbonized at 700°C for 1 hour under nitrogen flow in oven.
- 6) Afterwards, the samples were carbonized again at 700°C under nitrogen flow but with different time which is 30 minutes.
- 7) The samples were washed with hot water to neutralize the pH. Next, the samples were dried at 120°C for 1 day in a vacuum dryer.
- 8) The samples of kapok ACF were analyzed with FESEM.

3.1.2.2 Kapok Performance Study – Removal of Lead (Pb)

Experiment A - The Effect Of pH Value

- 1) Aqueous solution of $\text{Pb}(\text{NO}_3)_2$ was prepared with constant concentration of 50 mg/L according to the amount of solution needed.
- 2) The pH value of the solution was adjusted using 0.1M NaOH solution. The lead solution pH was adjusted from pH 3.0 to 9.0 in an increment of 1.0 unit.
- 3) 0.2 g of the kapok ACF was shaken at 25°C in a flask containing 50 mL of the lead solution for 60 minutes.
- 4) The material was filtered and the residual solution was analyzed for the detection of heavy metal ion concentrations using AAS.
- 5) The experiment was repeated for 3 times.

Experiment B - The Effect Of Contact Time

- 1) Aqueous solution of $\text{Pb}(\text{NO}_3)_2$ was prepared with constant concentration of 50 mg/L according to the amount of solution needed.
- 2) The pH value of the solution was adjusted using 0.1M NaOH solution. The lead solution pH was kept at pH 6.0 during this experiment.
- 3) 0.2 g of the kapok ACF was shaken at 25°C in a flask containing 50mL of the lead solution from 20 – 140 minutes.
- 4) The material was filtered and the residual solution was analyzed for the detection of heavy metal ion concentrations using AAS.
- 5) The experiment was repeated for 3 times.

Experiment C - The Effect Of Lead Initial Concentration

- 1) Aqueous solution of $\text{Pb}(\text{NO}_3)_2$ was prepared with different concentration of lead in the solution: 40, 50, 60, 80, 120, 160, 200 mg/L according to the amount of solution needed, 150 mL for each concentration level.
- 2) The pH value of the solution was adjusted using 0.1M NaOH solution. The lead solution pH was kept at pH 6.0 during this experiment.

- 3) 0.2 g of the kapok ACF was shaken at 25°C in a flask containing 50 mL of the lead solution for 60 minutes.
- 4) The material was filtered and the residual solution was analyzed for the detection of heavy metal ion concentrations using AAS.
- 5) The experiment was repeated for 3 times.

Experiment D - The Effect Of Dosage Of Activated Kapok Adsorbent

- 1) Aqueous solution of $Pb(NO_3)_2$ was prepared with constant concentration of 50mg/L according to the amount of solution needed.
- 2) The pH value of the solution was adjusted using 0.1M NaOH solution. The lead solution pH was kept at pH 6.0 during this experiment.
- 3) Different amount of the kapok adsorbent ranging from 0.1 to 0.4 g with an increment of 0.05g was shaken at 25°C in a flask containing 50 mL of the lead solution for 60 minutes.
- 4) The material was filtered and the residual solution was analyzed for the detection of heavy metal ion concentrations using AAS.
- 5) The experiment was repeated for 3 times.

The summary of Experiments A, B, C and D is outlined by Table 3.2.

TABLE 3.2 Experimental Condition and Values Summary

Experiment	Variable	Value	Constant
A	pH Value	3.0, 4.0, 5.0, 6.0, 7.0, 8.0 and 9.0 pH	Contact time = 60 mins Concentration = 50 mg/L Kapok dosage = 0.2 g
B	Time	20, 40, 60, 80, 100, 120 and 140 minutes	Concentration = 50 mg/L Kapok dosage = 0.2 g pH = 6.0
C	Concentration	40, 50, 60, 80, 120, 160 and 200 mg/L	Contact time = 60 mins Kapok dosage = 0.2 g pH = 6.0
D	Kapok Dosage	0.1, 0.15, 0.2, 0.25, 0.3, 0.35 and 0.4 g	Contact time = 60 mins Concentration = 50 mg/L pH = 6.0

2.1 Project Flow Chart

This is the process flow for this research project that must be so that the objectives of the study can be successfully achieved as shown in Figure 3.1.

