# **Oil Spill Cleanup using Raw Kapok as the Sorbent Material**

by

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Progress report submitted in partial fulfillment of
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Bachelor of Engineering (Hons)
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CERTIFICATION OF APPROVAL

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Approved by,

______________________
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TRONOH, PERAK
August 2013

# 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 AQILAH BT NORDIN

# ABSTRACT

A natural and biodegradable material that has good oil absorbtivity, high durability and reusability is one of the most demanded materials in the industry nowadays. It is believed to have potential to replace polypropylene function as the commercialized sorbent. One of material that suits this function is kapok fibre or commonly known as cotton. Using kapok, decomposition process, which always become an issue when dealing with polypropylene, is no more a problem. The nature of Kapok will allow it to decompose naturally without any harm threat to the environment. Besides, kapok has fibrous microstructure that becomes the key factor to absorb oil efficiently. Without any chemical modification, it can absorb significant amount of oil like polypropylene. This project was conducted to evaluate kapok capacity as the natural sorbent in terms of application. A series of test was done to analyze the surface microstructure, sorption capacity, dynamic oil retention and its reusability. Two experimental setups are prepared for sorption and desorption analysis. Findings of this paper are found base on the weight of oil recovered and HPLC test for seawater composition. It is believed that this project can prove the potential of Kapok as sorbent for oil spill cleanup at the end of this paper.

# ACKNOWLEDGEMENT

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# CHAPTER 1

## INTRODUCTION

### 1.1 PROJECT BACKGROUND

Despite the positive development of oil and gas industry in this last decade, there is something more valuable have been put on the line. The environment needs to pay for any single mistake made by human. One of the greatest mistakes in humankind history is oceanic oil spill. Oil spill refers to an accidental release of oil into a body of water, as from a tanker, offshore drilling rig, or underwater pipeline. It has become a major environmental problem that the world is facing right now.

History has shown how worst this problem could go. From the first major commercial spill of 1967 in the United Kingdom, up to the most recent one, at BP Deepwater Horizon Oil Rig, that leaked thousands barrel of oil into the Gulf of Mexico since April 2010. Most large scale oil spills have been disastrous and the marine wildlife is still recovering. This is proven on March 2012, when scientists lead by chemist Helen White of Haverford College in Pennsylvania, were surprised to find coral communities covered in a brown flocculent material and showing signs of tissue damage. Hence, the tension for a better cleanup technology increases.

Oil spill cleanup using sorbent material is one of the most used technologies whereby synthetic sorbent material like polypropylene is used to trap oil from seawater. By using the same concept, this research was conducted to find a natural and biodegradable sorbent that can replace the existing one. Kapok was identified.

## 1.2 PROBLEM STATEMENT

To recover large amounts of crude from a spill has never been easy. It needs large manpower, thousands of dollars, more time and careful planning. That is why, it is important to choose the right technique and keep improvising it. Wrong technique could lead to a bigger problem. For example like the usage of dispersant to handle oil spill in Mexico. The study found that mixing the dispersant with oil increased toxicity of the mixture up to 52-fold over the oil alone. Oil skimming technique on the other hand, even though is safe, it is not as effective as oil can easily escape from the skimmers. The only option left is by using sorption method and up until now, it is believe to be the best method compare to the others.

In the sorption method, the most common type of sorbent use is from synthetic materials like polypropylene. Polypropylene sorbent can absorb oil significantly without any major issue. However, this approach does have the downsides that give a room for improvement. Polypropylene is a non-biodegradable material. Hence, problems arise when decomposing the used sorbent. Considers that at one point there was 28,000 feet of polypropylene boom deployed in the Gulf of Alaska, that need to be incinerate due to its hardly decompose nature. In this state, a temporary site is needed just to dispose this saturated sorbent while waiting for it to be incinerated. Oil will eventually escape through pores and this leads to secondary pollution, which is going to be worst. Besides, incineration process is not a cheap technology and this is a real economic liability. Hence, the world is in need for a sorbent material that can decompose naturally without much effort.

A biodegradable sorbent with excellent biodegradable properties would be advantageous with this respect (Sunni et al., 2004). There are many types of biodegradable material available but among them; the best that matches the idea is kapok. It is not only natural, but also can absorbs oil as goods as polypropylene sorbent. In fact, some research proves that raw kapok can absorb up to 40wt% of its weight. The effects and application have been tested thoroughly. Figure 1 below shows the secondary pollution from decomposition of synthetic sorbent.

Figure 1: The disposal problem with synthetic sorbent (source: ITOPF)

The disposal problem arises when using polypropylene sorbent in large amount. The current treatment technology was forced to use incineration process to decompose all the saturated sorbent. If not, secondary pollution can occur when the crude oil retained escapes from the sorbent to the land, thus initiates the land pollution. However, the downside of the incineration process is its very high cost and hydrocarbon gas emissions. That is why, natural decomposition of material is most preferred and development of biodegradable sorbent is required.

## 1.3 OBJECTIVE AND SCOPE

The main objective of this research is to evaluate the capacity of raw kapok as the natural sorbent for oil spill cleanup. In order to evaluate a sorbent material, few criteria need to be tested. Thus, these criteria are taken as the scope of study to conduct the experiment. The scope of study of this research is:

i. To characterize and study the surface morphology of raw kapok.
ii. To test the oil sorption capacity of kapok sorbent.
iii. To evaluate the oil retention performance of saturated kapok.
iv. To test the reusability of kapok as recycled sorbent

In Chapter 2, a thorough research was conducted to evaluate the scope mentioned above. Relevant data was extracted to help setting the scope and boundary of this project. Current application and findings by other researchers were further reviewed.

## 1.4 FEASIBILITY

In real life, if this project managed to prove that kapok can absorb significant amount of oil and perform good characteristics of sorbent, the oil cleanup process will become easier, more efficient and cost saving as it has good reusability rate and indeed can decompose naturally. There is no need for incineration. The secondary pollution also can be avoided. In other word, this project is relevant with current demand nowadays especially when the industry is having difficulties with the current cleanup technology. Besides relevancy, feasibility study is also important. Feasibility analysis can be divided into three which are technical feasibility, budget feasibility and time feasibility. Below are the findings of the analysis.

**Technical Feasibility**

This project will be using two major equipments which are the SEM Microscope and High Performance Liquid Chromatography (HPLC). Both are available here in UTP and the technician is skillful enough to help in handling the test. Besides, the raw materials needed are not difficult to get. Hence, technically this project is feasible.

**Cost Feasibility**

Every project was allocated for RM500 only. Hence, with this tight budget, feasibility study is important. Even though the market price for raw kapok is quite expensive, the sample size is not that big. The characterization process will consume most of the budget as each sample tested was charged with min RM78 and above. After a quantitative analysis, it was found that the overall cost is still feasible and on budget.

**Time Feasibility**

The overall time to complete this project is 2 semesters or 8 months. The first 4 months are the planning phase which is by now supposed to be completed. The next 4 months are the execution phase. This project is expected to finish on time if there is no major issue and project progress is following the gantt chart provided in Chapter 3.

# CHAPTER 2

## LITERATURE REVIEW

### 2.1 OVERVIEW OF THE PROCESS

Whenever oil is spilled, there would be a potential to cause significant environmental impact (Bucas and Saliot, 2002). Oil spill in the marine environment undergoes a wide variety of weathering processes, which include evaporation, dissolution, dispersion, photo-chemical oxidation, microbial degradation, adsorption onto suspended materials, agglomeration, and others (Jordan and Payne, 1980). These physico-chemical changes enhance oil dissolution in seawater (Payne and Phillips, 1985). Seawater can contain oil in three major forms: 1. free oil larger than 20 microns in diameter, 2. emulsified oil that have been mechanically or chemically reduced in size to smaller than 20 microns in diameter, and 3. dissolved oil as molecular-scale particles capable of dissolving in water (TurnKey Solutions, Inc.).

The contained oil in seawater can be removed either by mechanical or chemical means. Mechanical method involves the use of skimmers. Skimmers work by taking advantage of the adhesive nature of the oil, which will cling to any surface that it comes into contact with. Oil sticks to the rope and is carried back to the drive unit for removal. The primary types of oil skimmers include belt, brush, mop and floating suction (Skimoil, Inc., 2010). The main limitation of this technique is its high cost and inefficient trace level adsorption (Wardley-Smith,1983). When sitting on highly concentrated oil, skimmers tend to take on large amounts of water with the oil (WiseGeek, 2010).

