Investigation of modified kapok fibre on the sorption of oil in water

by

Kalisvaran Muniandy

14995

Dissertation submitted in partial fulfillment of

the requirement for the

Bachelor of Engineering (Hons)

(Chemical Engineering) 2015

APRIL 2015

Supervisor: A.P. Ir. Abdul Aziz Omar

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CERTIFICATION OF APPROVAL

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TRONOH, PERAK

APRIL 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 unspecified sources or persons.

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Kalisvaran Muniandy

ABSTRACT

In despite of rapid oil and gas industry development, a major problem has been faced by the world is oil spill. During transportation of crude oil, accidents or unwanted events may happen thus causing catastrophic incident of oil spilling over ocean causing huge amount of loss and damage to environment. Currents methods of responding to oil spills are mostly to preventing the oil from damaging the ecosystem and making sure the oil is disintegrated or removed quickly as possible from the exposed environment of ocean. However, this existing practical methods does not recover the oil from ocean but merely preventing further damage to ocean and its ecosystem. In this study, Kapok fibre or scientific name *Ceiba Pentandra* – an agricultural product which has excellent water repellency will be investigated on its oil absorbency. This fibre naturally has hydrophobic and oleophilic character which allows it to selectively absorb oil from water without absorbing any water. This study will be focused on studying the modified Kapok fibre and their performance on oil absorbency. Modification is an act of changing the Kapok's fibre structure using chemical. Chemical that was being used in this study are Sodium Hydroxide and Sodium Chlorite. Investigation was conducted over the Kapok fibre to find out how does the modification affects the sorption of oil in water. Tests such as Absorbency test, Reusability test and Optimum Time test were conducted in order to prove that modification has effects on the Kapok fibre oil sorption. From the findings, Kapok fibre has shown promising future in being sorbent material for boomers to be used in oil spilled clean up.

ACKNOWLEDGEMENT

I have taken a great deal of endeavours amid these two semesters to finish the Final Year Project. Then again, it would not been conceivable without the kind direction and backing of people for this undertaking. Along these lines, I also want to express profound appreciation to my supervisor, A.P. Ir. Abdul Aziz Omar for his direction, remarks and patience during this project.

I am highly indebted to Ms. Kritheka and Mr. Ikhmal who are researchers at Veterinary Research Institute (VRI). They provided me with sufficient kapok that I need for this project and they gladly gave the materials for free which saved a lot of cost. Much thanks and I truly admire each help that has been given to me. I am as ever, particularly obligated to my family for their help and trust for the duration of my life. Aside from that, incredible gratefulness was given to my companions who continually giving their backing and inspiration all through the semesters.

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CHAPTER 1

INTRODUCTION

1.1 Project Background

Oil spill in the ocean has been always a threat to the environment and the oil companies where they are bound to cleaning the oil spill which cost them millions. Oil spill accident is not a new issue that causes damage to nature. For instance, in March 1989, the incident where Exxon Valdez spilled 11.2 million gallons of raw petroleum into coastal water of Prince William Sound, Alaska bringing on extreme ecological harm (Teas et al., 2001). The obliteration of oil stockpiling tanks in Kuwait, 1991, amid the Gulf war resulted in 4-inch thick oil spill that spread over 4,000 square miles in the Persian Gulf (Moss, 2010). The oil slick episodes which add to the oil contamination would influences in numerous angle including ocean life, economy, tourism and recreation exercises.

Currents methods of responding to oil spills are mostly to preventing the oil from damaging the ecosystem and making sure the oil is disintegrated or removed quickly as possible from the exposed environment of ocean. Therefore, methods such as dispersants and biological agents such as microorganisms are used to speed up the degradation process of the oil. Famous way of handling oil spills will be boomers and skimmers where boomers acts as a container or passage blocker and the mechanical skimmers collect the most oil from the surface. The boomer contains absorbents such as hair, saw dust or synthetic products.

In this study, modified Kapok fibre will be studied in order to find out whether they are a suitable replacement as a sorbent material in recovering the oil from ocean. If Kapok fibre shows good result then it can be suggested for boomers to have Kapok fibres as their ingredient whereby this could mean oil spilled into ocean are not totally wasted but recovered and reused.

Kapok fibre also known as *Ceiba Pentandra* is an agricultural product, has a waxy property which is a reason for its hydrophobic-oleophilic character. This fibre is weighless, fluffy, non-toxic, odourless if not been used, non-allergic and non-toxic. It has always been indulged in making of life saving equipment concerning water as it has excellent buoyancy and can support up to 20 times its own weight on water. It exhibits a very water repellent character and favours oily subject though it might decrease slightly in its water repellent ability if being immersed in water for days.

1.2 Problem Statement

In recent years, oily water in inland waterways and coastal zone has become one of the most serious issues of water pollution, and one of the pollution control measure is to remove oil from wastewaters produced by various industries. Oils in wastewaters can exist in free, dispersed or emulsified form. Conventional methods such as gravity separation, dissolved air flotation, coalescence, centrifugation, flocculation and coagulation either fail to remove the oil cost-effectively to meet discharge standards or contaminate the oil such that it cannot be recycled. Membrane filtration, for example, microfiltration and ultrafiltration are in fact a standout among the best options for oily water detachment. Then again, the costly capital and working expense has been the prevention to the wide application of the membrane filtration advances. Regardless of the possibility that the application of membrane filtration of oily water is reasonable, pre-treatment of the oily water is frequently needed for forestalling untimely film fouling.

Kapok fibre shows great water repellence behaviour, high oil adsorption capacity, and well reusable trademark, exhibiting its potential as an option for application in oil contamination control. In addition, Kapok fibre might have advantage over most of the oil spill because it can be mechanically pressed out to recover the oil simply because it does not contain water due to its hydrophobic character. Then again, oil absorption mechanism, the role of empty lumen and surface wax on the oil retention capacity of kapok fibre still can't be generally perceived and whether the oil absorbed by the Kapok fibre will contain any traces amount of water is still questionable.

1.3 Objectives

The objectives of this study are:-

i) To prepare activated kapok for oil removal using NaOH and $NaClO_2$

ii) To find the impact of NaOH and NaC lO_2 on the kapok oil absorbency

iii) To find the reusability of kapok fibre and optimum time for kapok to be exposed in oil spilled ocean.

1.4 Scope of Study

The scope of study involves preparation of modified Kapok fibre using chemicals and it will be subjected to test in terms of oil absorbency, life cycles and optimum time to be exposed in water. Two chemicals that will be used to treat kapok in this are Sodium Hydroxide and Sodium Chlorite. Meanwhile, diesel will be used as the oily subject in this study where diesel will be representing low-viscosity oils such as light crude oil, kerosene and gasoline. Raw kapok fibre will be characterized using Field Emission Scanning Electron Microscope (FESEM).