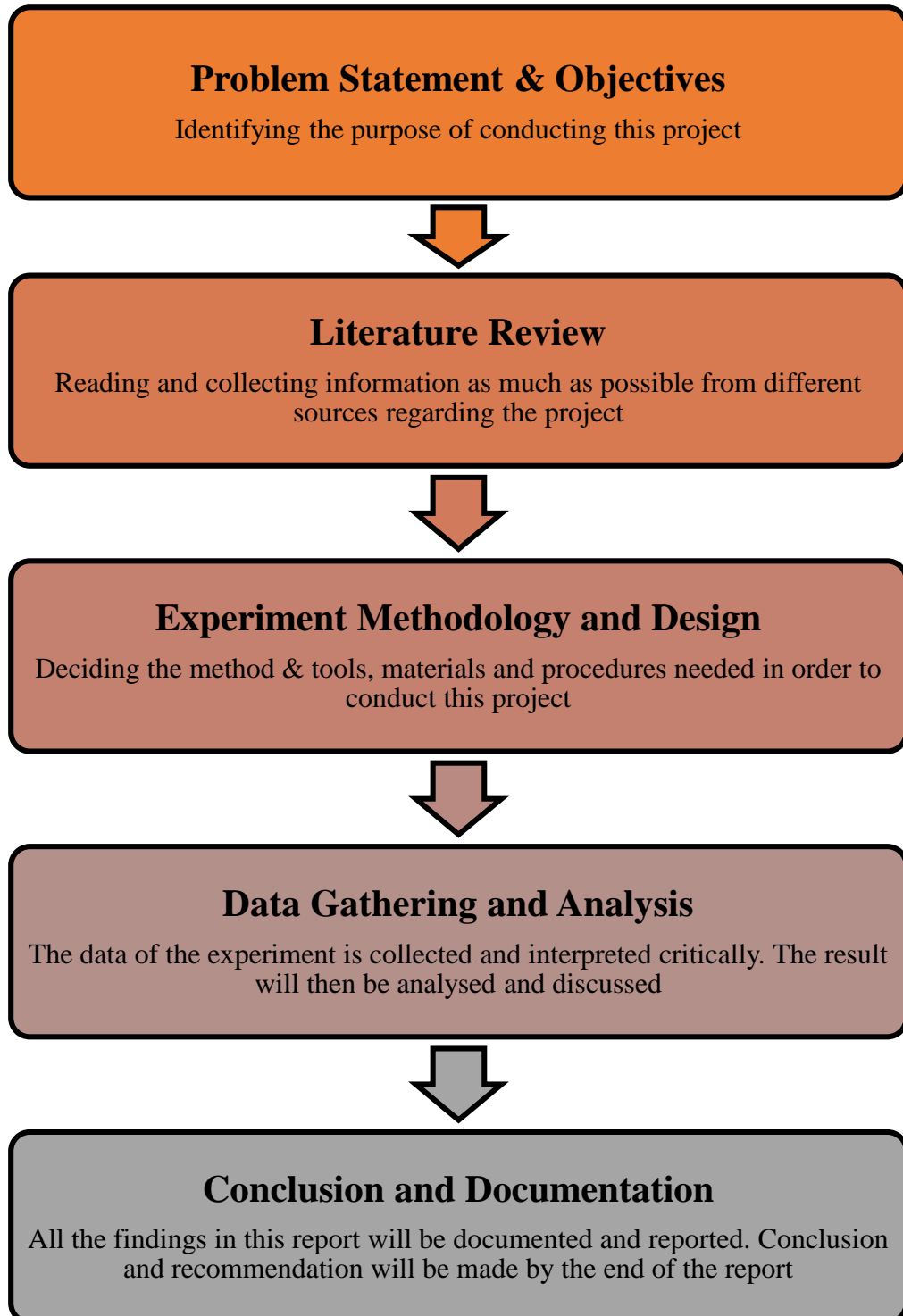


FIGURE 3.1 Project Flow Chart

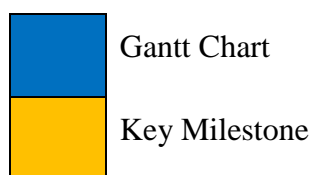
3.2 Gantt Chart and Key Milestones

3.2.1 Final Year Project I Guideline (Planning Phase)

Table 3.3 shows the Gantt chart and key milestones for the Final Year Project I.