On the other hand, chemical method by using dispersant is very efficient. However, most of the dispersants are often inflammable and cause health hazards to the operators and potential damage to fowl, fish and marine mammals. They can also lead to fouling of shorelines and contamination of drinking water sources (NRC, 1989). But according to National Oceanic and Atmospheric Administration (NOAA), since both the dispersants and the dispersed oil are toxic (in varying degrees, according to the type of oil spilled and the chemical agent used) to animals living underwater, coral reefs and other marine life, decisions to use dispersants involve trade-offs between decreasing the risk to water surface and shoreline habitats while increasing the potential harm to organisms in the water column and on the seafloor.

For these reasons, decisions concerning the use of dispersants are usually based on a broad assessment of the overall environmental impact of a specific spill (Pam ,2010). But, there is evidence that dispersed oil degrades more quickly than oil that has not been dispersed. So, ultimately, a successful dispersant operation would end in dispersed oil droplets being processed in the marine ecosystem and degrading into naturally occurring substances. This process starts with the droplets of oil and dispersant being colonized by bacteria that then begin to decompose. Next, protozoans and nematodes (small worms) join the colonies. Eventually, the oil may be further broken down and incorporated into the food web. (NOAA, 2008) However, some research raises concerns that certain microbes may chew up the dispersant molecules instead of dining on the oil (ACS, 2010).

Figure 2: Dispersant of chemical and synthetic boomers method

Among others, removal of oil by sorption process has been observed to be one of the most effective techniques for complete removal of spilled oil under ambient conditions. Absorbent materials are attractive for some applications because of the possibility of collection and complete removal of the oil from the oil spill site (Adebajo, Frost, Kloprogge, Carmody and Kokot, 2003). The addition of sorbents to oil spill areas facilitates a change from liquid to semi-solid phase and once this change is achieved, the removal of the oil by removal of the sorbent structure then becomes much easier. Furthermore, these materials can, in some cases, be recycled. The sorbent material can be made by natural and synthetic materials. The natural sorbents are environmentally safe and stimulate community involvement (Choi and Cloud,1992).

## 2.2 CHARACTERISTIC OF SORBENT

The characteristics of an ideal sorbent material used for oil spill cleanup include hydrophobicity or oleophilicity, high uptake capacity and high rate of uptake of oil, buoyancy, and retention over time, durability in aqeous media, reusability, biodegradability and recovery of oil (Praba, Rengasamy and Dipayan, 2011). However, it is almost impossible to find a material that has all the properties as mentioned. Hence, the material which perform high sorption capacity, with good reusability and oil recovery will be chosen. Various sorbents such as exfoliated micas, chalk powder, ekoperl, sawdust, foams of polyurethane or polyether, fibers of nylon, polyethylene have been studied for this purpose (Wardley-Smith, 1983). These materials all show porosity and ability to absorb oil in the presence of water.

It has been suggested that oil-sorbent materials can be grouped into three major classes namely inorganic mineral products, synthetic organic products and organic vegetable products (Melvold, Gibson and Scarberry, 1988). Mineral products include materials such as zeolites, silica, perlite, graphite, vermiculites, sorbent clay and diatomite (Scharzberg, 1971). Synthetic organic products include polymeric materials such as polypropylene and polyurethane foams which are the most commonly used as commercial sorbents in oil spill cleanup due to their oleophilic and hydrophobic characteristics (Graham and Trotman, 1980).

A major disadvantage of these materials is that they degrade very slowly in comparison with mineral or vegetable products and are not as naturally occurring as mineral products (Choi and Cloud, 1992). Examples of organic vegetable products (or natural sorbents) that have been reported include straw, corn corb, wood fiber, cotton fiber, cellulosic kapok fiber, kenaf, milkweed floss and peat moss (Melvold, Gibson and Scarberry, 1988).

## 2.3 PROPERTIES OF KAPOK

To the naked eye, kapok fiber from the kapok tree species Ceiba Pentandra is yellowish and lustrous. The fiber feels silky and springy to the touch and relatively lightweight and warm in the hand (Paradigm Aupost, 2010). A bushel of kapok fiber appears consistent in color and quality. Unused Kapok fiber should be odorless.

Figure 3: Kapok fibre from kapok fruits

Individual kapok fibers are 1 to 1.5 cm in length. Under a microscope, kapok fibers present a long narrow cell with frequent folds. Its cell has thin walls and a hollow structure. Its walls form a smooth, closed tube with a large cavity called a lumen. Each kapok fiber is coated with a waxy substance called cutine. The cutine and lumen are thought to give kapok its buoyancy (Anthony, 1994). Chemically, kapok fiber consists mainly of cellulose, although also containing pentose-containing polysaccharides called pentosan and an inert plastice-like material called lignin.

Kapok fiber's essential attributes are many: buoyant, resilient, moisture resistant, vermin resistant and smooth, kapok possess powerful performance in a lightweight package. It 's said that kapok fiber repels water like rain on a ducks back. When a substance does this we call it, hydrophobic. This hydrophobic quality results in the quick-drying, buoyant and moisture-resistant properties, which makes kapok fiber remarkable among natural fibers (Deschamps, Caruel, Borredon, Bonnin and Vignoles, 2003). Kapok fiber supports as much as 38.6 times its own weight in water. Buoyancy is lost slowly; with one test showing only 10 percent loss after 30 days of water immersion. No other natural fiber is better than kapok for water-safety equipment. When kapok fibers are put under tension they completely return to their original length when the tension is removed. Kapok fiber is devoid of nutritional content thus kapok fiber is vermin resistant. Kapok fibers are smooth and missing the scales of animal hair so it won't mat or felt easily. It weighs only one-eighth as much as cotton, is as warm as wool and is as smooth as silk.

Several materials such as milkweed and Kapok fibers (Choi, 1996) have been developed as they exhibit much higher oil sorption capacity than polypropylene fiber or polyurethane foam and more importantly, kapok is biodegradable. A biodegradable material with excellent sorption properties comprise majorly of cellulose of the dry fiber weight. The remaining components are impurities such as, proteinaceous material, 0.3 to 1% waxes, 07 to 1.2% pectins and small amounts of organic acids and as producing inorganic materials (Segal, 1985). Typical analysis indicate that the Kapok fibers comprise 64% cellulose, 13% lignin and 23% pentosan. Besides these constituents, they also contain wax cutin on the fiber surface which makes them repelling water (Kobayahi *et al.*, 1977). This is why it can absorb oil in the presence of water and it exhibit good performance as synthetic sorbent.

For example, previous report shows that the oil sorption of cellulosic kapok fiber used in a mat, block, band or screen was approximately 1.5-2.0 times greater than that of polypropylene mat which was observed to sorb 11.1 g B-heavy oil and 7.8 g machine oil in water (Kobayashi *et al*., 1977). It has also been reported that milkweed and cotton fibers sorbed higher amounts of crude oil than polypropylene fiber and polypropylene web from the surface of an artificial sea water bath containing crude oil and from a crude oil bath (Choi, 1996). Further more, research by agricultural engineer and ASABE Fellow Wm. Stanley Anthony at the U.S. Department of Agriculture (Anthony, 1994) demonstrated that cotton can absorbs up to 80 times its weight in oil with chemical modification.

Kapok sorbent exists as a continous phase on which sorption takes place by hydrophobic interaction and a capillary region in which sorption occurs by capillary action. The capillary action in the fibre assembly occurs between the fibres and/or within the fibres (Praba, Rengasamy and Dipayan, 2011). Micro pores on the wall of the natural fibres and hollow lumen are available in fibres like kapok. In recent years, kapok has received increasing attention as an oil-absorptive material due to its distinct hollow structure and hydrophobic characteristics (Lim and Huang, 2007a,b; Hori et al., 2000; Abdullah et al., 2010; Huang and Lim, 2006) and accordingly, they are preferable as an oil-absorbing material.

