ADSORPTION OF HEAVY METAL IN WASTE WATER BY $NACLO_2$ TREATED KAPOK FIBER

By

Muhammad Afham Bin Mohd Ali

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

MAY 2014

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 unspecified sources or persons.

(MUHAMMAD AFHAM BIN MOHD ALI)

ABSTRACT

Heavy metal pollution in water is an important issue of water pollution. Heavy metal pollution not only influences the quality of the water body, but also affects the production and quality of the crops and aquatic products, and threatens the health and life of animals and human beings through the food chain. Due to harmful effects of heavy metal ions pollution in water, there is a pressing need to find efficient methods to combat this kind of pollution. Kapok fiber, or scientifically named as *Ceiba Pentandra* (L.) *Gaertn* is a natural hollow fiber with thin shell and large cavity, has rarely been used as adsorbent for heavy metal ions. Kapok fiber is a naturally renewable material with large lumen and hydrophobic characteristics, which enables it to show good oil sorption capacity. This research project evaluates and compares the performance of different type kapok fibers in application of removing heavy metals. To further improve the efficiency for adsorption, in this study, kapok fiber was treated with the solvent of Sodium Chlorite ($NaClO_2$). The structure of untreated and treated kapok fibers was investigated and compared using Fourier transform infrared (FTIR) spectroscopy and Field–Emission Scanning Electron Microscope (FESEM). In addition, the sodium chlorite treated kapok fiber exhibits at least 8% better absorbency compare to the raw kapok fiber, suggesting its great potential for heavy metal removal.

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"Praise to Allah, the most Gracious and the most Merciful"

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CHAPTER 1

PROJECT BACKGROUND

1.1 Background Study

The major potential environmental impacts related to landfill leachate are pollution of groundwater and surface water. Landfill leachate contains pollutants that can be categorized into four groups (dissolved organic matter, inorganic macro components, heavy metals, and xenobiotic organic compounds) (Kjeldsen P, et al. 2002)

Sanitary landfill leachate is a high and complex polluted wastewater. Leachate pollution is the result of biological, chemical and physical processes in landfills combined with waste composition and landfill water regime. A characteristic of this wastewater is the change of some components with the change of biological conditions in landfills (Heyer and Stegmann).

There are three reaction in landfill which are biological reaction, chemical reaction, and physical reaction. Biological reaction is aerobic and anaerobic degradation of organic waste. Chemical reaction is dissolution and evaporation of material, dehalogenation and decomposition of organics, redox of metal. Physical reaction is movement of gas and leachate in the landfills.

A solid waste landfill can be conceptualized as a biochemical reactor, with its unique inputs and output partially biodegraded organic material and other inorganic waste materials stored in the landfill. Landfill gas control systems are a necessary activity.

Leachate is the liquid that is percolated through solid waste. Leachate is composed from external source such as rainfall and liquid produced from waste decomposition. Composition depends on landfill age. New landfill (<2 years) leachate has high organic, highly biodegradable (BOD/COD>0.5), low pH (acid phase decomposition). Mature landfill (>10 years) leachate has lower organic, low biodegradable (BOD/COD<0.2), higher pH (methane fermentation).

Many materials that end up as waste contain toxic substances. Over time, these toxins leach into our soil and groundwater, and become environmental hazards for years. Electronic waste is a good example. Waste such as televisions, computers and other electronic appliances contain a long list of hazardous substances, including mercury, arsenic, cadmium, PVC, solvents, acids and lead.

1.2 Problem Statement

The presence of high percentage of heavy metal in leachate has polluted fresh water sources and this has resulted in a need for the development of several new materials with potential for the removal of heavy metal from leachate.

Kapok fiber exhibited good water repellency, high oil adsorption capability, and well reusable characteristic, demonstrating its potential as an alternative for application in oil pollution control. However, oil absorption mechanism, the contribution of hollow lumen and surface wax on the oil absorption capability of kapok fiber still cannot be well recognized.

1.3 Objective

1) To explore on modifications and characterization kapok fibers for heavy metal removing study.
2) To optimize sorption of heavy metal in polluted sample study
3) To evaluate and compare the performance of raw and modified kapok fibers in removing heavy metal

1.4 Scope of Study

The scope of the study involves preparation of two different types of kapok which is raw kapok fiber and sodium chloride ($NaClO_2$) treated kapok fiber. The characteristic of kapok fibers after treatment is studied. Each of kapok fibers will be characterized after the preparation using for the morphology and chemical analysis using Scanning Electron Microscopy and Spectrum One FTIR respectively. The comparative study on the sorption of different kapok fibers will be studied in solution with different concentration

CHAPTER 2

LITERATURE REVIEW

2.1 The Contents of Landfill Leachate

The compounds may be found in leachate from landfills: for example, borate, sulfide, arsenate, selenite, barium, lithium, mercury, and cobalt. However, in general, these compounds are found in very low concentrations and are only of secondary importance. Leachate composition may also be characterized by different toxicological tests, which provide indirect information on the content of pollutants that may be harmful to a class of organisms (Kjeldsen et al., 2002).

Figure 1: Landfill leachate before treatment

The initial leachate characterization study was conducted to determine the most significant heavy metals that will be the parameter of interest. The results of initial leachate characterization study are manganese (Mn), nickel (Ni), calcium (Ca), magnesium (Mg), zinc (Zn), iron (Fe), copper (Cu), chromium (Cr), cadmium(Cd), aluminium (Al), and lead (Pb) (Kamarudzaman et al., 2011).