TABLE 3.3 Gantt Chart and Key Milestones FYP I

No.	Details	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection Of Project Title	■	■												
2	Preliminary Research Work And Literature Review		■	■	■	■	■	■							
3	Submission Of Extended Proposal							■							
4	Preparation For Proposal Defense							■	■						
5	Proposal Defense									■					
6	Project Work Commencement										■	■	■		
7	Detailed Literature Review										■	■	■		
8	Preparation Of Interim Report										■	■	■		
9	Submission Of The Draft Of Interim Report												■		
10	Submission Of Interim Report Final													■	

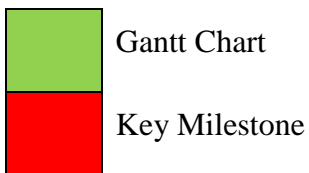


3.2.2 Final Year Project II Guideline (Execution Phase)

Table 3.4 shows the Gantt chart and key milestones for the Final Year Project II.

TABLE 3.4 Gantt Chart and Key Milestones FYP II

No.	Details	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work														
2	Submission of Progress Report														
3	Project Work Continues														
4	Pre-SEDEX														
5	Submission of Draft Final Report														
6	Submission of Dissertation (Soft Bound)														
7	Submission of Technical Paper														
8	Oral Presentation														
9	Submission of Project Dissertation (Hard Bound)														



CHAPTER 4

RESULTS AND DISCUSSION

4.1 Kapok Fiber Characterization (FESEM)

Field-Emission Scanning Electron Microscope (FESEM) was used in this project to analyze the fiber morphology of kapok before and after surface activation step. Figure 4.1 below showed the micrograph image of the raw kapok fiber before the kapok is modified to activated surface kapok and Figure 4.2 displayed the image of the activated surface kapok.

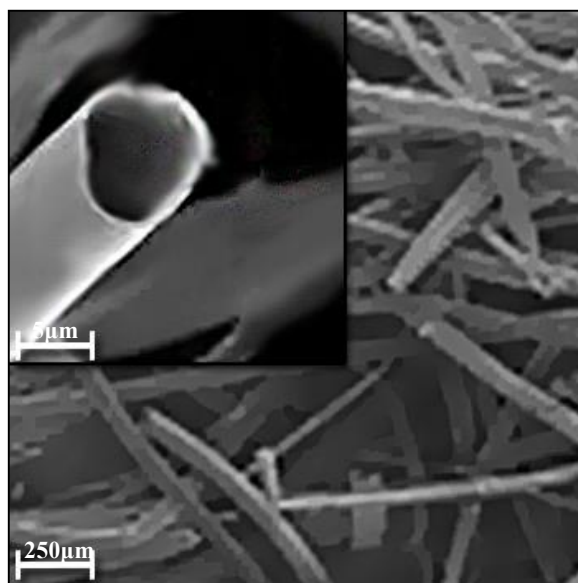


FIGURE 4.1 Raw Kapok FESEM Image

Based on the observation of the micrograph of raw kapok in Figure 4.1, it can be seen that raw kapok fiber has quite a smooth surface without ripple or ridges and also the presence of a hollow tube-like structure.

As described in the literature review, the tubular structure allows it to absorb material, however the smooth surface is due to the wax that makes the kapok float a bit in water. The hollow structure covers up to 90% of the whole raw kapok surface area which will ease the adsorption process later.