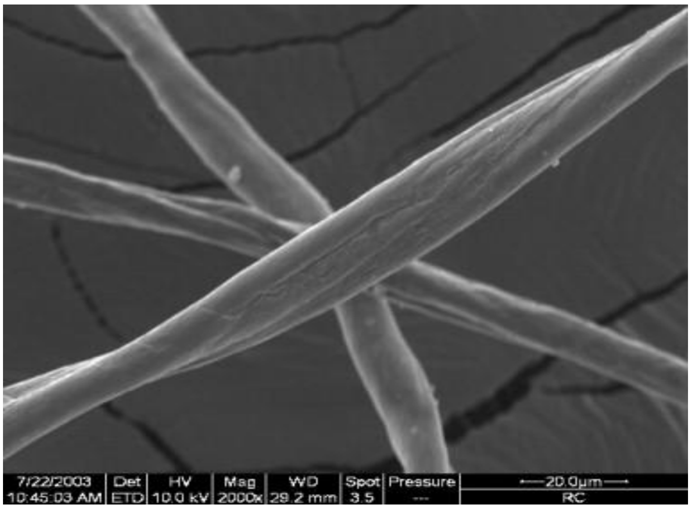



Figure 4: SEM image of raw kapok fibres sourced from Goondiwindi (BC)

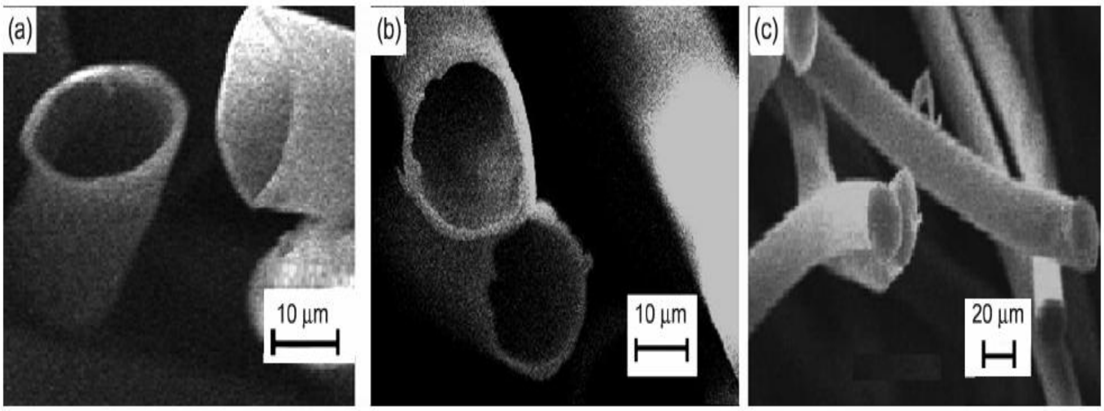



Figure 5: SEM image of fibres kapok, milkweed and polypropylene

## 2.4 SORPTION AND DESORPTION PROCESS

Adsorption and absorption are both sorption processes. Absorption occurs when atoms pass through or enter a bulky material. During absorption, the molecules are entirely dissolved or diffused in the absorbent to form a solution. Once dissolved, the molecules cannot be separated easily from the absorbent. Adsorption is generally classified into physisorption (weak van der Waal's forces) and chemisorption (covalent bonding). It may also occur due to electrostatic attraction. The molecules are held loosely on the surface of the adsorbent and can be easily remove

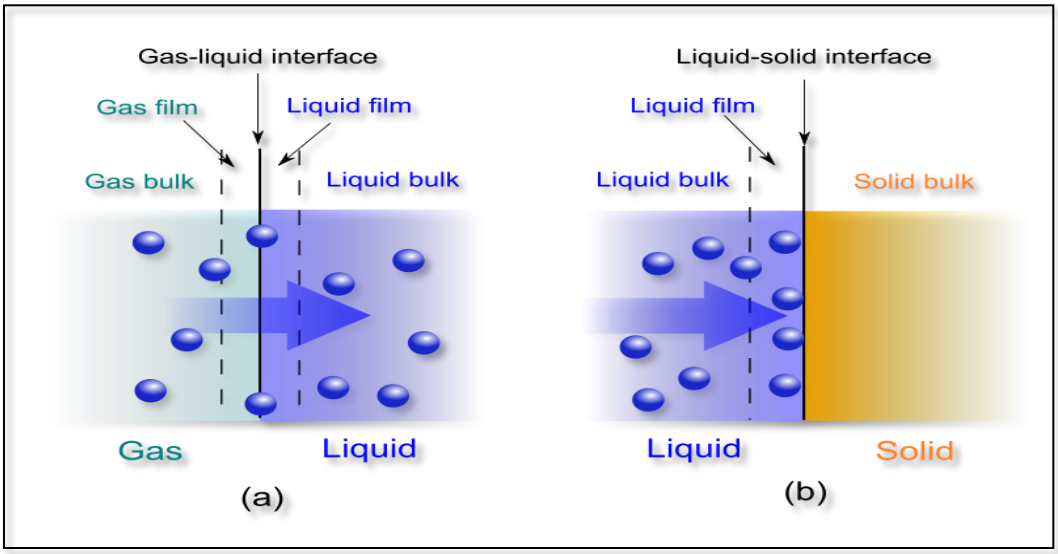



Figure 6: Graphical representation of the sorption process (a) adsorption and (b) absorption. (Source from Google.com)

During oil cleanup process, adsorption and absorption take place between oil and adsorbent. Adsorption and absorption are both sorption process. Unless it is clear which process is operative, sorption is the preferred term. Adsorption is a surface-based process where a film of adsorbate is created on the surface while absorption involves the entire volume of the absorbing substance. Choi *et al.* suggests that volume on or between the fibres is generally responsible for the majority of the material's sorbent capacity. As a result, it is proposed that adsorption by interfibre capillaries is the main adsorption mechanism for kapok sorbent. Natural organic product like kapok were found to absorb significantly more oil compared to commercial synthetic sorbent materials (Choi and Cloud, 1992; Sun *et al.*, 2002; Choi, 1996; Kobayashi *et al.*, 1977).

Sorbent can capture oil by three mechanisms (Hoskin, Underwood and Archambault, 2001); (i) adsorption to the surface of sorbent, (ii) absorption into spaces among agregated granules or fibres of sorebent (secondary absorption); (iii) exploit at least two of these mechanism. Since sorbents may encounter water before oil in a spill cleanup, it is important to know the oil sorption characteristics of water-soaked sorbents (Choi and Cloud, 1992). Because sorbent materials take advantage of the differences in the specific gravities of water and oil to achieve separation, the closer the density of the oil or the grease is to that of water, the lower the efficiency of oil sorption process. On the other hand, clear double layer will enhance absorbtion of oil into sorbent pores until saturation level. The hydrophilicity properties of Kapok also affecting its function to absorb oil. Hence, studies are done to enhance wax layer with chemical modification and thus repelling more water. However in this project, analysis of raw kapok is done without modification to evaluate its natural wax capacity, hydrophobicity and hydrophilicity nature.

Kapok fibre shows overwhelmingly high oil-to-water sorption (O/W) ratios ranging from 19.35 to 201.53. (Ali, Harbawi, Jabal and Yin, 2012). This suggests that kapok fibre is a highly effective oil sorbent even in well-mixed oil-water media. An oil sorbent suitability matrix is proposed to aid stakeholders in evaluating customized oil removal usage of the natural sorbents. Besides the oil sorption capacity, adsorbent retention time is another important parameter to be considered. The weight of recovered oil can cause a sorbent structure to sag and deform, and when it is lifted out, it can release oil that is trapped in its pores causing secondary contamination. That is why, Choi et al. (1993), Wei et al. (2003) and Lim and Huang (2006) reported that retention capacity is an evaluation of the ability of sorbents to retain the absorbed oil during transfer and handling operations.

An adsorbed species present on a surface at low temperatures may remain almost indefinitely in that state. As the temperature of the substrate is increased, however, there will come a point at which the thermal energy of the adsorbed species is such that the species may desorb from the surface and return into the gas phase. This is called the desorption process. Desorption process is studied during the oil recovery and dynamic retention test (Sun et al,2002).

In the absence of decomposition the desorbing species will generally be the same as that originally adsorbed but this is not necessarily always the case. The sorbed crude oil could desorbed from the saturated natural sorbents by a simple mechanical retrieval equipment suggesting that the sorbents could be recycled several times in oil spill cleanup (Choi and Cloud, 1992). The sorbent is considered reusable if a loaded sorbent can easily compress or squeezed to its original size and shape even if there was a tendency toward decrease in sorbent efficiency with repeated sorption and desorption (Elsunni and Collier, 1996). Cotton can be reused through several cycles after removal of absorbed oil although the oil absorption decreases (Deschamps et al., 2003). The irreversible deformation of partial hollow lumens, the contraction of fiber interspace and the presence of residual oil in fiber bundles are all related to the reduction of oil absorbency (Deschamps et al.,2003a,b). Good reusability of Kapok indicated that the material could be applied as efficient oil absorbent for several uses (Radeti, *et al.*, 2003).