Leachate production is the result of precipitation, evaporation, surface runoff, infiltration, storage capacity etc. Under humid climatic conditions the average difference between precipitation and evaporation also with different vegetation covers is positive (in Germany and many other parts of Europe). Beside evaporation the infiltration could be reduced by surface runoff. But preventing of erosion problems needs a limitation of surface runoff. In many Northern German Landfills the measured leachate flow rates are often between 12 and 22 % with 18 % as mean value of precipitation. Values lower than 10% are from very young landfills. Values above 25% of precipitation are from landfills where the end of storage capacity can be observed. Beside storage fill up another process of water consumption is the anaerobic biological degradation with water consumption of the biological process and water transport by gas. Over long time both processes - storage fill up and biological water consumption – must decrease. The result must be an increase of leachate flow up to a value of precipitation minus evaporation.

To control long-term leachate flow and reducing treatment costs the reduction of infiltration could be helpful and sometimes necessary (especially for old landfills without bottom sealing). The strategy for water input control is strictly related to the quality of the waste to be landfilled. In the case of non-biodegradable waste and according to its hazardous potential for the environment, prevention of water infiltration can be adopted as the main option (normally by means of top sealing). On the contrary, in the case of biodegradable waste, a water input must be assured until a high degree of biostabilization is achieved. In this case the water input should be limited to the strictly necessary amount and minimization techniques should be applied (Heyer and Stegmann).

2.2 Characteristic of Kapok

Among of those natural materials, kapok has the advantages over traditional oil absorbing materials: low cost, biodegradability, intrinsic hydrophobic characteristic and high sorption capacity and accordingly, they are preferable as an oil-absorbing material. In these studies, kapok fiber exhibited good water repellency, high oil adsorption capability, and well reusable characteristics, demonstrating its potential as an alternative for application in oil pollution control (Wang et al., 2012).

Figure 2: Kapok tree

The kapok fiber exhibits a rather thin cell wall with a huge hollow lumen full of air, and thus exhibits a low density, high bulkiness, good oil absorptivity and water-repellent nature. Water cannot easily penetrate into the lumen for the presence of negative capillary entry pressure due to the large contact angle (>90_) between water and kapok fiber wall, and the large surface tension against air in the lumen. Furthermore, waxy cutin on the kapok fiber surface makes it water repellent notwithstanding it is mainly composed of cellulose. Compared to cotton fiber, the kapok fiber exhibits a lower content of cellulose and a higher content of lignin (Liu et al., 2012).

Figure 3: Raw kapok fiber

Malaysian Kapok (Ceibapentadra, Gaertn.) is commonly found in northern parts of peninsular Malaysia. The fruit soft his 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 and cushions. The seeds are normally discarded. In rural areas, however, the seeds are roasted and consumed after removing the husk. Sometimes they are germinated prior to use. Apparently they often upset the stomach and hence they are consumed only in small quantities. Georgi (1922) reported that the oil content of Kapok seeds, from different parts of Malaysia is in the range of 20 to 25 percent (Berry, 1979).

2.3 Kapok as natural sorbent

Ceibapentandra (L.) Gaertn or known as kapok is an agricultural product. Its fiber is lustrous, yellowish-brown in color which derived from the fruits of silk-cotton tree mainly composed of cellulose, lignin polysaccharide and small amount of waxy coating. To date, kapok fiber have received great attention as a natural sorbent material due to its hydrophobic – oleophilic characteristics. The kapok fiber has a hollow structure with large lumen which contributed to its great oil absorbency and retention capacity. Apart from that, the high amount of waxy cutin on the kapok surface has also contributed towards its high water repellency (Abdullah et al., 2010)

Table 1: Chemical composition of kapok fiber (Karan et al., 2011)

Chemical composition	Percentage
Cellulose	64%
Lignin	13%
Pentosan	23%

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 ± 6.5µm. The raw kapok fiber showed smooth surfaces with density of 1.3g/cm3. Besides, the oil sorption and retention capacity of kapok fibers was affected by its physicochemical characteristics such as surface wax, molecular arrangement, physical configuration and porosity (Abdullah et al., 2010).

The plant wax is the main contributor to the water repellency and solubility. Kapok fiber contain high wax content of about 3% compared to cotton and high acetyl content where it was found the it has contributed to its high hydrophobicity (Abdullah et al., 2010). In some research, esterification as chemical modification has been used in increasing the hydrophobicity of fibers to enhance the oil sorption capacity. For example the modification that has been applied for sawdust, rice husk, bagasse and banana trunk has proved the improvement in their sorption capacity (Wang et al., 2013)

There are many types of fibers or modified fibers, including polyester, polyacrylonitrile, polypropylene, and so on, which have thus far been used for the adsorption of heavy metal ions, or to enrich trace amounts of heavy metals from aqueous solution (Chung et al., 2008). All of these techniques make possible moderate to efficient heavy metal removal. However, as the result of their ready availability and cost effectiveness, biological waste materials including starch, straw, saw-dust, peat moss, sugar cane pulp, coconut hulls, sheep manure waste, groundnut shell and sawdust, jute fiber, activated carbon fiber, thiol cotton fiber as well as other materials, have attracted the attention of investigators into adsorption and ion exchange removal (Chung et al., 2008).