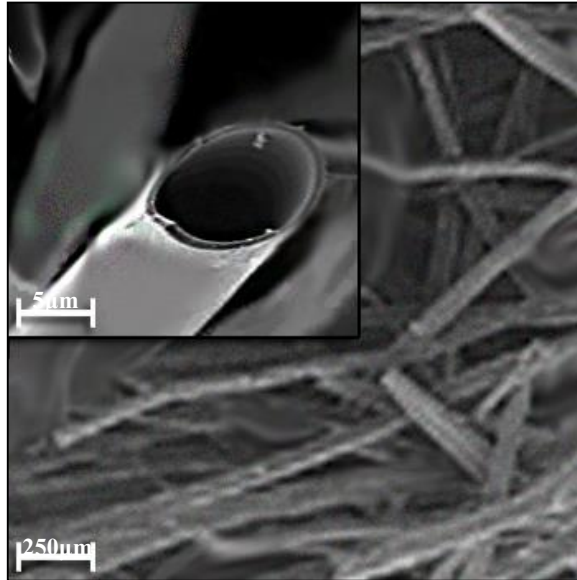


FIGURE 4.2 Activated Surface Kapok FESEM Image

After the preparation of activated surface kapok, the sample is analyzed by FESEM to study the morphology of the modified kapok. Comparing Figure 4.1 with Figure 4.2, there were hardly visible changes, however there are some differences. It can be seen that the hollow tube of the kapok fiber widens a little bit and the surface became rougher due to washing the raw kapok with NaOH that washed the wax on the surface. The activated surface kapok will have greater surface area that can enhance the adsorption rate of heavy metals.

4.2 Lead (Pb) Removal Analysis (AAS)

Adsorption test of activated surface kapok using different testing parameters; pH value, initial concentration of lead (Pb), kapok adsorbent dosage and contact time, was carried out to evaluate the removal efficiency of kapok as an adsorbent. Table 4.1 below showed the standard atomic absorption conditions for lead (Pb) in Atomic Absorption Spectroscopy (AAS).

TABLE 4.1 Standard Atomic Absorption Conditions for Lead (AAS Booklet)

Standard Atomic Absorption Conditions for Pb					
Wavelength	Slit	Relative Noise	Characteristic Concentration	Characteristic Concentration Check	Linear Range
(nm)	(nm)		(mg/L)	(mg/L)	(mg/L)
283.3	0.7	0.43	0.45	20.0	20.0
217.0	0.7	1.0	0.19	9.0	20.0
205.3	0.7	1.4	5.4	250.0	---
202.2	0.7	1.8	7.1	350.0	---
261.4	0.7	0.35	11.0	500.0	---
368.3	0.7	0.40	27.0	1200.0	---
364.0	0.7	0.33	67.0	3000.0	---

4.2.1 Tabulated Results

Percentage of lead (Pb) removed can be calculated from the Equation 1 below. Table 4.2 is the tabulated data collected and calculated from the experiment results.

$$\% \text{ Removal} = \frac{C_0 - C_1}{C_0} \times 100\% \quad (1)$$

C_0 is the initial concentration of the lead solution and C_1 is the final concentration of the lead solution. The data for C_0 and C_1 are the average values for the repeated experiment. From the tabulated data in Table 4.2, four graphs were plotted and analyzed to show the relationship between the parameters and the percentage of lead removed.

TABLE 4.2 Experimental Data

Experiment A – The Effect of pH Value			
pH value	C₀ (mg/L)	C₁ (mg/L)	% Removal
3.0	50.00	11.10	77.8
4.0	50.00	8.75	82.5
5.0	50.00	6.90	86.2
6.0	50.00	3.30	93.4
7.0	50.00	3.70	92.6
8.0	50.00	3.95	92.1
9.0	50.00	4.25	91.5
Experiment B – The Effect of Contact Time			
Time (min)	C₀ (mg/L)	C₁ (mg/L)	% Removal
20	50.00	8.75	82.5
40	50.00	6.90	86.2
60	50.00	3.30	93.4
80	50.00	3.70	92.6
100	50.00	3.95	92.1
120	50.00	4.25	91.5
140	50.00	4.60	90.8
Experiment C – The Effect of Lead Initial Concentration			
C₀ (mg/L)		C₁ (mg/L)	% Removal
40.00		3.40	91.5
50.00		4.50	91.0
60.00		5.88	90.2
80.00		9.20	88.5
120.00		15.60	87.0
160.00		33.76	78.9
200.00		49.80	75.1
Experiment D – The Effect of Dosage of Activated Kapok Adsorbent			
Kapok Dosage (g)	C₀ (mg/L)	C₁ (mg/L)	% Removal
0.10	50.00	21.75	56.5
0.15	50.00	14.10	71.8
0.20	50.00	11.40	77.2
0.25	50.00	6.60	86.8
0.30	50.00	4.15	91.7
0.35	50.00	4.15	91.7
0.40	50.00	4.10	91.8