The recent finding was succesfully completed and published on Februari 2013. Seshadri Ramkumar, associate professor of Nonwoven materials at The Institute of Environmental and Human Health (TIEHH), has contributed to Fibertect technology. It is a three-layer design consisting of a top and bottom layer of cotton to absorb oil and a middle layer of carbon that absorbs hydrocarbons and harmful carcinogenic vapors released from the oil. The technology is simply taking what nature provides and applying it in new ways. The natural wax on the cotton helps to hold the oil together. So, wax has affinity towards oil, and then the carbon has affinity towards vapor, it holds the vapor. Because Fibertect is all-natural, unlike synthetic plastic booms previously used to clean oil spills, it is 100 percent biodegradable and one sheet can be wrung and reused up to five times. Even though this paper will not discuss more on this finding, his recent technology had proves that the industry is keep improving Kapok potential to be commercialize as sorbent and its nature and microstructure alone is economicaly attractive without the aid of modification.

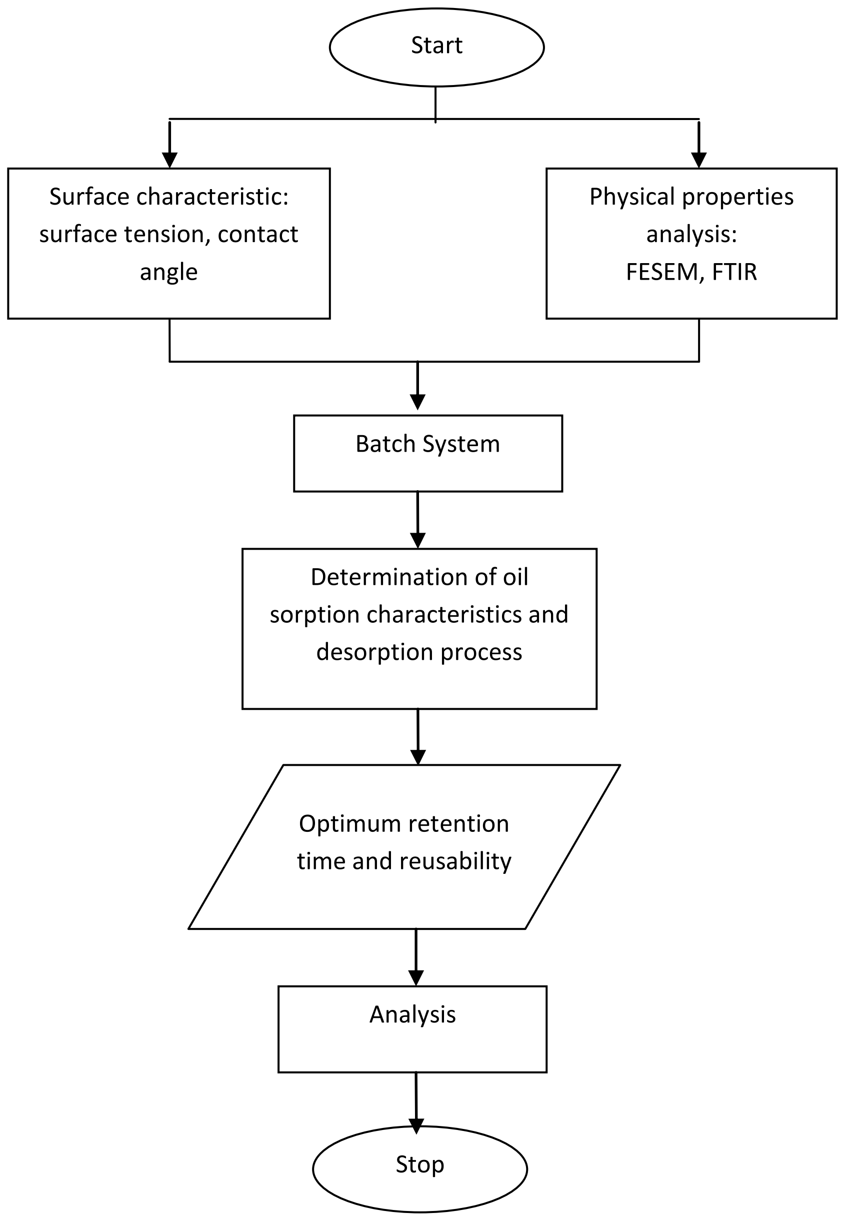
Table 1: Oil sorption capacity of selected material. Source from Journal of Porous Material [8]

| Material | Oil Type | Oil Uptake[a] | Form |
|---|---|---|---|
| Bregoil (waste-wood fibers) | crude | 7 X | sponge |
| Urethane-isocyanate-alcohol polymer | motor | 34.4 | granular |
| Acrylate-nitrile-alcohol polymer | crude | 12 X | device |
| Polypropylene | crude | 7 X | device |
| Polypropylene | light crude | 10 X | fibre/web |
| Polypropylene | light cycle/ heavy crude | 4.5 X | non-woven web |
| Cellulosic fibre | light gas<br>heavy crude | 3.75 X<br>5 X | wooden chips |
| Expanded perlite | light cycle | up to 3.5 X | granular |
| Expanded perlite | heavy crude | up to 3.25 X | granular |
| Milkweed floss (*Asclepias)* | light crude | ~ 40 X | granular |
| Exfoliated graphite | crude | 80 X | device |
| Berthinate (hydrophobic treated peat) | crude | 6 X | granular |
| Gum rubber + polyolefin | crude | 4 X | powder |
| Clay + $NR_4^+$ | ATF[b] | 6 X | powder |
| Clay + $NR_4^+$ | mineral | 0.5 X | powder |
| Cellulose | crude | 18 to 22 X | device |
| Polyvinylalcohol/polypropylene | motor | 2 X | powder |
| Cellulose acetate | crude | 9 X | device |
| $CH_3SiCl_3$ treated fly ash | gear | 0.5 X | powder |
| Hydrous calcium silicate | gear | 4.9 X | powder |
| Hydrous calcium silicate | crude | 6.3 X | powder |
| $CF_3$-functionalised silica aerogel | crude | 4 to 16 X | powder |
| $CF_3$-functionalised silica aerogel | crude | up to 237 X | powder |
| Silica aerogel | crude | < 0.1 X | powder |
| Acetylated rice straw | machine | 16.8 to 24 X | straw |

# CHAPTER 3

## METHODOLOGY

### 3.1 RESEARCH METHODOLOGY



## 3.2 PROJECT ACTIVITIES

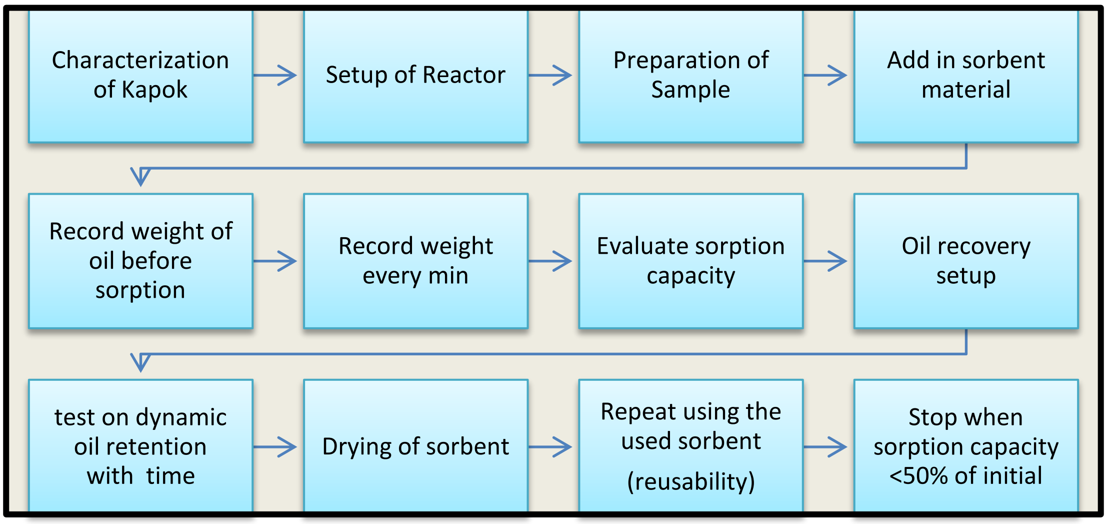



Figure 7: Sequence of project activities

### 3.2.1 Experimental Procedures/Approach

The figure below shows the general experimental procedures that will be implemented in this research project.