Kapok fiber, which is traditionally used as fiberfill in pillows, quilts, and some soft toys, is quite fine (ca. 8~10 μm diameter), and has a homogeneous hollow tube shape, with a wall thickness of ca. 0.8~1.0 μm. In addition, this material is significantly hydrophobic, and does not become wet in water, as the result of its high surface tension (7.2×10−4 N cm−1 at 20oC against air) and chemical characteristics, including a high ratio of syringyl /guaiacyl units (4~6) and a high level of acetyl groups (13.0%) as compared with normal plant cell walls about 2~4% (Hori et al., 2000). In the present study, we utilized chemically oxidized kapok fibers for the adsorption of heavy metal ions, including Pb, Cd, Cu, and Zn, from aqueous solutions in order to alter the hydrophobicity of this material to hydrophobicity.

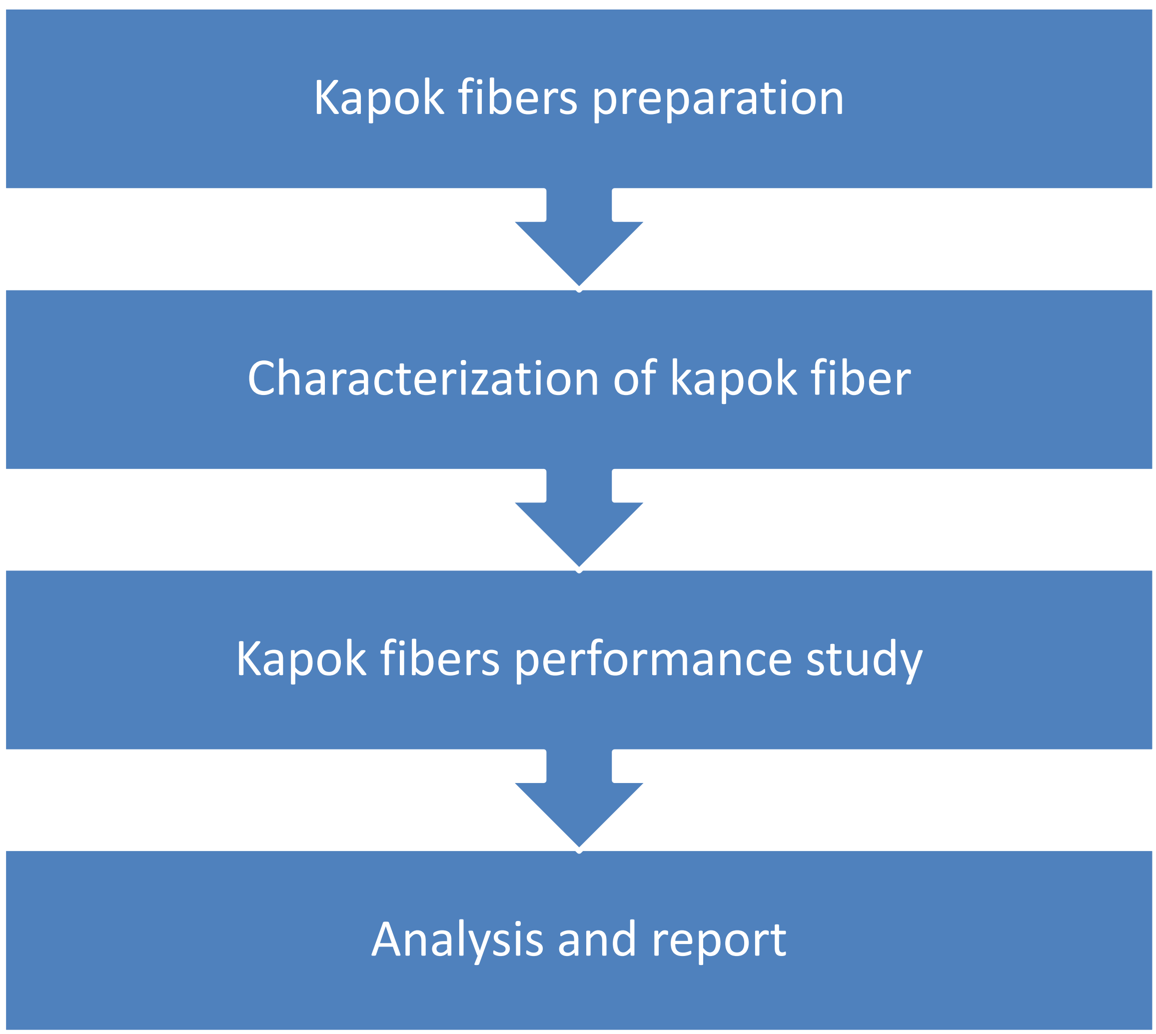
2.4 Solvents For Modified the Kapok Fiber

The physico-chemical properties of kapok fibers were altered via the combination processes of chlorite-periodate oxidation using sodium chlorite, in order to assess their efficacy as a heavy metal adsorbent. The chemically-oxidized kapok fibers were found to harbor a certain amount of polysaccharides, together with lowered lignin content. This alteration in lignin characteristics was clearly confirmed via Fourier transform infrared (FTIR) and Field–Emission Scanning Electron Microscope (FESEM). Moreover, chemically oxidized kapok fibers retained their hollow tube shape, although some changes were noted. The chemically oxidized kapok fibers evidenced elevated ability to adsorb heavy metal ions with the best fit for the Langmuir adsorption isotherm model (Chung et al., 2007).