1.5 Relevancy and Feasibility of the Project

This study emphases on optimization and modification of Kapok fibre via chemical treatment method. Boomers has been also stuffed with hair, sawdust or synthetic material as absorbent. Kapok fibre inside boomers can bring a change to this conventional method. It is important to determine the reusability of the kapok and how kapok will perform on real problem situation.

This study is within capability of a final year student to be executed with help and guidance from the supervisor and the coordinator. The time frame is also feasible and the study can be completed within the time allocated.

CHAPTER 2

LITERATURE REVIEW

2.1 Oil Spill Pollution

As the crude oil is a very complex mixture of many different chemicals, consequently the effects of an oil spill on the marine environment of the oil spilled, as well as such other factors as the prevailing weather conditions and the ecological characteristics of the affected region. According to the complicated nature of oils, they will not behave as the same in the environment. Some constituents are noted for they tendency to vaporize while others clearly to bind to solids; some oil hydrocarbons extremely un-reactive while other interacts with light, so they have different toxicological effects on the aquatic life and hence on human being [4].

Some of the worst incidents of oil spills are The Haven in 1991 with 1,450,00 tons of oil spill, ABT summer in 1991 with 260,000 tons of oil spill, Fergana Valley in 1992 with 285,00 tons of oil spill and the worst of all is Gulf War oil spill in 1991 with 1,360,000 tons of oil spill.

The most noticeably bad oil spill ever, the Gulf War oil spilled an expected 8 million barrels of oil into the Persian Gulf after Iraqi strengths opened valves of oil wells and pipelines as they withdrew from Kuwait in 1991. The oil spill came to a greatest size of 101 miles by 42 miles and was five inches thick.

Some of the recent cases of oil spills are OT Southern Star 7 spilled over 300 tonnes of oil near Bangladesh on 9 December 2014, Napocor Power Barge 103 spilled over 170 tonnes of oil near Philippines on 8 November 2013 and Taylor Energy wells Platform 23051 which spilled over 70 tonnes of crude oil in Gulf of Mexico on 16 September 2004.

2.2 Oil spill response techniques

A number of advanced response mechanisms are available for controlling oil spills and minimizing their impacts on human health and the environment. The key to effectively combating spills is careful selection and proper use of the equipment and materials best suited to the type of oil and the conditions at the spill site [5]. Most spill response equipment and materials are greatly affected by such factors as conditions at sea, water currents, and wind. Damage to spill-contaminated shorelines and dangers to other threatened areas can be reduced by timely and proper use of containment and recovery equipment.

Chemical and biological methods can be used in conjunction with mechanical means for containing and cleaning up oil spills. Dispersing agents and gelling agents are most useful in helping to keep oil from reaching shorelines and other sensitive habitats. Biological agents have the potential to assist recovery in sensitive areas such as shorelines, marshes, and wetlands [6].

Physical methods are used to clean up shorelines. Natural processes such as evaporation, oxidation, and biodegradation can start the clean up process, but are generally too slow to provide adequate environmental recovery. Physical methods, such as wiping with sorbent materials, pressure washing, and raking and bulldozing can be used to assist these natural processes [5].

Table 1 Comparison of techniques used to clean up oil spill

Methods	**Operating mechanism**	**Advantage over kapok**	**Disadvantage against kapok**
Surface dispersants	Chemical dispersants, which have been used throughout the oil spill, are sprayed by boats, aircraft and workers on the shore. Chemical dispersants pull apart oil particles suspended in water, reducing the oil slick to droplets that can	Oil dispersing effect may take place faster than kapok absorbing the oil.	Oil will be degraded which means it cannot be recollected.

	be degraded by naturally occurring bacteria.		
Controlled burns	A fireproof boom corrals leaked oil into smaller, denser pockets that can be ignited remotely from the air and burned off. The process of burning removes large portions of oil from the water's surface, keeping it away from the shoreline.	Muck quicker way in controlling oil spill from reaching shoreline.	Environmental impact issue, burning the oil produces CO_2 where kapok is environmental friendly.
Boomers	Another technique is using booms to remove oil from the water's surface. Booms are used to collect oil in concentrated areas. This booms may contain hair, sawdust or other synthetic sorbent.	Cheap material such as hair or sawdust can be used as sorbent while kapok costs more.	The oil that been absorbed cannot be reused. Usually booms are disposed or degraded by microorganism.
Skimmers	Skimmers and boomers often work together. Where the oil that has been contained by the boomer is skimmed using mechanical skimmers.	Collects oil much faster than the kapok and most effective way.	Skimmers only can collect thick oil and leaves thin layer of oil.
Gelling	Using the motion of the sea, the gelling agent turns the oil into rubbery substance that can be easily removed from water with nets, suction devices or skimmers.	It is a quick process where it takes much less time due to solidification of oil takes less time	This method would require 3 times the oil amount to solidify the oil. Impractical to move such amount to oil spill area.
Biological agents	Biological agents increase the rate at which oil naturally	Requires less amounts than	Spilled oil must be cleaned up quickly to

	biodegrades. During this process, known as bioremediation, chemical agents, fertilizers and microorganisms are applied to oil, which breaks it down into a simpler and more easily removed compound.	amount of kapok needed to treat oil spill.	reduce potential damage to the environment. Unfortunately, biodegradation is a time-consuming process that can take years.
Peat moss	A Norwegian company, Kallak Torvstrofabrikk, has come up with an absorbent peat moss that can be tossed directly into the oil-soaked water. Apparently, it has been tested and worked during a 2009 spill off the coast of Norway.	Peat moss is commercial than kapok hence readily available in market.	Some portions of the soaked peat moss may sink to the bottom of sea.

2.3 Characteristic of sorbent

Insoluble material where raw or mixture of materials that recover liquids via the channels of absorption or adsorption or sometimes both (Praba Karan et al., 2011). Different sorts of sorbent materials have been tried and researched and additionally has been considered as best in oil removal application. Sorbent materials can be categorised into three sorts which are organic natural materials, synthetic organic polymers and inorganic mineral materials. Some of the examples for inorganic mineral materials are graphite, perlite, vermiculite, silica, zeolites, natural earth and fly fiery debris whereby the cases of synthetic organic polymers materials is polypropylene, polyethylene and polyacrylate. Natural characteristic materials utilized as sorbent includes straw, corn cob, sawdust cotton fibre, wool fibre, coconut husk and kapok filaments (Abdullah et al.,2010).

In general synthetic sorbents are commonly accepted as the most efficient way of absorbing oil. At times a proportion by weight of oil to sorbent of 40:1 can be achieved.