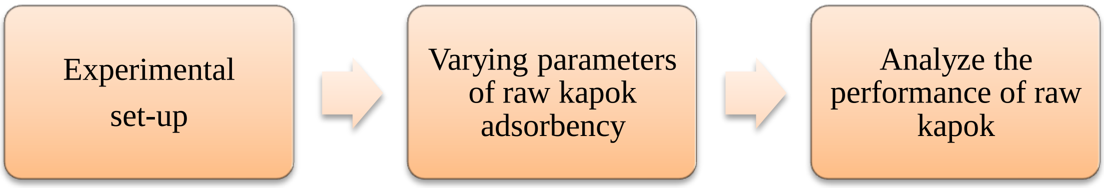



Figure 8: Three steps experimental approach

The reactor used will be a simple batch reactor that being setup manually. The layout of the reactor is shown in the diagram below. To prepare the reactor, seawater from Lumut was taken as sample under test. Diesel crude is used as the experimental oil as it represents low-viscosity oil such as light crude oil and cooking oil. This oil was investigated in favor of the crude oils or lightweight hydrocarbon oils because it is less volatile and has better compositional uniformity, which minimizes transient change in its chemical and physical characteristics during experiment. Without further purification, 40ml diesel was put into the reactor to represent oil spill condition. The reactor was mounted on electronic balance in order to record the change in weight of oil inside the beaker. Wire mesh is used to contain 1g of kapok as the sorbent material and this is how sorption process will take place. After

15minutes, proceed with the desorption process. Experimental setup for this process is shown in the figure below. Observe the oil retention capacity of kapok with time.

Repeat the procedure three times and take the average. If one sample deviate more than 20%, reject and repeat the sample. Replace the sorbent material with polypropylene and analyze the comparison. When conducting the experiment, take note of the sorption time and leaching of oil. The saturated sorbent is further process by squeezing oil from it using piston. The unsaturated sorbent was left to dry and recycle back as sorbent. The cycle was stop when the sorption capacity is less than 50% of initial capacity. Figure 2 below shows two main experimental setup being used throughout the research.

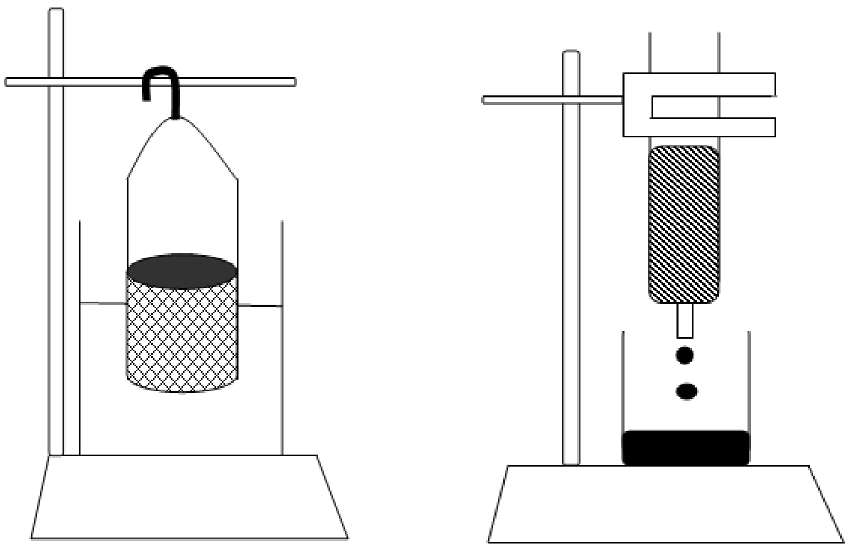


Figure 9: Experimental setup (a) Batch reactor for sorption process and (b) oil recovery using desorption process

### 3.2.2 Equation and Parameter

$$\text{Oil Sorption Capacity} = \frac{[S_I - S_F]}{S_A}$$

Where:

$S_I$= the weight of the oil before sorption inside the oil bath (g)

$S_F$ = the weight of the oil inside the beaker at1 min dripping (g)

$S_A$ = dry weight of kapok (g)

The percentage of dynamic oil retention was calculated as follows:

$$\text{\% dynamic oil retention} = \frac{W_{t=1}}{W_{t=i}} \text{ x } 100$$

Where:

$W_{t=1}$ = weight of oil bath (g) at1 min dripping

$W_{t=i}$ = weight of oil bath (g)at t min dripping, $i = 2,3,4 \ldots$.

The value of surface tension is calculated from the drop shape of the image recorded by SEM, using the Laplace equation:

$$\triangle P = \frac{2\gamma}{r} \cos\theta$$

$\triangle P$ = pressure difference between gas and liquid phase (bar)

$\gamma$ = surface tension ($\frac{MN}{m}$)

$\theta$ = contact angle

r = droplet radius (mm)

### 3.2.3 Materials and Tools

Table 2: List of materials and tools needed

| Materials | Equipments/ Tools |
| --- | --- |
| Raw Kapok | SEM Microscope |
| Sea Water | FTIR Equipment |
| Crude Oil | Electronic Balance |
| Sodium Chloride | Mounted Shaker |

Figure 10: Scanning Electron Microscope (SEM)

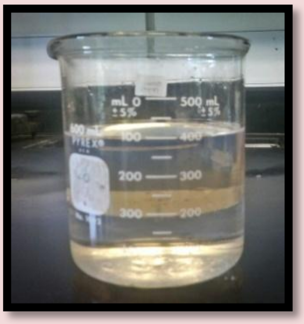
Figure 3.3 shows SEM microscope that is available in Block P. This equipment is very helpful in determining the micro-structure and porous surface of kapok fibre. The image of SEM is shown in Figure 2.3. The image resolution is adjustable.

### 3.2.4 Work Flow Summary

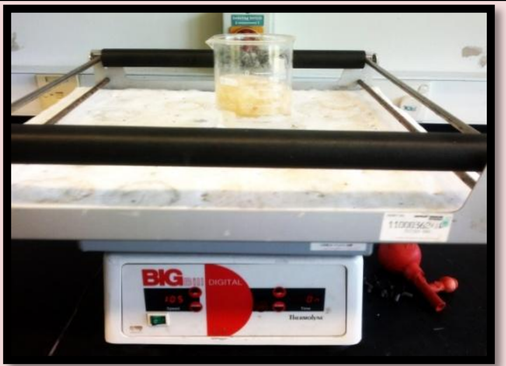
**TEST 1: Characterization of raw and saturated Kapok**

- FESEM and FTIR analysis to study the porous and cellulosic structure
- Prepare 2 sets of sample for raw and saturated sorbent

**TEST 2: Evaluate oil sorption capacity**



- Prepare 200ml of oil and 200ml of water inside a beaker.
- Add in 1g of raw kapok sorbent.



- To give the wave and environmental effects, put the sample on a stirrer at 115rpm.
- Let adsorption process takes place for 30minutes

**TEST 3: Evaluate oil retention behavior**

- Recover oil from the saturated sorbent using experimental setup (b)
- Weight of oil retained at the bottom is recorded for 30 minutes
- After 30 minutes, squeeze out the left oil mechanically using syringe
- Let the sorbent dry naturally for few days

**TEST 4: Evaluate the reusability of kapok**

- Repeat test 2 by using the recovered sorbent from test 3
- Stop the cycle when the sorption capacity fall below 50% of initial sorption capacity.