CHAPTER 3

METHODOLOGY

3.1 Project Flow Chart



3.2 Experiment Methodology

The methodology if this research is based on experimental work where two different types of kapok fiber will be prepared including raw kapok fiber and sodium chlorite treated kapok fiber. In this study, raw kapok fiber and sodium hydroxide treated kapok fibers will be denoted as SKF and TKF. For the performance study, these two types of the kapok fibers will be use and the measurement as well as evaluation on the sorption capacity and efficiency will be conducted. Ultraviolet–visible (UV-VIS) spectroscopy and Field–Emission Scanning Electron Microscope (FESEM) would be used for the characterization to compare and contrast the structural and composition of the raw and modified kapok fibers.

The steps for conducting the experiment are;

3.3.1 Kapok fibers preparation

Different types of kapok fibers will be prepared for the research project

- Raw kapok fiber
- $NaClO_2$ treated kapok fiber

<u>Raw kapok fiber</u>

Raw kapok fibers are cut into a small shape using cutting tool

<u>$NaClO_2$ treated kapok fiber</u>

1) 25g kapok fibers were treated in 400ml $NaClO_2$ (10g) and concentrated acetic acid (2mL) for 1 hour at 70-80 ºC in order to break the phenolic compounds.
2) Kapok fibers after the $NaClO_2$ treatment were suspended in H_2O until the pH of the filtrate reach neutrality.

3) These kapok fibers were dried overnight in a vacuum oven at 40 ºC.

Kapok Performance Study

1) The treated kapok fiber was utilized in the adsorption experiment. One gram of the materials was shaken at 30^{o}C in a flask containing 50 mL of the heavy metal ion solutions for a predetermined equilibrium time of 150 min.
2) The material was then filtered, and the residual solution was analyzed for the estimation of heavy metal ion concentrations, using an inductively coupled plasma-atomic emission spectrometer.
3) All experiments were repeated five times, and blank experiments were also conducted in order to ensure that no adsorption occurred on the walls of the apparatus used.

3.3.2 Tools and Equipment

For confirming alteration in lignin characteristics, Fourier Transform Infrared (FTIR) spectroscopy and Field Emission Scanning Electron Microscope are needed. To dry the fibers, vacuum oven is used. Others equipment needed are measuring cylinder, thermometer, flask, reflux condenser and stirrer.

3.3.3 Substances and Chemicals

The substances needed are kapok fibers and copper nitrate solution ($CuNO_3$) landfill leachate. The chemicals that will be used in the experiment are NaClO2, acetic acid, and acetone.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characterization of Kapok Fiber

Raw kapok fiber (RKF) and sodium nitrate treated kapok fiber (TKF) physical properties were characterized based on morphology. The fibers morphologies were analyzed using Field- Emission Scanning Electron Microscope (FESEM). The SEM microphotographs of SKF and TKF are shown in Figure 4 below.