Table 2 Types of natural sorbent material and oil uptake (Ur Rahmah, Anisa. 2009)

Organic sorbent materials	**Oil type**	**Oil Uptake(g oil/g fibers)**	**Form**
Cotton	Diesel oil	30.6	Ground fiber
Kenaf bast	Diesel oil	7.2	Ground fiber
Kenaf core	Diesel oil	7.1	Ground fiber
Garlic peels	Crude oil	0.4	Sheets
Onion peels	Crude oil	0.5	Sheets
Natural wool	Motor oil	5.6	Fiber
Wool	Crude oil	31.8	Fiber
Milkweed	Crude oil	39.0	Fiber
Kapok	Crude oil	38.5	Fiber

2.4 Process of sorption and desorption

Absorption happens when particles go through or enter a massive material. Amid absorption, the atoms are totally dissolved or diffused in the absorbing material to form a solution. After dissolving into absorbent, the atoms cannot be divided effortlessly from it. Adsorption is commonly divided into physisorption (feeble van der Waal's strengths) and chemisorption (covalent holding). It might likewise happen because of electrostatic attraction. The particles are held weakly on the surface of the adsorbent and can be effectively uproot.

Amid oil cleanup procedure, absorption and adsorption happen among oil and adsorbent. Adsorption and absorption are both sorption process. Only if it is clear which process is agent, sorption is the favoured term. Absorption is where the whole mass is responsible in capturing the adsorbates while adsorption is a surface grounded process where a layer of adsorbate will be formed. Choi et al. recommends that volume on or between the fibres is for the most part in charge of most of the material's sorbent limit. Subsequently, it is suggested that adsorption by interfibre vessels is the fundamental adsorption component for kapok sorbent. Regular natural item like kapok were found to retain essentially more oil contrasted with business manufactured sorbent materials (Choi and Cloud, 1992; Sun et al., 2002; Choi, 1996; Kobayashi et al., 1977).

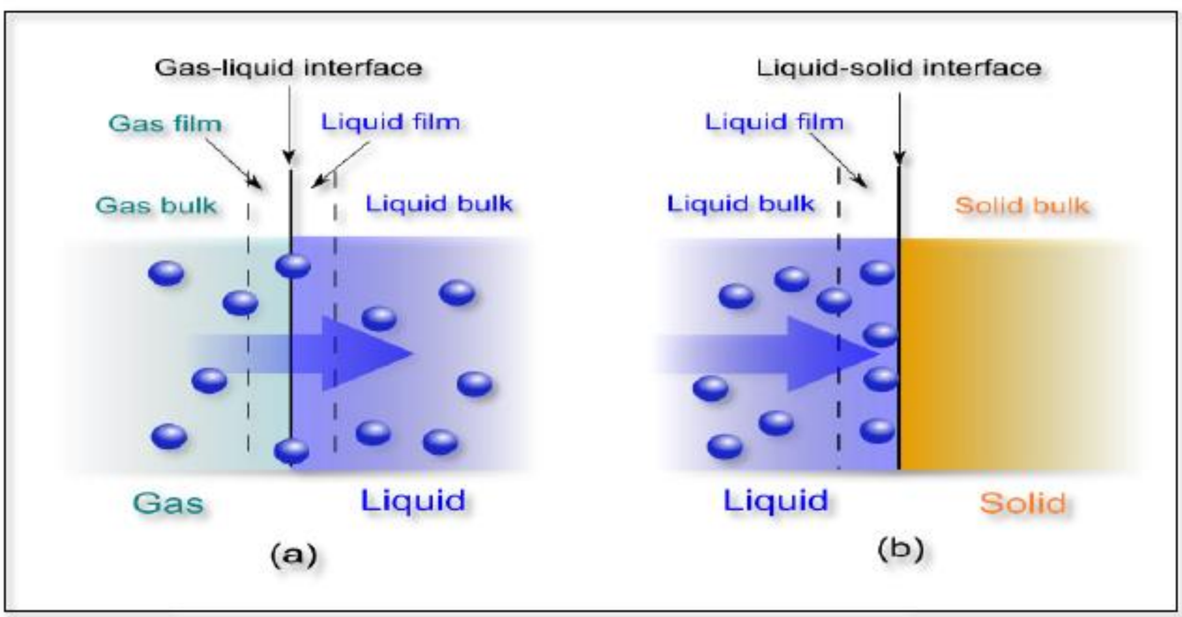


Figure 1: Demonstration of sorption (a) adsorption and (b) absorption in graphical

2.5 Character and role of raw Kapok fibre

Malaysian kapok is widely found in peninsular Malaysia especially the northern parts. It has leading up to now been perceived that the fibre of kapok, which are of a silky character, (being a types of silk cotton tree naturally identified with the cotton plant, and which is become in extensive amounts in Java, Ceylon and different spots having comparable climatic conditions) are made of surprising delicate quality, warmth and lightness, yet it has to this point been basically difficult to card and twist kapok fibre in an immaculate condition, unmixed with different fibres. In these studies, kapok fibre exhibited good water repellence, high oil adsorption capability, and well reusable characteristics, demonstrating its potential as an alternative for application in oil pollution control [10].

Figure 2: Kapok fibre

The kapok strands are used provincially as fillings in pillows, blankets, and some delicate toys. Kapok fibre was detached and broke down minutely, and the physicochemical properties were determined by spectroscopic systems. A few tests were carried out to focus the possible usage of kapok fibre. Minute examination of the higher structure of kapok fibre gave very distinctive results from cotton fibre, which has a fundamentally homogeneous empty tube shape and is made out of cellulose (35 % dry fibre), xylan (22%), and lignin (21.5%). Kapok fibre is portrayed by having an abnormal state of acetyl group (13.0%). Generally cell dividers of plants contain around 1%–2% of acetyl groups connected to non cellulose polysaccharides. Kapok fibre is altogether hydrophobic and does not get wet with water. Along these lines, the absorptivity of oil was tried. The fibre specifically assimilated critical measures of oil (40 g/g of fibre) from an oil suspension in freshwater and seawater. It is proposed that this fibre could be utilized to recuperate oil spilled in seawater.

Table 3 Chemical composition of kapok fibres (Praba Karan et al., 2011)

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

2.6 Kapok fibre modification

Modification is an act of changing the Kapok fibre structure using chemicals. The natural fibre have been treated with caustic soda and sodium chlorite with the target of evacuating surface polluting influences and creating fine structure alterations under mercerisation process. Kapok fibre is a commonly renewable material with huge lumen and hydrophobic attributes, which empowers it to show great oil sorption capacity. To further enhance the effectiveness for oil captureness, kapok fibre is treated with different chemicals, including water, HCl, NaOH, $NaClO_2$ and chloroform. The structure of untreated and treated kapok strands will be examined and analyzed utilizing Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The impacts of treatment concentration, temperature and time on oil absorbency of kapok fibre usually surveyed with toluene, chloroform, n-hexane and xylene as the model oils. The chemical treated kapok fibre will display better reusability, proposing its extraordinary potential for oil recovery.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology and Project Activities

Experiment is to find the best chemical to prepare the treated kapok among the two chemical chemicals. In this phase kapok will be treated with selected chemical chemical under same methods and parameters such as temperature and time for the treating is kept constant.