4.2.2 The Effect of pH Value

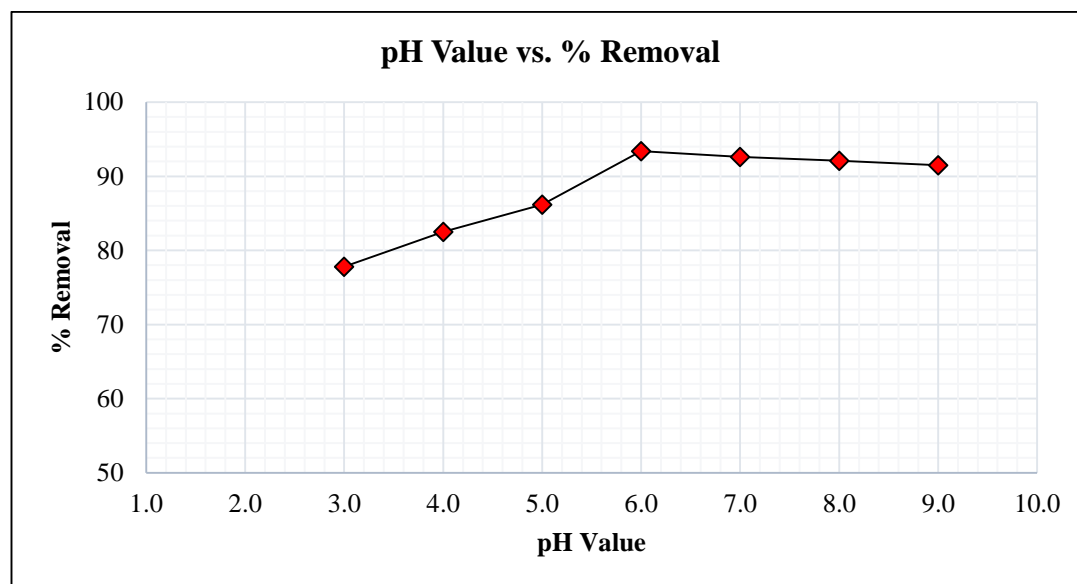


FIGURE 4.3 Graph of pH Value vs. % Removal

The effect of lead nitrate solution pH value on the removal of lead is shown in Figure 4.3. Rao et al. (2008) mentioned in their analysis that pH value affected the binding of heavy metal ions onto the surface functional group of the adsorbent thus making pH value one of the important parameter in determining the effectiveness of the activated surface kapok. From the graph above, the percentage lead removed increased as the pH value moves to less acidic until pH 6.0, but showed declining values as the pH value going towards the base value, pH 9.0. Thus, pH 6.0 is the optimum pH value for the adsorption of Pb using kapok.

This condition is due to the competition among the elements involved in the adsorption process. At small pH (acidic), the surface of the adsorbent which is negatively charged was surrounded by hydronium ions (H_3O^+) that compete with the lead ions (Pb^{2+}). This situation inhibits the Pb^{2+} ions from moving towards the binding sites on the kapok adsorbent (Rao et al., 2008). The optimum pH value at pH 6.0 indicates the high efficiency of kapok in adsorbing lead from wastewater or drinking water.