### 3.3 KEY MILESTONE

Key milestones was set initially to guide the progress of gantt chart and this guideline will make sure that project being completed within specified target.

| Bil | Milestone | Week |
|---|---|---|
| 1 | Selection of project title | 1 |
| 2 | Completion of preliminary research | 3 |
| 3 | Completion of materials and apparatus lists | 5 |
| 4 | Completion of Extended Proposal report | 6 |
| 5 | Completion of Oral Proposal Defense | 9 |
| 6 | Completion of research about sorbent process | 10 |
| 7 | Preparation of experimental procedures | 11 |
| 8 | Submission draft of Interim Report | 13 |
| 9 | Submission of Interim Report | 14 |
| **Bil** | **Milestone** | **Week** |
| 2 | Completion of characterization process | 2 |
| 3 | Preparation for experimental setup | 3 |
| 4 | Completion of primary phase experiment | 7 |
| 5 | Submission of Progress Report | 8 |
| 6 | Preparation Pre-Sedex | 11 |
| 7 | Completion of secondary phase experiment | 12 |
| 8 | Submission of Dissertation (soft bound) and Technical Paper | 13 |
| 9 | Completion of Oral Presentation | 14 |
| 10 | Submission of Dissertation (hard bound) | 15 |

## 3.4 GANTT CHART

### 3.4.1 Final Year Project I Guideline
**[Planning Phase]**

Table 3: Timeline allocated for FYP I (Jan-Apr2013)

| NO | DETAIL / WEEK | 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 Defence | | | | | | | | | | | | | | | |
| 4 | Preparation for Oral Proposal Defence | | | | | | | | | | | | | | | |
| 5 | Oral Proposal Defence Presentation | | | | | | | | | | | | | | | |
| 6 | Detailed Literature Review | | | | | | | | | | | | | | | |
| 7 | Preparation of Interim Report | | | | | | | | | | | | | | | |
| 8 | Submission of Interim Draft Report | | | | | | | | | | | | | | | |
| 9 | Submission of Interim Final Report | | | | | | | | | | | | | | | |

### 3.4.2 Final Year Project II Guideline [Execution Phase]

Table 4: Timeline for FYP II (May-Aug2013)

| NO | DETAIL WEEK | 1 | 2 | 3 | 4 | 5 | 6 | 7 | | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Characterization of Material | | | | | | | | | | | | | | | |
| 2 | Experimental Setup | | | | | | | | | | | | | | | |
| 3 | Experimental Work | | | | | | | | | | | | | | | |
| 4 | Submission of Progress Report | | | | | | | | | | | | | | | |
| 5 | Experimental Work Continue | | | | | | | | | | | | | | | |
| 6 | Pre SEDEX | | | | | | | | | | | | | | | |
| 7 | Submission of Dissertation (softbound) and technical paper | | | | | | | | | | | | | | | |
| 8 | Oral Presentation | | | | | | | | | | | | | | | |
| 9 | Submission of Dissertation (hardbound) | | | | | | | | | | | | | | | |

# CHAPTER 4

## RESULTS AND DISCUSSION

### 4.1 CHARACTERIZATION PROCESS

#### 4.1.1 FESEM Analysis

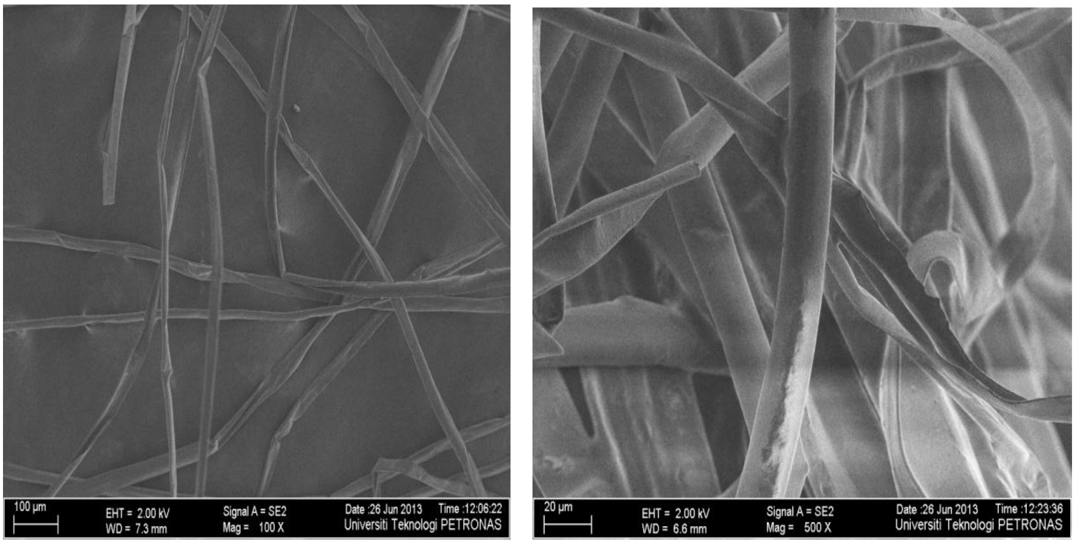



Figure 11: (a) FESEM image of raw kapok while and
(b) FESEM image of saturated kapok

Scanning electronic microscope model was used to study the fiber surface morphology. Figure 3 (a) presented SEM micrograph of surface morphologies of raw kapok. It shows strands of cellulosic fiber entangled to each other. The fibrous material increases the sorption surface area of raw kapok. Figure 3 (b) on the other hand is the representation image of saturated kapok after being dried and compressed to recover oil. It shows the expansion of fibrous entrains due to oil leftover at the surface pores. Thus, effecting the reusability of kapok sorbent.

### 4.1.2 FTIR Analysis

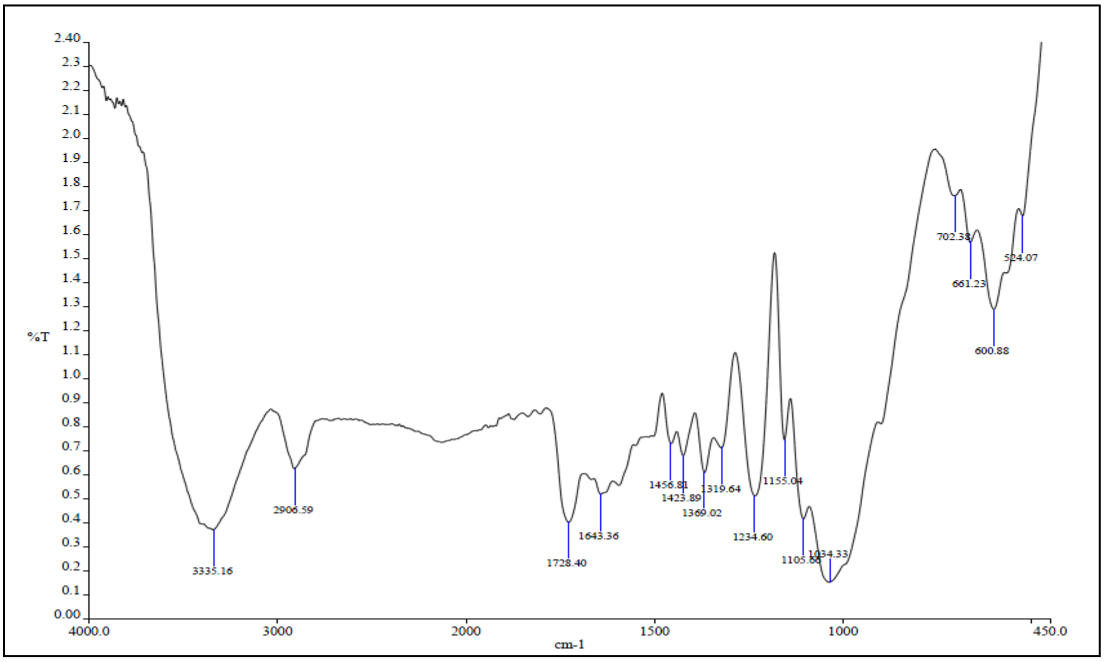



Figure 12: The FTIR analysis for raw kapok. It shows the nomenclature of existing particle on kapok