3.1.1 Analysis of Raw Kapok fibre

The raw Kapok fibre is analyzed using Field Emission Scanning Electron Microscope (FESEM) to obtain the micrograph of the fibre's single strand and network of strands. This is done to study the structure of single strand which is the building unit of the whole fibre. In addition, Kapok fibre that has been used to absorb oil then cleaned is also analyzed to understand how to structure of fibrous change with the usage as oil sorbent. Raw kapok fibre will be also sent to Fourier Transform Infrared (FTIR) spectroscopy to obtain their spectrum to analyze their frequency groups.

3.1.2 Kapok fibre Modification

Raw kapok fibre will be pressed into compact shape where these will be used in the preceding procedures. The pressed sample around 1g will be placed into 200mL chemical in a container such as flask to be left for an hour. Two chemicals are used at this stage. The specimen after being solved are washed with distilled water. This is done to remove any soluble non-structural constituents. The sample is then dried using dryer for four hours at 70°C to get a constant weight. It is worth mentioning that, the time and temperature for treating the kapok was kept constant. The drying temperature is kept low with long duration of heating so that the heating does not change the structure of the Kapok fibre and any modification on the structures are the result of the chemical alone.

3.1.3 Measurements of Oil Absorbency, Q

The sample that has been dried will be tested for their efficiency in absorbing oil at room temperature. The sample will be taken out from the oil at intervals and weighed. The sample should be wiped clean to remove excess oil before it is weighed. Next, the oil absorbency of the sample will be calculated using a formula where weigh of sample before and after being immerse in oil water will be taken into account. The following is the formula that will be used:

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

Q will represent the oil absorbency in grams of oil per gram of sample, whereby m_1 is the weight of sample before test and m_2 is the weight of sample after test. The unit of Q will be g oil/1 gram of sorbent.

3.1.4 Reusability capability test

In a nutshell, this is basically repeating the measurements of oil absorbency test using the same Kapok fibre several times without the calculation carried out but only the weight is recorded. The sample that has been subjected to oil absorbency test is removed from its oil water, wiped excess oil and weight is recorded. This sample is then removed to a cylinder where it will be squeezed by putting pressure on the piston. This is done to remove all the oil absorbed by the kapok which will be again immersed into oil water and repeated for five times. This test will determine the Kapok fibre’s life cycle or the maximum number of usage for the fibre to be recycled and reused.

3.1.5 Apparatus and Chemicals involved

No	Equipment / Chemicals	Uses
1.	Thermometer	To measure temperature of the chemical with sample
2.	Flask	To contain the chemical
3.	Measuring cylinder	Measure volume of chemical
4.	Fourier Transform Infrared (FTIR) spectroscopy	To analyse the spectra of kapok raw Kapok fibre
5.	Field Emission Scanning Electron Microscope (FESEM)	To obtain the micrograph of Kapok fibre single strand and network of strands
6.	Oven	Drying treated kapok
7.	Piston cylinder	To remove the oil from the kapok
8.	Filter paper	To contain the oil contaminated kapok and to wipe excess oil
9.	Electronic weighing machine	To weigh the before and after the kapok has absorbed oil
10.	Sodium Hydroxide, NaOH	Chemical to treat the Kapok modification
11.	Sodium Chlorite, $NaClO_2$	Chemical to treat the Kapok modification
12.	Diesel	To resemble crude oil (identical to oil spilled condition)

3.1.6 Range of Variable

a) Study of weight change of kapok with respect to chemical

Chemical used	Weight of kapok sample after immersed in oil (g)
Raw kapok	
Sodium Chlorite, $NaClO_2$	
Sodium Hydroxide, NaOH	

b) Oil absorbency calculations

Chemical used	Weight before immersed (g)	Weight change,Δm (g)	Oil absorbency, Q
Raw kapok			
$NaClO_2$			
NaOH			

c) Reusability test

Trial no.	1	2	3	4	5
Weight(g)					

d) Optimum time test

Time (h)	1	2	3	4	5	6
Weight(g)						

3.2 Experimental Procedure / Approach

Referring to the previous research work done by Jintao Wang, Yian Zheng and Aiqin Wang in their project, Effect of kapok fibre treated with various chemicals on oil absorbency. [10] Therefore using their method, procedures for the experiment going to be conducted was structured as follows:

3.2.1 Kapok Modification

1. The kapok is compressed into small shape possible and weighed to 1g.
2. 200mL of chemical is prepared in a flask.
3. The kapok is added into the chemical. (in case where the kapok floats, place a glass rod on the kapok to submerge the kapok into the chemical)
4. Sample is collected after an hour and wash with adequate distilled water to remove any soluble non-structural constituents.
5. The sample is then put into a dryer where it will be dried for 4 hours at 70°C
6. Repeat step 1 to 5 for the second chemical.

3.2.2 Kapok fibre Oil Absorbency and Reusability Test

1. Place the modified or raw kapok into oily water beaker.
2. Leave the fibre in the beaker for an hour.
3. Collect the sample, weigh and record the reading. (Use filter paper at this point to avoid contaminating the weighing machine)
4. Use a piston to press out the oil and repeat the step above for another 5 times and record the readings. (Reusability test)
5. Complete the data table.