4.2.3 The Effect of Contact Time

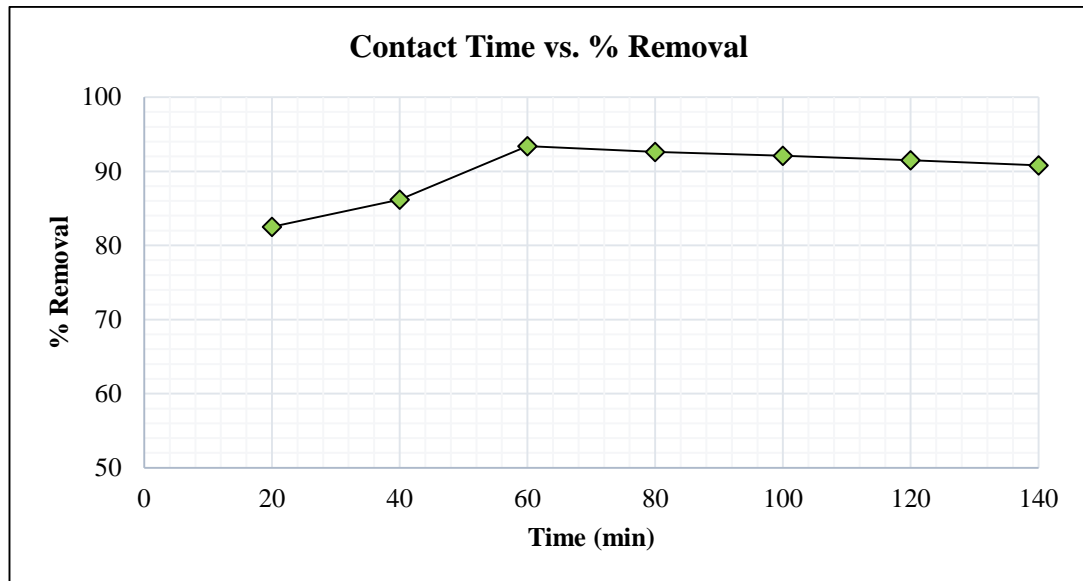


FIGURE 4.4 Graph of Contact Time vs. % Removal

Besides pH value, contact time is also one of the important factor affecting removal of heavy metal. Figure 4.4 display the relationship between the contact time and the amount of lead removed. As the contact time became longer, the percentage of lead removed increased but then starting after the contact time of 60 minutes, the slope changed direction, where the percentage of removal is decreasing. So, the contact time of 60 minutes is the optimum time for the efficiency of activated surface kapok.

This graph behavior where the percentage removal increased rapidly at the beginning of the adsorption process, but also showed a slow reduction after minute 60 is called as the equilibrium state. The reason why it showed rapidly increased rate of adsorption from 0 to 60 minutes of contact time is feasibly due to the presence of a huge surface area of kapok adsorbent at the beginning of the adsorption process so a lot of heavy metal is adsorbed. After 60 minutes, the active sites on the activated kapok surface started to became concentrated with Pb ions that resulted in less lead adsorbed and decrease in adsorption rate.

4.2.4 The Effect of Lead (Pb) Initial Concentration

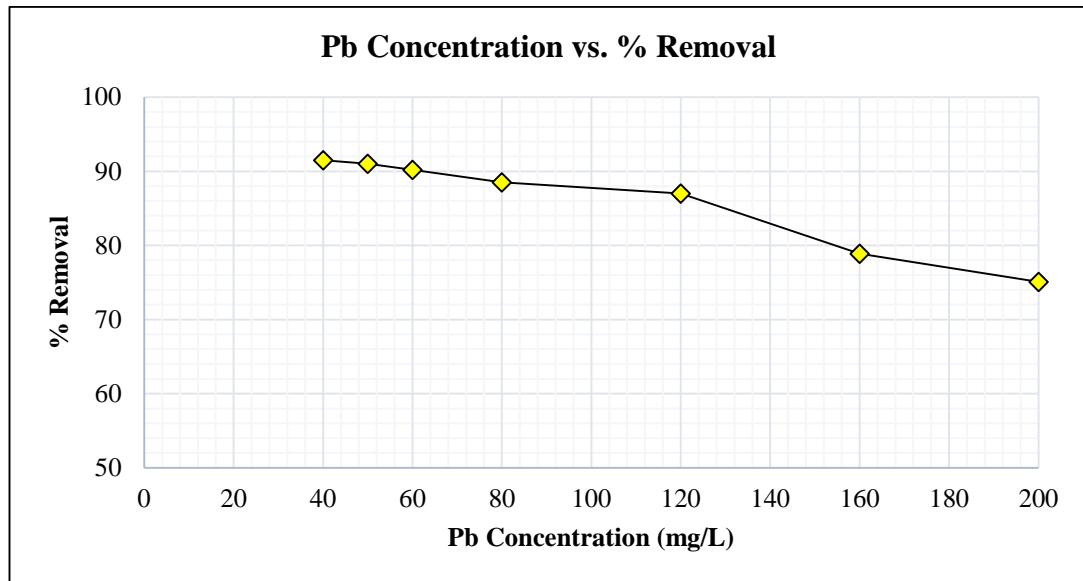


FIGURE 4.5 Graph of Initial Pb Concentration vs. % Removal

Based on the third experiment, Experiment C that test the effects of varying the initial concentration of heavy metals ranging between 40 mg/L to 200 mg/L of lead solution. Even though the graphs of pH value and contact time versus the percentage removal demonstrated an almost similar behavior, however the graph in Figure 4.5 showed an inverse trend. The amount of lead removed reduces as the initial lead concentration increased. The optimum value for lead initial concentration is between 40 – 50 mg/L.