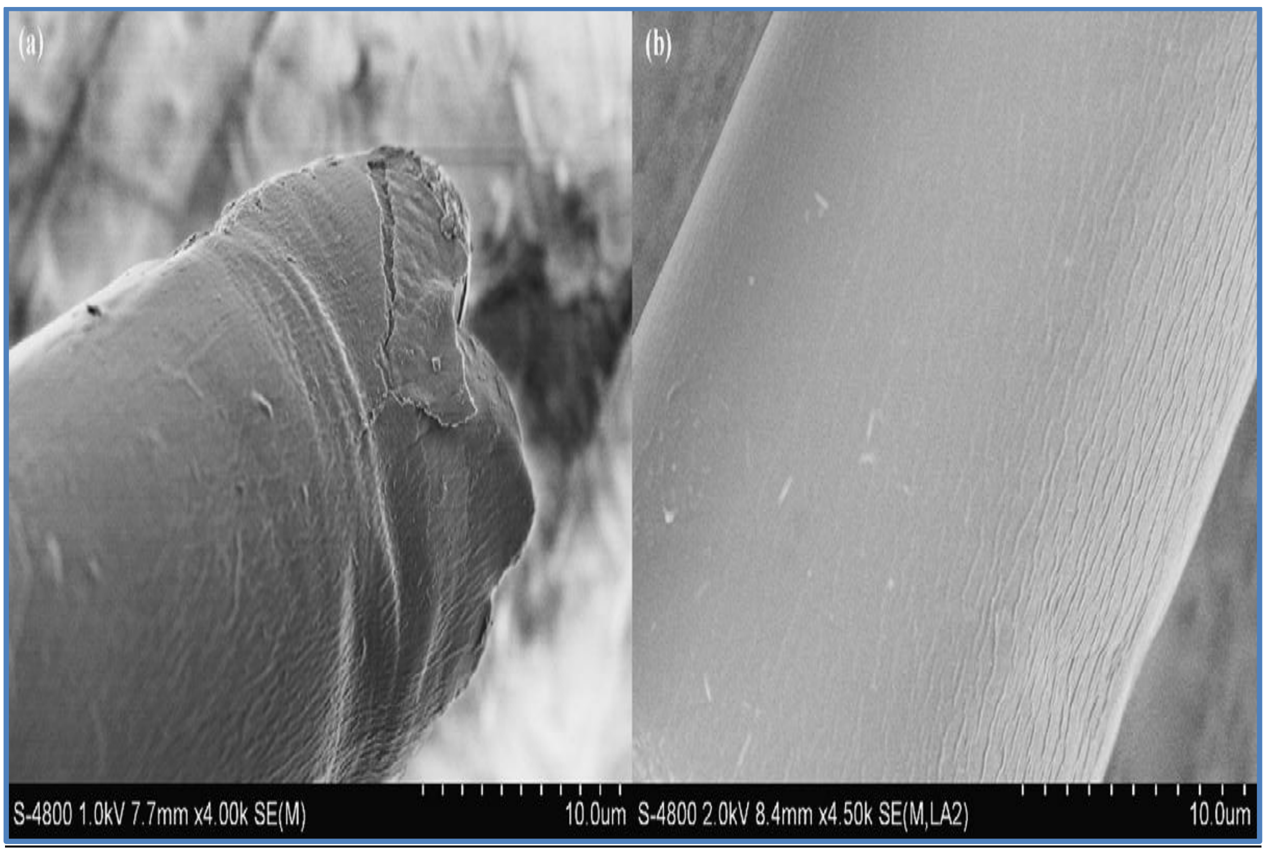


Figure 4: SEM micrograph of SKF (a) and TKF (b)

From the Figure 4(a), it is obvious that raw kapok fiber (RKF) has a hollow tubular structure or lumen and smooth surface area without any ripple. The smooth surface exhibit by the RKF is concluded due to the plant wax coverage on the surface (Wang et al., 2012) and the structure of the fiber prove the buoyant features exhibit by the kapok fibers and the use of kapok fibers as stuffing materials (Praba Karan et al.,2011). However in according to Figure 4(b), the structure of the sodium chlorite treated kapok fiber (SKF) has shown a significant different as compared to raw kapok fiber (RKF). The hollow structure is no longer exhibit by the fibers and it seems that the fiber has been compressed with no tubular configuration as well. This confirmed that the alkaline treatment of the kapok fibers damaged the structure and caused the collapse of the hollow tube. The significant different of these kapok fibers suggest the removal of the plant wax and the hydrophilic surface has been exposed after the treatment (Wang et al., 2012; Abdullah et al., 2010).

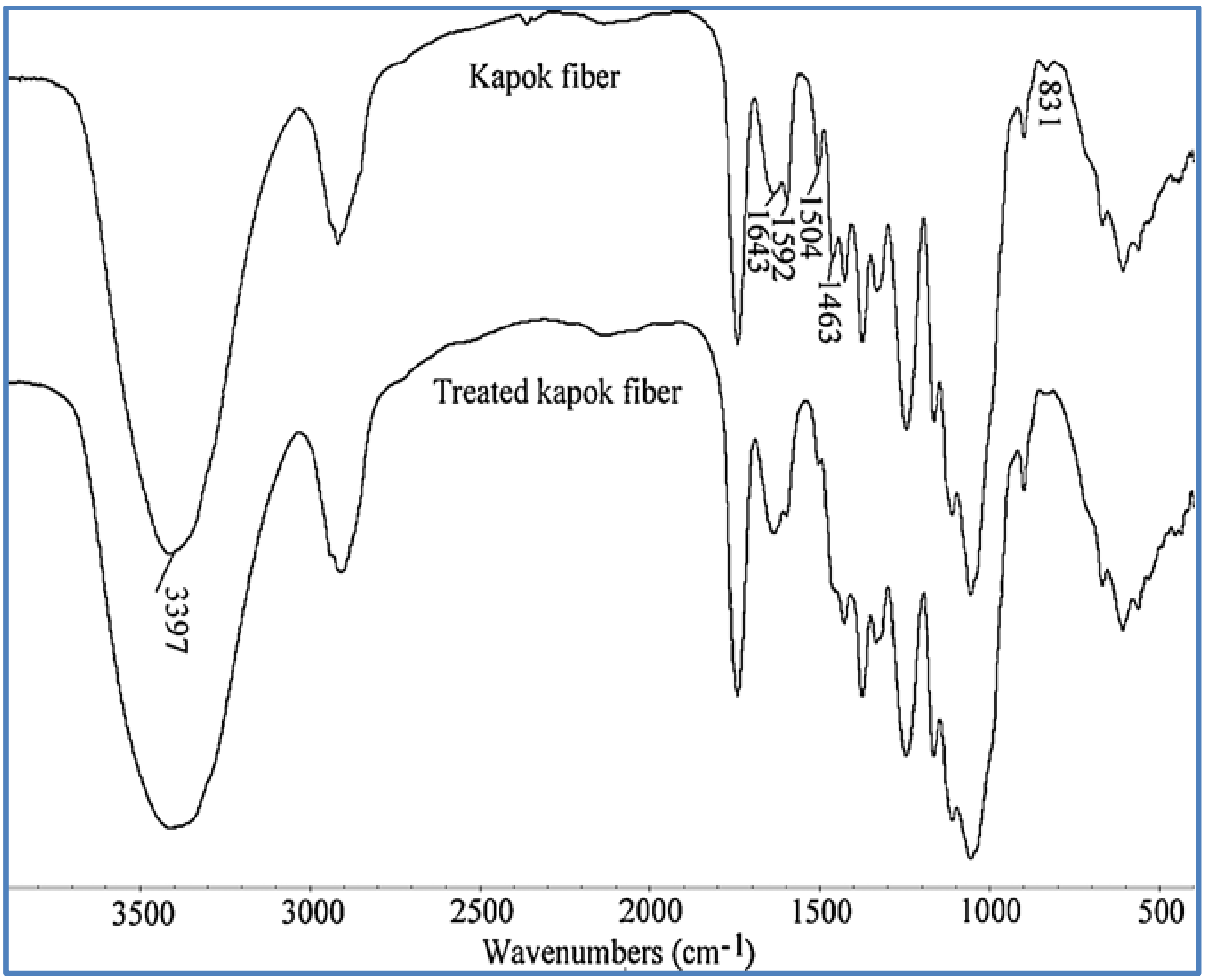


Figure 5: Comparison of FTIR spectra of kapok fibers before and after treatment with NaClO2