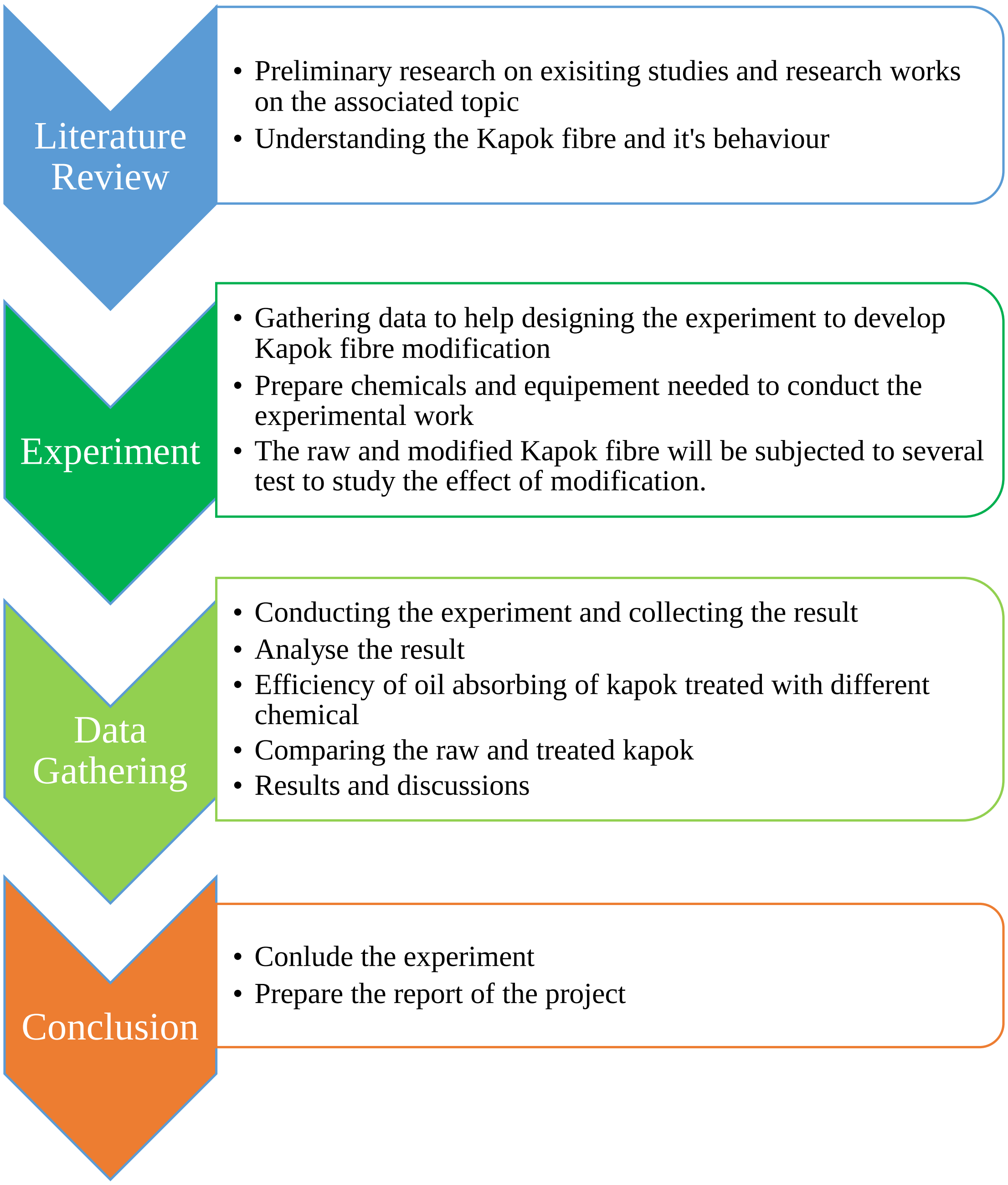
3.2.3 Optimum Time test

1. Place the modified Kapok fibre into oily water beaker.
2. Every one hour, remove the fibre from the beaker.
3. Weigh and record the value.
4. Put the fibre back into the beaker and repeat the steps until 6 hours.

3.2.4 Simulation of Oil spill in ocean

1. Prepare diesel, salt and tap water.
2. Add 35 g of salt for 1kg or 1 liter of tap water to imitate sea water.
3. The amount of diesel in the mixture was 2.5% by weight, therefore 0.025kg for each litre of tap water.
4. Using a mechanical stirrer operated at 1000 rpm, at which tap water and diesel were evenly mixed with no visible foam formed.

3.3 Key Milestone



3.4 Project Gantt Chart

3.4.1 Final Year Project I Guideline [Planning Phase]

No	Detail	Week 1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Title Selection and Allocation	■													
2	Submission of Title Selection Form	■													
3	Title and Supervisor Allocation		■												
4	Preliminary Research Work		■	■	■										
5	Preparing Extended Proposal				■	■	■								
6	Submission of Extended Proposal						■								
7	Proposal Defence								■						
8	Project Work Continues						■	■	■	■	■	■	■	■	■
9	Submission of Interim Draft Report												■		
10	Submission of Final Interim Report														■

3.4.2 Final Year Project II Guideline [Execution Phase]

No	Detail	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Lab Booking														
2	Experimental Setup														
3	Experimental Work Phase One														
4	Submission of Progress Report														
5	Experimental Work Phase Two														
6	Submission of Dissertation (softbound and technical paper)														
7	Oral Presentation														
8	Submission of Dissertation (hardbound)														

CHAPTER 4

RESULT AND DISCUSSION

4.1 Raw kapok fibre Characterization

4.1.1 FESEM Analysis

Physical properties of raw kapok was characterized grounded on morphology. Field-Emission Scanning Electron Microscope (FESEM) was used to analyse the fibres morphologies.

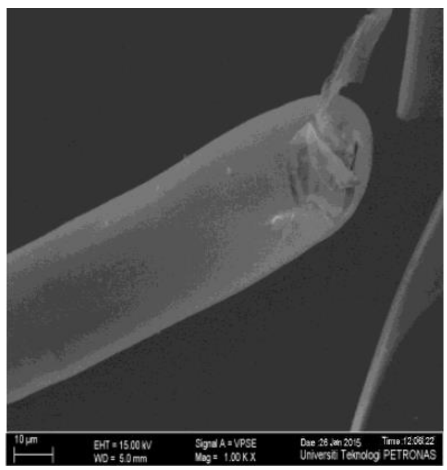


Figure 3: Scanning Electron Micrograph of raw kapok fibre

From the Figure 3, it can be seen that raw kapok fibre has an even surface area without any ripple and a hollow tubular structure or lumen. The smooth surface showed by the raw kapok is determined due to the plant wax layer on the surface (Wang et al., 2012) and the structure of the fibre demonstrate the buoyant capability displayed by the kapok fibres and the utilization of kapok strands as stuffing materials (Praba Karan et al.,2011). This hollow ratio can reach up to 90 percent of the whole structure. Hollow segment of the fibre is where the oil goes when being absorbed by the fibre.

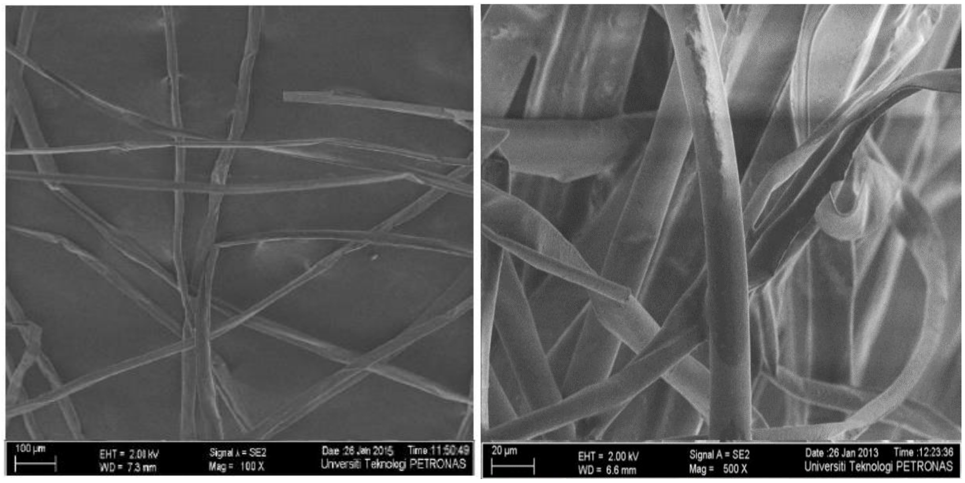


Figure 4: SEM Micrograph of (a) raw kapok fibrous and (b) saturated kapok fibrous