This is because adsorbent has a limited number of active sites that will become saturated after some degree of concentration. The ratio of metal ions present to the available active site on adsorbent is small at low concentration condition. Nevertheless, by adding the kapok adsorbent dosage can probably solve this situation since the amount of kapok dosage is kept at constant (0.2 g) throughout the Experiment C. However, raising the amount of activated surface kapok used will also add up the cost of adsorption process if we consider it using the industrial aspect.

4.2.5 The Effect of Dosage of Activated Kapok Adsorbent

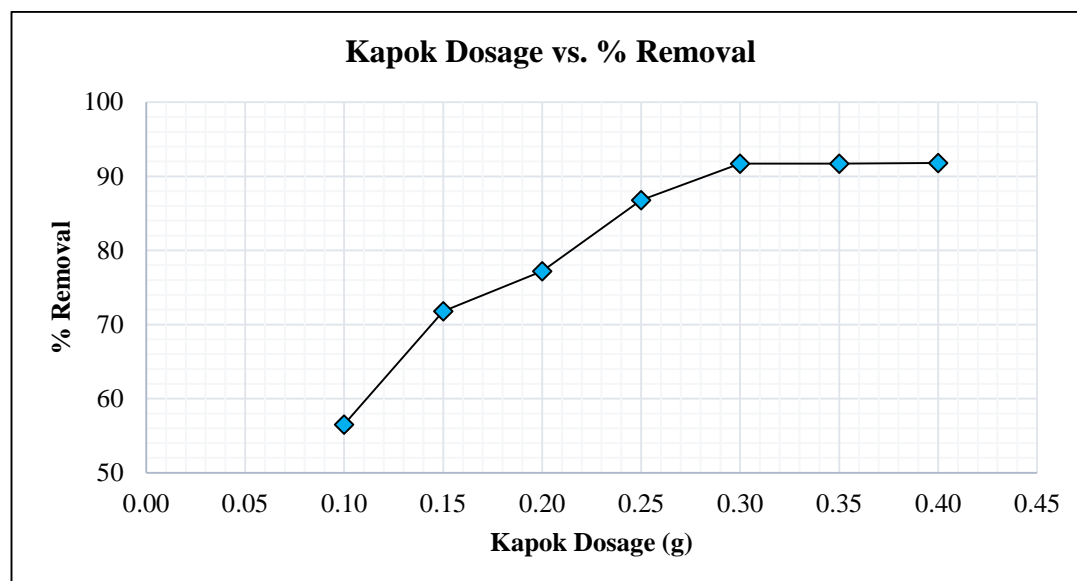


FIGURE 4.6 Graph of Kapok Dosage vs. % Removal

The removal of heavy metals is also dependent on the adsorbent dosage used in the solution. This can be proven from Figure 4.6 where it shows that as more kapok dosage is added to 50 mg/L $\text{Pb}(\text{NO}_3)_2$ concentrated solution, a higher percentage of removal is obtained. The amount of lead removed increased exponentially when we used 0.1 to 0.3 grams of kapok adsorbent but display a constant rate at kapok dosage of 0.3 to 0.4 grams. Hence, it is determined that 0.3 grams of activated surface kapok is the optimum amount for the adsorption.

As the activated kapok adsorbent amount increased, more surface area is available, providing more active sites therefore it allows more interaction with lead ions in the solution. Similar result is also shown in the Rao et al. (2008) report. Their optimum amount of adsorbent is 0.3 g used on 50 mg/L of lead solution. The percent removal became constant after 0.3 g kapok dosage because it already adsorbed all the Pb ions that can be adsorbed and since the solution concentration is kept constant at 50 mg/L, the final concentration, C_1 obtained were almost similar (See Table 4.2).