FTIR spectra are commonly used to identify the groups involved in the reaction process and thus the FTIR spectra of raw and treated kapok fiber are investigated and shown in Figure 5. The spectra of both kapok fiber show a similar pattern. In despite of similar pattern in raw kapok fiber and sodium chlorite treated kapok fiber, several findings have been observed like the disappearance of a few of peaks and the reduction of the intensity at each peak in the sodium chlorite treated kapok fiber and Table 2 shows the assignments absorption of infrared band of typical cotton which comparable to this study and has been used as reference to this study (Adebajo., 2004; Mwaikambo et al., 2002; Wang et al., 2012).

The broad band at about 3397 cm−1 ascribed to the stretching vibration of OH in cellulose, became broader after treatment, which may be due to part of hydrogen bonds and lignin was broken, thus leading to increase in the amorphous part in cellulose and release of more hydroxyl groups. The band at 1643 cm−1 may correspond to bending vibration of the water molecules and it had almost no change after treatment. The absorption bands at 1592, 1504 and 1463 cm−1, were corresponding to stretching vibration of C-C in different substituted aromatic rings in lignin, and almost disappeared. The absorption band at 831 cm−1, ascribed to the wagging vibration of C H in 1,4-disubstituted aromatic ring in lignin, almost disappeared after treatment. All the information indicated that lignin in the kapok fiber was broken by the treatment.

Furthermore, it can be also verify by the fact that the treated kapok fiber exhibited good hydrophilic property as it was fully wet after in distilled water compare to the raw kapok fiber which was always floated on the distilled water.

Table 2: Assignments of Infrared absorption bands (Adebajo., 2004; Mwaikambo et al., 2002; Wang et al., 2012)

Frequency (cm-1)	Assignment
3337	OH stretching
2891	CH stretching of CH2 and CH3 groups
1742	Carbonyl C=O stretching ester
1420	OH and CH2 bending
1368	C-H bond in –O(C=O)-CH3 group
1337	CH3 bending or OH in-plane bonding
1312	O-H deformation and/or CH2 wagging
1234	C-O stretching of acetyl group
1203	OH in-plane bending
1157	C-O-C anti-symmetric bridge stretching in cellulose and hemicelluloses
1017	C-O stretching in cellulose, hemicellulose and lignin
609-668	-OH
467	Symmetric stretching vibration of Si-O-Si

4.2 Kapok Performance Study

Based on Wang et. al., (2012), the kapok fiber absorbency is determined by;

$$Q = \frac{m_2 - m_1}{m_1}$$

where m_1 and m_2 are the weight of kapok fiber before and after absorption respectively.

Table 3: Result for kapok performance study

	Experiment 1		Q	Experiment 2		Q	Experiment 3		Q
	m_1 (g)	m_2 (g)		m_1 (g)	m_2 (g)		m_1 (g)	m_2 (g)	
RKF	5.03	7.36	0.46	5.08	6.98	0.37	4.98	7.16	0.44
TKF	5.11	7.88	0.54	5.01	7.51	0.50	5.15	7.80	0.52

The tabulated data in Table 3 is further analysed as per Figure 6 below and it is observed that there are not much difference between both raw kapok fiber and treated kapok fiber in term of the absorbency. As comparison, the absorbency of the treated kapok fiber shows the higher value compared to raw kapok fiber. This indicates that the sodium chlorite treated kapok fiber has the best performance in heavy metal adsorption as compared to raw kapok fiber. Abdullah et al. (2010) suggest that the absorbency of treated kapok fiber increased after the treatment as the result of removal of wax from the kapok surfaces. This was supported by Lim, T., (2007) where he reported that the interactions and van der Waals forces between the oils and the wax on the kapok fiber initiated the absorption mechanism of oil into the hollow lumen. It was further supported by Wang et al. (2012) which described that the alkalization can change the fine structure of kapok fiber where the hollow lumen to store the oils disappears partially or completely and the damaging effect on tubular structure which making the surface "rougher".