Figure 4 (a) is representing SEM micrograph showing the strands of cellulosic fibre ensnared to one another. The fibrous material expands the surface area for sorption of raw kapok. Meanwhile Figure 4 (b) is the representation of where the oil goes when it is being absorbed by the fibre. The picture is actually of a dry kapok that was soaked in oil which was then pressed to remove the oil. This shows the development of fibrous entrains because of oil residue on the surface pores. Hence, this proves that kapok has limited reusability due to not entire oil can be recuperated.

4.1.2 FTIR Analysis

Fourier Transform-Infrared spectroscopy (FTIR) was used to obtain the spectrum and define the constituent in the kapok fibre. FTIR spectroscopy was carried out for two sample; raw kapok fibre and the kapok fibre saturated with oil. Figure 5 below shows the spectra obtained for the raw kapok. The frequency was then categorized into their respective bands with the aid from FTIR done in previous research.

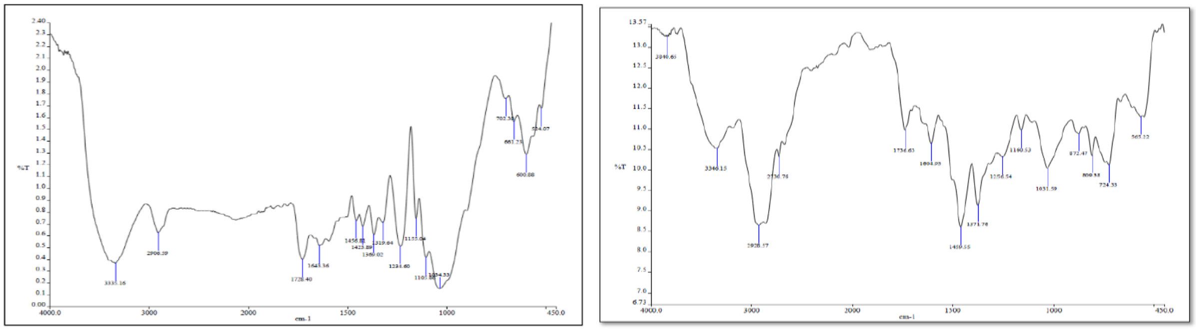


Figure 5: FTIR spectra of (a) raw and (b) saturated Kapok fibre

This will clearly show where the absorption bands are location hence modification of the Kapok fibre should be focused in altering these bands to obtain better oil absorbency of the fibre. It can be seen that when compared to spectrum obtained from raw kapok that the middle part has expanded proving the presence of hydrocarbon material.

Table 4 Infrared absorption bands and respective assignments (Adebajo., 2004; Mwaikambo et al., 2002; Wang et al.,2012)

Frequency (cm^{-1})	Assignment
3337	OH stretching
2891	CH stretching of CH_2 and CH_3 groups
1742	Carbonyl C=O stretching ester
1420	OH and CH_2 bending
1368	C-H bond in –O(C=O)-CH_3 group
1337	CH_3 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

Using the table obtained from research, the spectra obtained for raw kapok are assigned into their appropriate bands and tabulated in the table below:

Table 5 Infrared transmittance of frequency (cm-1) of kapok

Frequency (cm^{-1}) of raw kapok fibre	Bond type/Assignment
3335.16	OH stretching
2906.59	CH stretching of CH_2 and CH_3 groups
1728.40	Carbonyl C=0 stretching ester
1423.89	OH and CH_2 bending
1369.02	C-H bond in -O(C=O)-CH_3 group
1234.60	C-O stretching of acetyl group
1155.04	C-O-C anti symmetric bridge stretching in cellulose and hemicellulose
1034.33	C-O stretching in cellulose, hemicellulose and lignin
600.88	-OH

4.2 Properties of Oil Water

The oil water that is used to simulate the oil spill in ocean have important influence on the oil sorption capacity of kapok assembly. In this project, crude oil has been used to mimic the effect of crude oil in ocean. Diesel has a density of 823 kg/m^3 at 20°C and viscosity of 4×10^{-6} m^2/s (Wang et al., 2012). Where else, crude oil has density of 900 kg/m^3 and viscosity of 9×10^{-6} m^2/s. This shows that crude oil can be best represented by diesel. Density and viscosity can affect the project in such ways that density will have impact on the oil sorption characteristic where the increase in density will lead to increase in sorption capacity of the sorbent. Meanwhile, the viscosity contributes to oil sorption performance since it has direct effect on the flow resistance in the sorbent structure. Oil with higher viscosity will attain equilibrium later than oil with lower viscosity where oil would drain out within short time.

4.3 Modified Kapok fibre against raw Kapok fibre

4.3.1 NaOH modified Kapok fibre

Figure 6: Kapok fibre in NaOH solution

Figure 6 above shows how Kapok fibre is treated using the chemical where it will be left in the Sodium Hydroxide for one hour before it will be washed with distilled water and then placed in a dryer for 4 hours under 70°C and the dryer used is shown in the Figure 7 below.

Figure 7: Oven used for fibre drying

After the fibre has been dried, the modified Kapok fibre will be taken out and subjected to oil absorbency test and data for the runs were recorded. The result from oil absorbency test are tabulated in Table 6.

Table 6 NaOH treated fibre

Number of run	Weight before immerse, m_1 (g)	Weight after immersed in oil, m_2 (g)	Oil absorbency, Q
1	1.059	35.899	32.899
2	0.997	23.763	23.841
3	1.023	20.675	19.210
4	1.058	17.623	15.657
5	1.035	16.598	15.037

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

$$Oil\ absorbency = \frac{Weight\ after\ immersed\ in\ oil\ (g) - Weight\ before\ immerse\ (g)}{Weight\ before\ immerse\ (g)}$$

$$= \frac{35.899 - 1.059}{1.059}$$

$$= 32.899$$

The oil absorbency of Sodium Hydroxide modified Kapok fibre is compared with the raw Kapok fibre absorbency test and shown in Table 6. The result for raw Kapok fibre oil absorbency test is produced under Appendix A.