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This project is important as it deals with the severe issue of water pollution; heavy metals in wastewater treatment. The activated surface kapok is believed to have high selectivity and efficiency for the adsorption of heavy metals in wastewater. In addition, this project is considered to be feasible by taking into account the time constraint and the capability of final year student.

Based on the results of this project, it indicates that activated surface kapok is an effective adsorbent for the wastewater treatment in removing heavy metals especially lead. Analysis derived from each parameter that might influence the effectiveness of activated surface kapok led to the conclusion that:

- i. The optimum pH value for removal of lead using kapok is pH 6.0.
- ii. Equilibrium or optimum contact time is 1 hour for maximum removal of lead using activated kapok adsorbent.
- iii. About 300 mg of activated surface kapok is needed to optimize the adsorbent dosage with the highest amount of lead removed.
- iv. Using 0.3 grams of kapok adsorbent will give an optimum value for initial lead concentration between 40 mg/L to 50 mg/L.
- v. Increase in initial concentration of heavy metal will decline the rate of adsorbent thus adding more adsorbent will improve this problem.
- vi. FESEM analysis verified that there is some structural changes occurred to the chemically modified activated surface kapok compared to raw kapok fiber which can enhance the adsorption rate.

5.2 Recommendations

For future study on similar scope, there are a few recommendations for the project improvements:

- i. Investigate other conditions that might affect the effectiveness of activated surface kapok such as temperature of the heavy metals solution.
- ii. Conduct experiment that tested this parameters using different type of kapok as an adsorbent for example compare using raw kapok and different methods of preparing activated kapok to remove heavy metals.
- iii. Vary the type of heavy metals to be removed using the activated surface kapok to analyze whether kapok adsorbent is effective to any heavy metals not lead (Pb) metal only such as zinc (Zn) and cadmium (Cd).

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APPENDICES

a. Data extracted from the lead (Pb) removal Experiment

Experiment A – The Effect of pH Value				
pH value	R1 C₁	R2 C₁	R3 C₁	Average, C₁ (mg/L)
3.0	11.09	11.12	11.09	11.10
4.0	8.75	8.72	8.78	8.75
5.0	6.88	6.93	6.89	6.90
6.0	3.30	3.27	3.33	3.30
7.0	3.75	3.66	3.71	3.70
8.0	3.94	3.95	3.96	3.95
9.0	4.23	4.26	4.26	4.25
Experiment B – The Effect of Contact Time				
Time (min)	R1 C₁	R2 C₁	R3 C₁	Average, C₁ (mg/L)
20	8.72	8.76	8.77	8.75
40	6.89	6.92	6.89	6.90
60	3.32	3.27	3.31	3.30
80	3.70	3.74	3.66	3.70
100	3.97	3.94	3.94	3.95
120	4.27	4.23	4.25	4.25
140	4.61	4.55	4.64	4.60
Experiment C – The Effect of Lead Initial Concentration				
C₀ (mg/L)	R1 C₁	R2 C₁	R3 C₁	Average, C₁ (mg/L)
40.00	3.42	3.40	3.38	3.40
50.00	4.52	4.51	4.47	4.50
60.00	5.83	5.90	5.91	5.88
80.00	9.17	9.21	9.22	9.20
120.00	15.61	15.61	15.58	15.60
160.00	33.75	33.73	33.80	33.76
200.00	49.78	49.83	49.79	49.80
Experiment D – The Effect of Dosage of Activated Kapok Adsorbent				
Kapok Dosage (g)	R1 C₁	R2 C₁	R3 C₁	Average, C₁ (mg/L)
0.10	21.73	21.74	21.78	21.75
0.15	14.11	14.11	14.08	14.10
0.20	11.44	11.40	11.36	11.40
0.25	6.59	6.58	6.63	6.60
0.30	4.14	4.13	4.18	4.15
0.35	4.17	4.16	4.12	4.15
0.40	4.07	4.11	4.12	4.10

b. Calculation of the Average C_1

The equation for the average calculation is:

$$\text{Average } C_1 = \frac{R1 + R2 + R3}{3}$$

Where R1, R2 and R3 and Reading 1, Reading 2 and Reading 3 for each Experiment A, B, C and D repeated, respectively. The average C_1 values are then used in the Equation 1, and the obtained values are tabulated in Table 4.2 in the report.