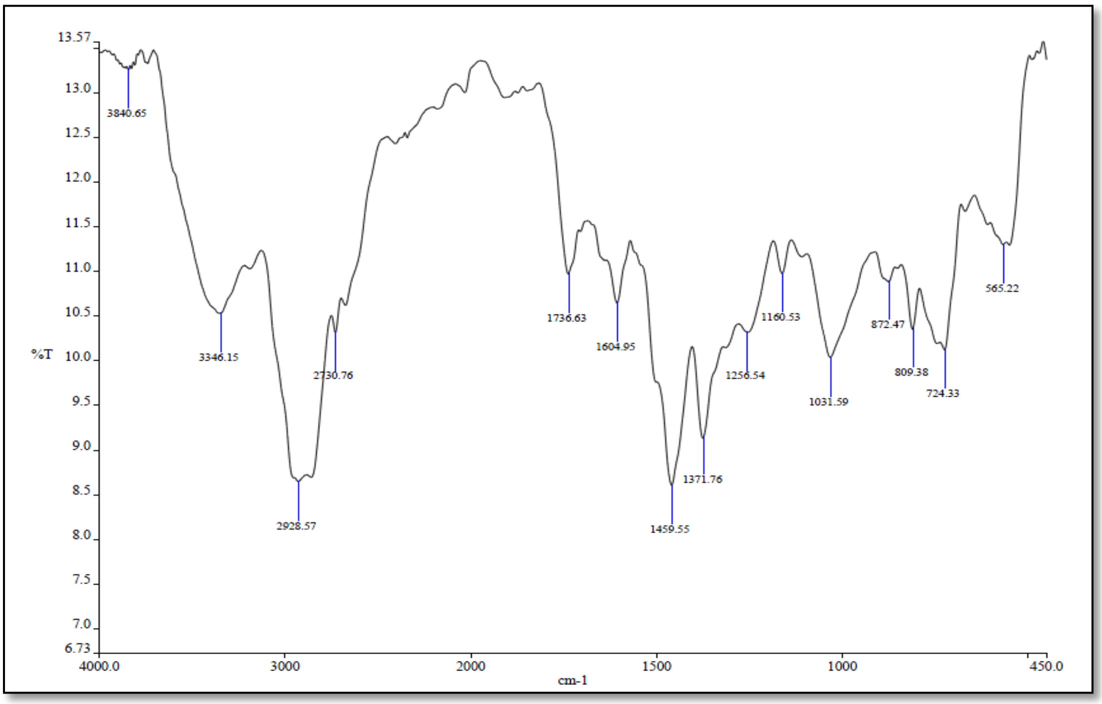



Figure 13: The FTIR analysis for saturated kapok. The O-H bond is stretch at the middle indicating the presence of hydrocarbon material.

## 4.2 OIL SORPTION CAPACITY

### 4.2.1 Test on the Hydrophobicity

The aim of this test is to see the performance of kapok to absorb oil at the presence of seawater. The hydrophobicity of kapok to repel water and absorb oil is evaluated. Throughout the process, amount of oil escaped and lost was tabulated and shown in Table 2 and Figure 5 below:

Table 5: Summary of data from the hydrophobicity test

| Number of Trials | Remaining Level (ml) | | Vol oil escape(ml) | Vol oil recovered (ml) | Vol Lost(ml) |
|---|---|---|---|---|---|
| | Oil | Water | | | |
| 1st trial | 100 | 200 | 34 | 60 | 6 |
| 2nd trial | 130 | 200 | 20 | 46 | 4 |
| 3rd trial | 110 | 200 | 23 | 62 | 5 |
| 4th trial | 120 | 200 | 29 | 45 | 6 |
| 5th trial | 100 | 200 | 40 | 47 | 13 |

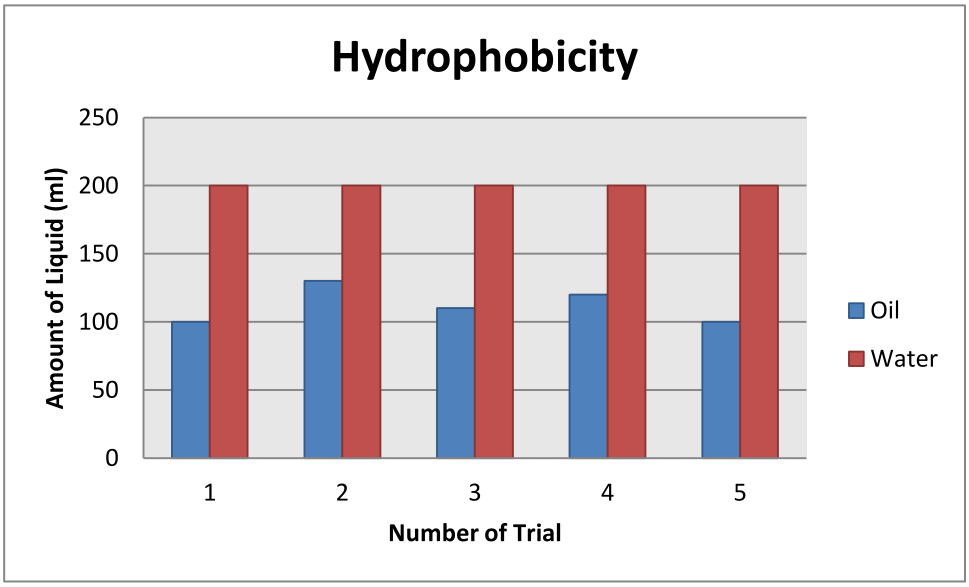



Figure 14: Oil and water lever at the end of trial

It shows that water does not affecting the sorption process. Kapok is repelling water. The density factor that create double layer of oil and water also enhance the sorption process.

From the data in Table 2, oil sorption capacity was calculated using the equation given in 3.2.3 (refer to appendix A) and the result is presented in the graph below:

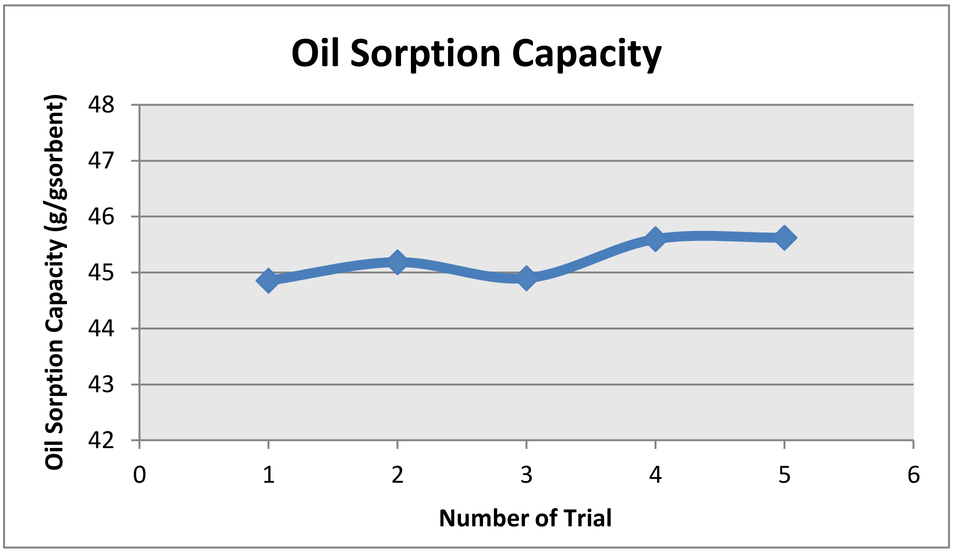



Figure 15: Oil sorption capacity of kapok fiber

### 4.2.2 Test on Effect of Sorbent Weight

The aim of this test is to see the relationship between different weight of sorbent to oil sorption capacity of kapok. The graph shows a directly proportional relationship.

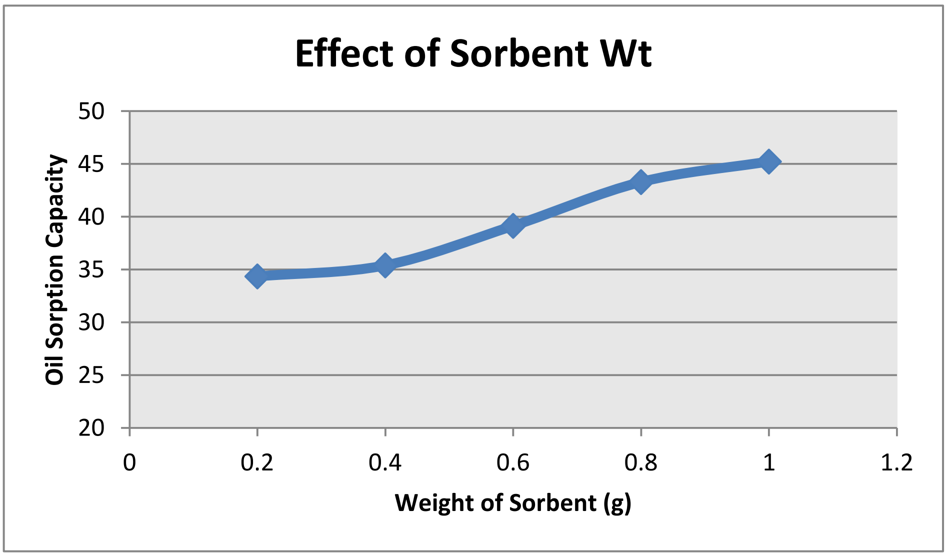



Figure 16: Graph of capacity versus sorbent weight

### 4.2.3 Test on 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 15min. The unsaturated sorbent then draws back the oil in, thus providing a sinusoidal graph as follows. This is what caused secondary pollution to happen.