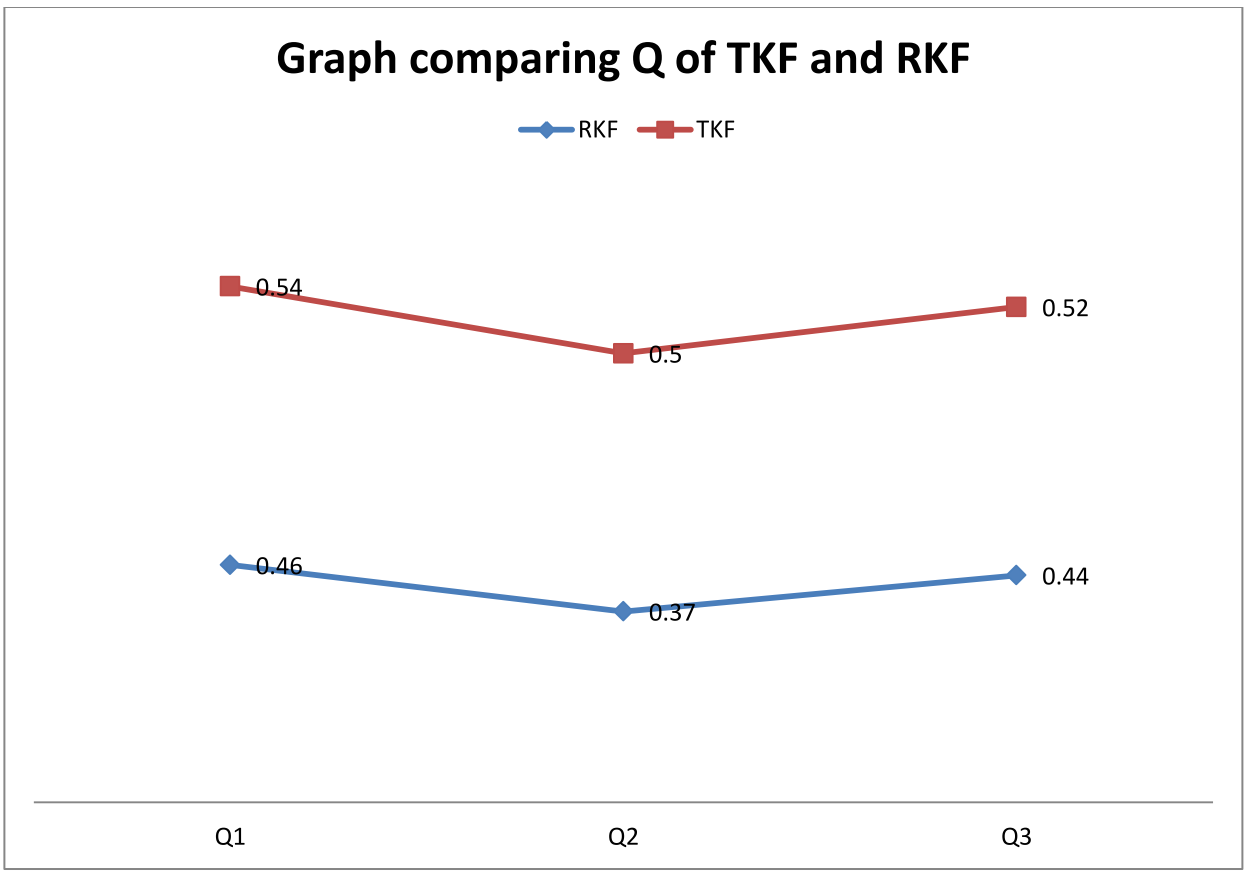


Figure 6: Absorbency of RKF and TKF

In this treatment, sodium chlorite alters the hydrophobicity of this material to hydrophilic. In addition, sodium chlorite modifies the chemical series inside kapok fiber. During adsorption, Na is released into the solution and replacement of H+ ions for heavy metal ions from the material, resulting in the reduction of pH. In other journal, (Wang et al., 2012) investigated the surface modification on the kapok fiber known as superhydrophobic which prepared by incorporation of silica nanoparticles onto the kapok fiber via sol-gel method. As a result, the oil/water selectivity has improved and the absorbency has become higher. This study conclude that the prepared fiber is promising as candidate for the replacement of heavy metal sorbent and applied in large scale removal of heavy metal in waste water.

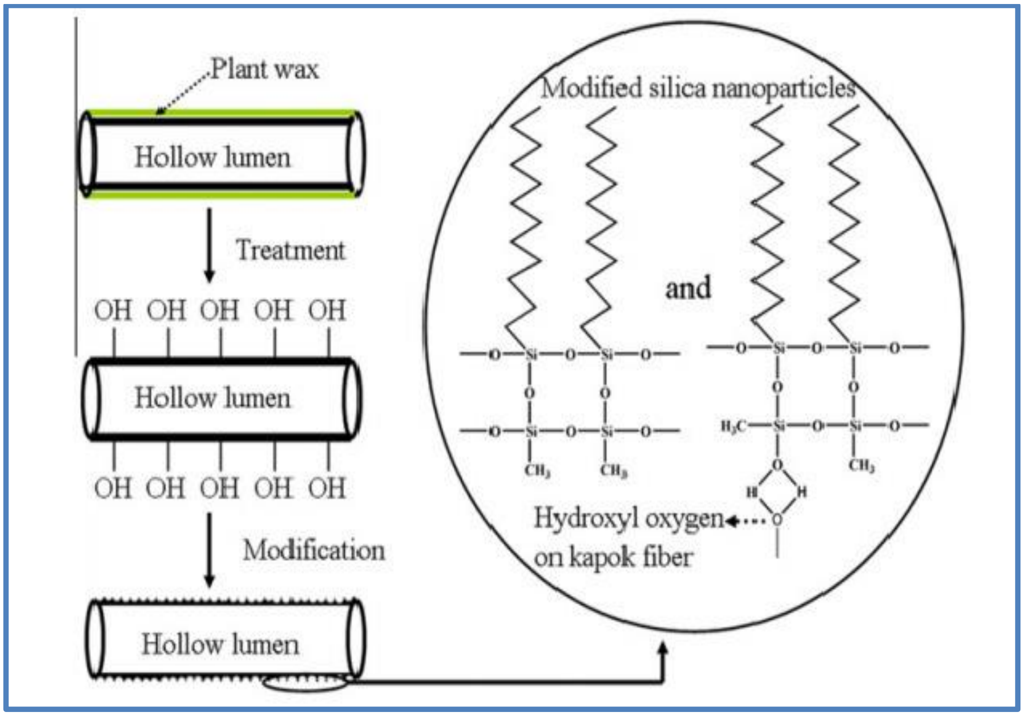


Figure 7: Schematic representation of transition from raw kapok fiber to superhydrophobic kapok fiber (Wang et al., 2012)