Table 7 Oil absorbency of raw and NaOH treated fibre

Number of run	Oil absorbency (g oil absorbed/g fibre)	
	Raw kapok fibre	Sodium hydroxide treated
1	35.899	27.185
2	23.763	21.704
3	20.675	19.219
4	17.623	17.219
5	16.598	16.971

The tabulated data in the table 7 is analysed in Figure 8 for their relative comparison of absorbency. This figure will contrast the effect of chemical on the fibre whether the oil absorbing efficiency has increased or decreased. Apparently, the fibre that has been treated with caustic soda is performing less than the raw fibre where the absorbency of the raw kapok fibre surpasses the NaOH treated fibre from the first run to the fifth run. This indicates that oil absorbing capability of fibre has reduced after being treated with sodium hydroxide.

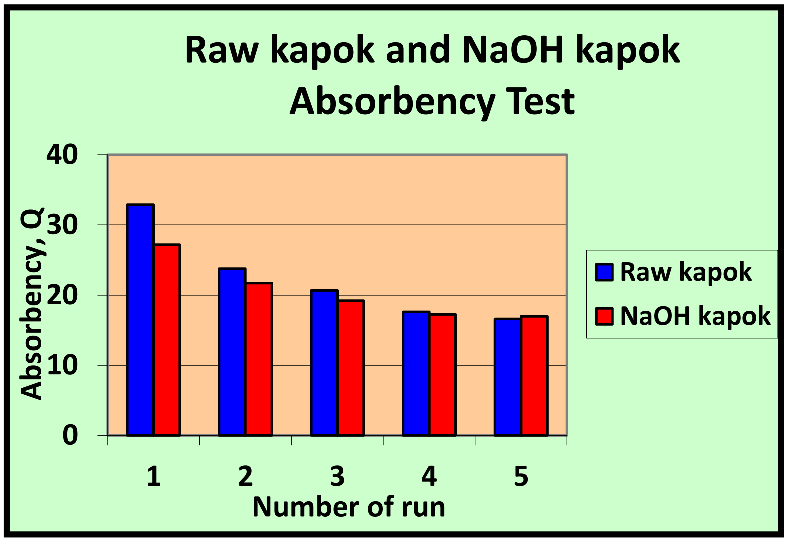


Figure 8: Raw fibre and NaOH treated fibre absorbency test

The decreasing value of the absorbency in caustic soda treated fibre may due to damage to the hollow lumen that is caused by alkalization. Hollow lumen as was shown in Figure 3 is the part where oil is stored upon being absorbed. This whereby means any impairment done to the lumen whether partially or completely will cause the absorption mechanism of oil into the hollow lumen blocked. This explains the poor performance of the NaOH treated fibre contrasted to non-treated fibre. This theory 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 fibre 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 modify the fine structure of Kapok fibre where the hollow lumen to store the oils vanishes partially or completely and the damaging effect on tubular structure is permanent.

4.3.2 NaClO$_2$ modified Kapok fibre

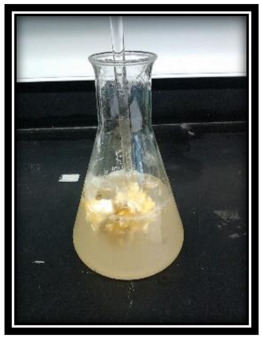


Figure 9: Kapok fibre in NaClO$_2$ solution

Kapok fibre treated with NaClO$_2$ was subjected to oil absorbency test and data for the runs are tabulated in the table below:

Table 8 Oil absorbency NaClO$_2$ treated fibre

Number of run	Weight before immerse, m_1 (g)	Weight after immersed in oil, m_2 (g)	Oil absorbency, Q
1	0.995	42.678	41.892
2	1.001	36.003	34.967
3	1.009	31.958	30.673
4	1.001	22.877	21.854
5	1.002	19.466	18.427

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

$$Oil\ absorbency = \frac{Weight\ after\ immersed\ in\ oil\ (g) - Weight\ before\ immerse\ (g)}{Weight\ before\ immerse\ (g)}$$

$$= \frac{42.678 - 0.995}{0.995}$$

$$= 41.892$$

The oil absorbency of Sodium Hydroxide modified Kapok fibre, Sodium Chlorite modified Kapok fibre and raw Kapok fibre is compared and shown in Table 7.

Table 9 Oil absorbency of raw, NaOH treated and $NaClO_2$ treated fibre

Number of run	Oil absorbency (g oil absorbed/g fibre)		
	Raw kapok fibre	Sodium hydroxide treated	Sodium chlorite treated
1	32.899	27.185	45.538
2	23.763	21.704	34.967
3	20.675	19.219	30.673
4	17.623	17.219	21.854
5	16.598	16.971	18.427

The tabulated data in the table is analysed in Figure 9 for their relative comparison of absorbency. This figure will contrast the effect of chemical on the fibre whether the oil absorbing efficiency has increased or decreased. Apparently, the fibre that has been treated with Sodium Chlorite is the best among all. This indicates that Sodium Chlorite shows positive modification on Kapok fibre. The oil absorption is higher as a result of rough surface and low surface energy in which the modification can make the oleophilic surface to be more oleophilic as a result of capillary result.

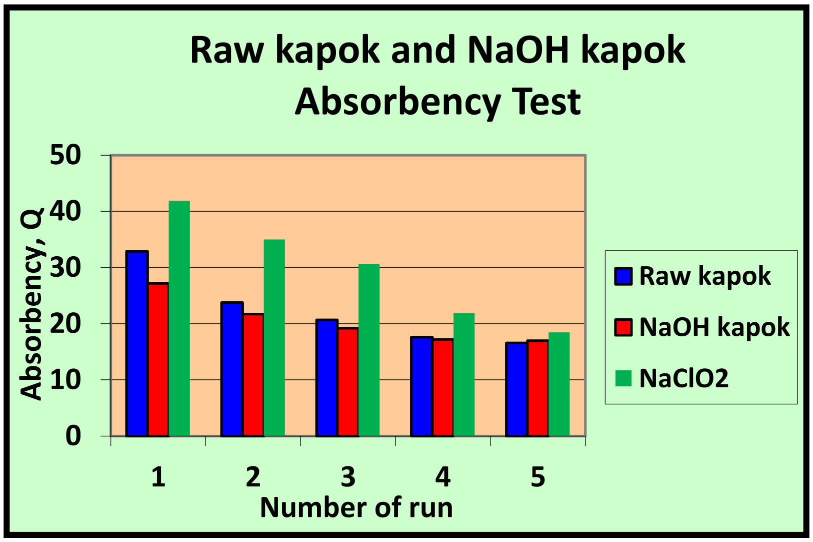


Figure 10: Raw fibre, NaOH treated and $NaClO_2$ treated fibre absorbency test

The Sodium Chlorite modified fibre shows a better oil absorbency than the other two fibre. Sodium Chlorite have seem to increase the surface roughness thus increase the oil affinity. From the characterization of Kapok fibre, we have learnt that the surface of Kapok fibre are smooth therefore by increasing the surface area of these Kapok fibre, oil absorption is seems to have increased. Sodium Chlorite might have created ripples that has contributed to the increase of the surface area of the fibre. This is supported by Wang et al. (2012) who stated in addition to the rougher surface, the super hydrophobic modified Kapok fibre also result in decreasing of the surface energy which affects the oil absorption.

4.4 Reusability Test

To meet the third objective where the reusability of the Kapok fibre has to be determined so that the number of life cycles can be known which holds the information on how many times the same fibre can be used before it must be replaced.

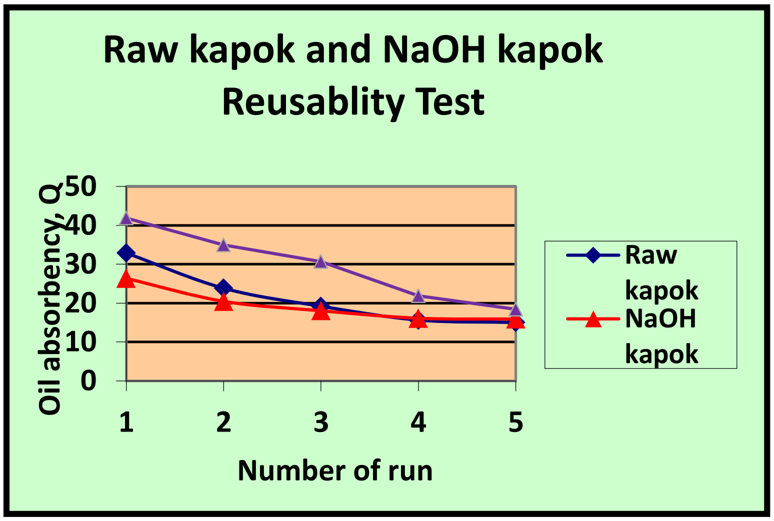


Figure 11: Raw fibre and NaOH treated fibre reusability test

Using the absorbency value and number of test run, reusability test could be figured out. It can be concluded that the reusability of fibre decreases immediately after the first run. This proves that sorption capacity of the fibre reduces with number of cycles. The outcome that can be determined is fibre could be used efficiently up to 3 cycles. However Choi, 1992; suggests that kapok fibre can be used up to four cycles. This may due to different techniques used in drying and treatment of kapok. It can also be seen that, the absorption capacity of treated does not reduces much as per raw kapok due to more stable fibrous material. Previously from the FESEM analysis, it was seen the fibrous strand expands after being used to absorb oil. This might explain why the reusability is limited since the absorbency of the fibre drops due to lumen stores oil from the previous absorption.

4.5 Optimum Time test

To find how long could the fibre left exposed to the oil water and when is the optimum time to recover the fibre in order to achieve the highest recovery of oil from ocean.

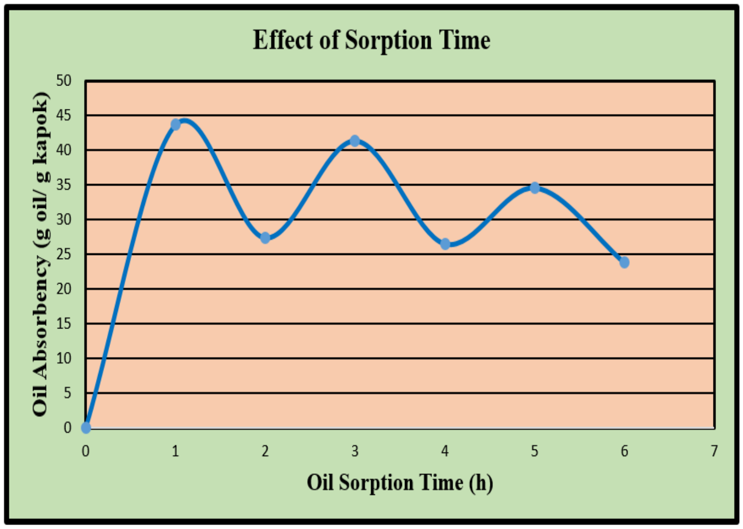


Figure 12: Effect of Sorption Time

The aim is to study the effect of longer sorption time to oil sorption capacity. The saturated sorbent was found to release oil back to surrounding after 1 hour. The unsaturated sorbent then draws back the oil in, thus providing a sinusoidal graph as follows. This is what caused secondary pollution to happen. This may due to absorption and desorption pattern of the fibre that produces the sinusoidal graph pattern meanwhile proving that secondary pollution will happen if the fibre is recollected at the wrong duration. Therefore the fibre should be avoided to be collected at the time where the absorbed value of oil is lower to ensure secondary pollution can be avoided.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

As a conclusion, this project is important as it provides an alternative way of removing oil from water body. Based on the characterization, kapok has fibrous cellulose that absorbs oil efficiently. The bands that are responsible for the oil absorbing in found out to be the middle part of the spectrum.

For Sodium Hydroxide, it was seen that modification was done on the deep fibrous where the surface wax was got rid by the chemical and permanently damages the lumen of the fibre. As a result, the oil absorbing capability reduces thus implying caustic soda may not be a good chemical to be used in treating the kapok. Sodium Chlorite will remove the wax on the fibre surface thus creating ripples on the smooth surface of the fibre. These ripples or wrinkles will eventually increase the surface area available for the oil sorption whereby more oil can adhere to surface and enter into the lumen of fibre with ease. The Kapok fibre reusability test showed that the fibre can be used up to 3 times. This limited number of life cycles are due to Kapok fibre strands which expands after being used. After that, the optimum time for the Kapok fibre to be left in the ocean is determined. From the result, it was found that the duration for the Kapok fibre to be left in ocean is one hour. This is because, at one hour the fibre shows the highest absorption level.

In a nutshell, Kapok fibre treated with Sodium Chlorite shows a good performance thus making the fibre as a suitable replacement for the sorbent material used in boomers and used together with skimmers. The future recommendations would be the optimization on the chemical treatment on kapok. Where, concentration and temperature could be manipulated to find the finest conditions to modify the kapok fibre.

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APPENDICES

Appendix A: Raw kapok oil absorbency test

Number of run	Weight before immerse (g)	Weight after immersed in oil (g)	Oil absorbency, Q
1	1.059	35.899	32.899
2	0.997	23.763	23.841
3	1.023	20.675	19.210
4	1.058	17.623	15.657
5	1.035	16.598	15.037

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

$$Oil\ absorbency = \frac{Weight\ after\ immersed\ in\ oil\ (g) - Weight\ before\ immerse\ (g)}{Weight\ before\ immerse\ (g)}$$

$$= \frac{35.899 - 1.059}{1.059}$$

$$= 32.899$$