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

This project is a comprehensive study about the effectiveness and ability of Sodium Chlorite Treated Kapok Fiber to adsorb heavy metal in the polluted water for wastewater treatment. With many studies and researches on the modification of kapok fibers that have been conducted for further improvement, a comparative study is very significant in determining the best fibers for effective and systematic heavy metal removal.

From the research done, treated kapok fiber is found to be efficiently extracting heavy metals such as Zn, Pb, Cr, Cd, and CU. High heavy metal adsorption, excellent reusability, and good biodegradability make treated kapok fiber a suitable alternative to remove the heavy metal from the leachate as well as low cost and easy to get.

Treated kapok fiber (TKF) shows greater absorbency than the raw kapok fiber (RKF). The fiber which has been treated show at least 8% better absorbency compare to raw kapok fiber. The treatment will transform the fiber from hydrophobic to hydrophilic which will help it boost its adsorption ability. In addition, the roughness of surface of the kapok after being treated plays a big role in increasing its absorbency. The sodium chlorite treated kapok fiber experienced a huge structural disruption and significant chemical constituent changes which related to plant wax remove after the treatment.

In conclusion, *Ceiba pentandra* (*L.*) *Gaertn* (Kapok) is a promising natural sorbent for waste water treatment treating kapok fiber will increase its ability to adsorb heavy metal in waste water.

5.2 Recommendations

This study has proved the high performance of kapok fibers as sorbent of heavy metal. Thus, treated kapok fiber which has shown greater absorbency should be further investigated for commercial and large scale application.

Other than that, another parameter includes packing configuration which the combination of different types of kapok fibers and treatment time should be further study in order to evaluate the performance. The parameters to achieve an optimum performance should also be evaluated like the temperature and the dimension for the kapok packing assembly. Besides, the kapok fiber should also be evaluated for the sorption of other type of effluent or waste such in order to widen the application of this natural sorbent

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APPENDICES

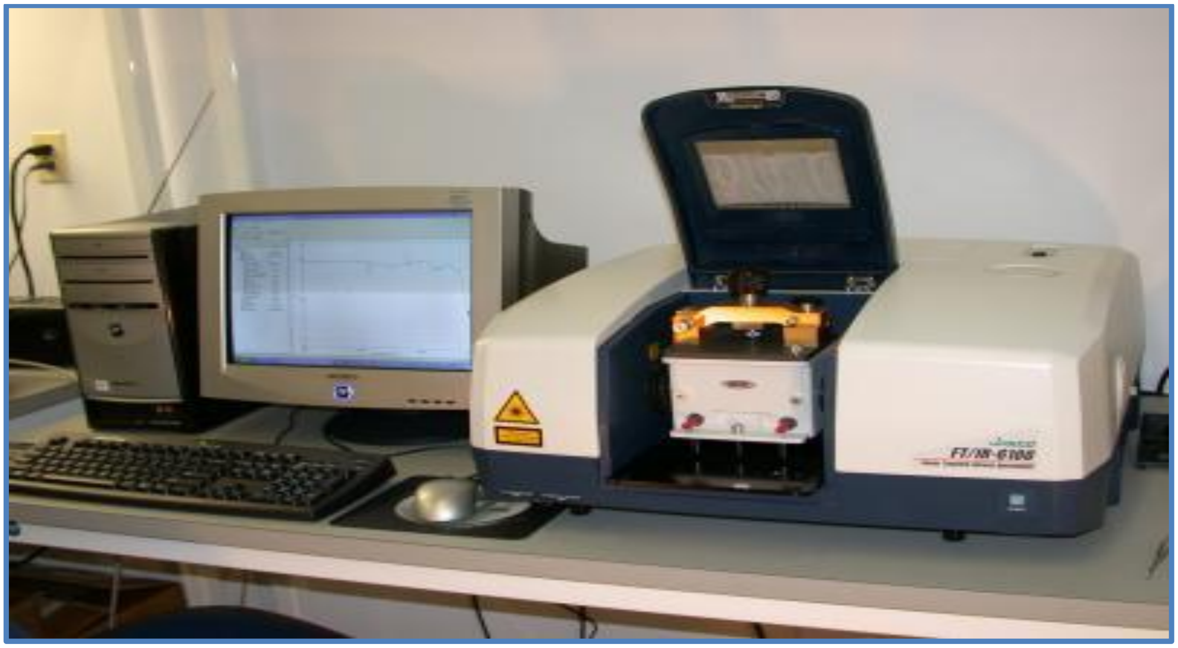

Figure 8 : Fourier Transform Infrared Spectroscopy machine

Figure 9 : Field Emission Scanning Electron Microscopy machine