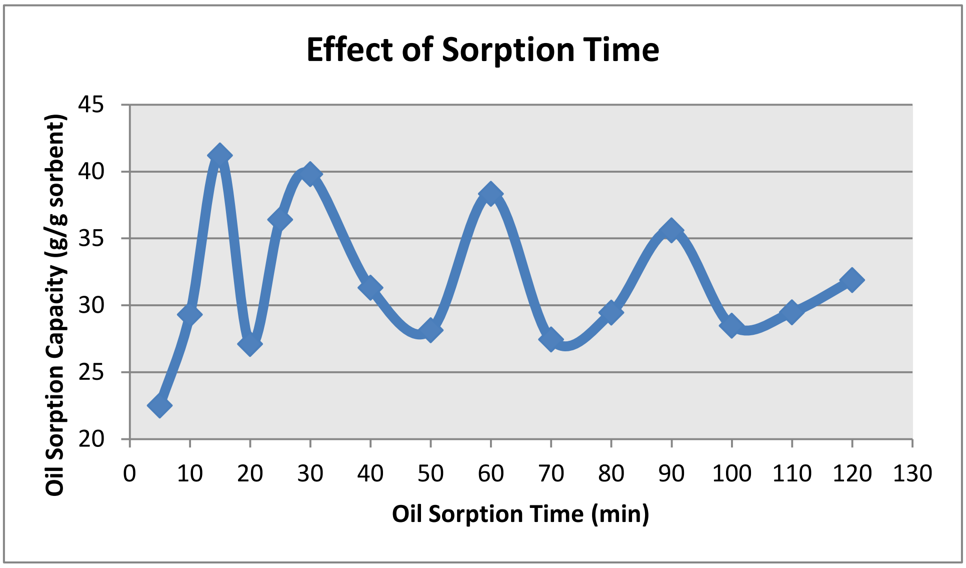



Figure 17: Graph of capacity versus sorption time

## 4.3 DYNAMIC RETENTION TIME

The aim of this test is to evaluate the dripping behavior of oil from saturated kapok. The kapok will then be dried naturally and reused back for reusability test. Figure below shows the image of saturated, wet recovered and dry sorbent.

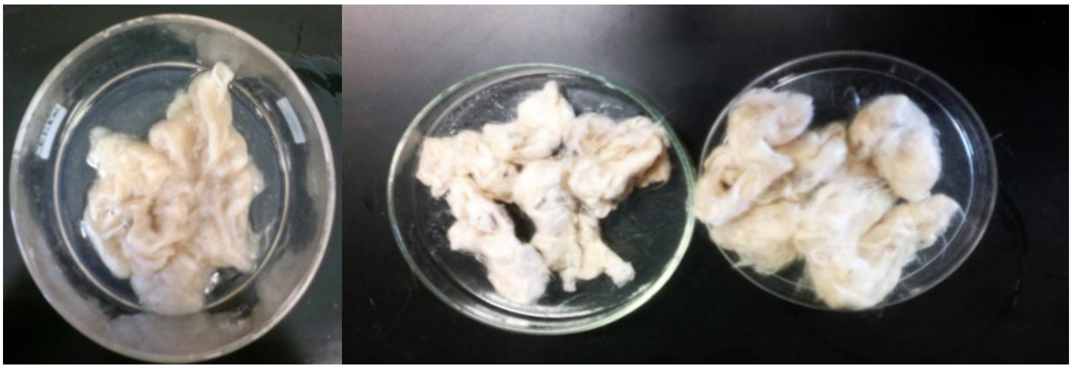


Figure 18: Image of (a) saturated kapok (b) wet kapok after the recovery process and (c) dry kapok ready for reusability

Figure 16 shows that value of oil retained is more rapid at the first 5 minutes of oil recovery process. Figure 17 thus is showing that the dynamic percentage of oil retention with respect to dripping time is decreasing. In general, this shows that the secondary pollution of oil during spill cleanup is more likely to happen at the early stage after the sorbent reach saturation level.

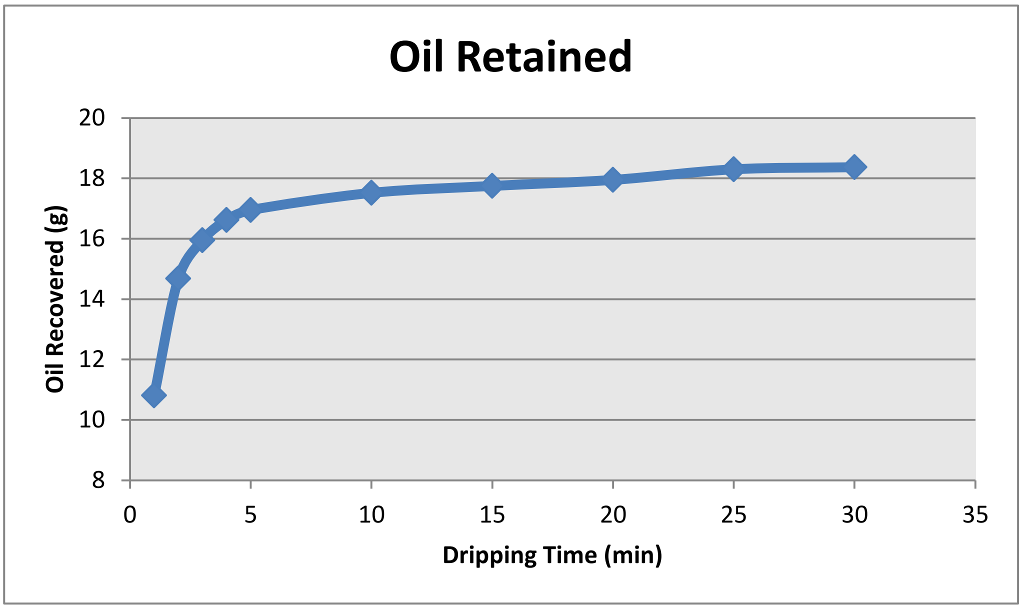



Figure 19: Graph of oil recovered (g) versus dripping time

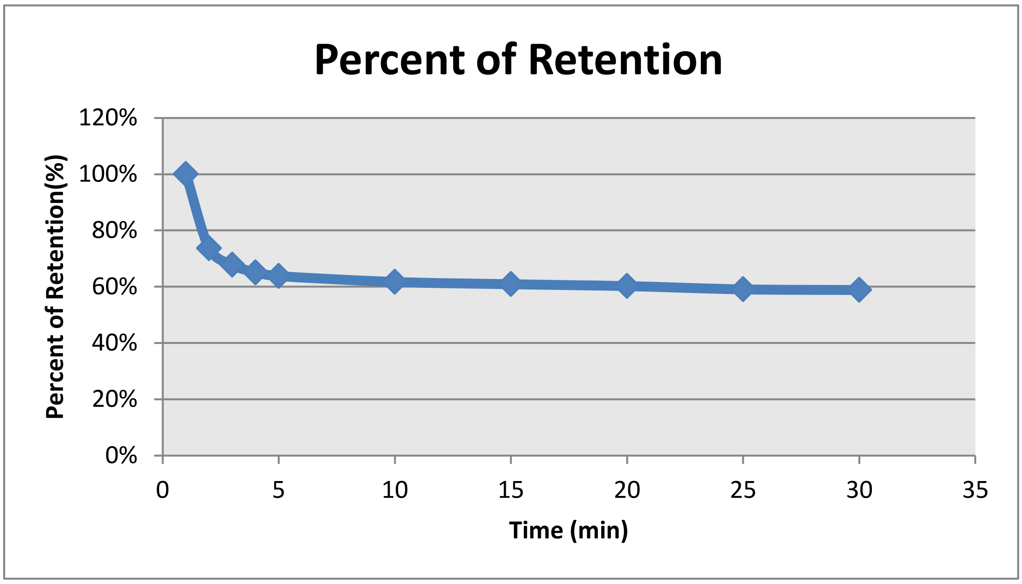



Figure 20: Graph of dynamic retention (%) versus time

## 4.4 REUSABILITY OF KAPOK

The figure below shows the sorption capacity of kapok sample after 3-4 cycles of usage without any chemical treatment.

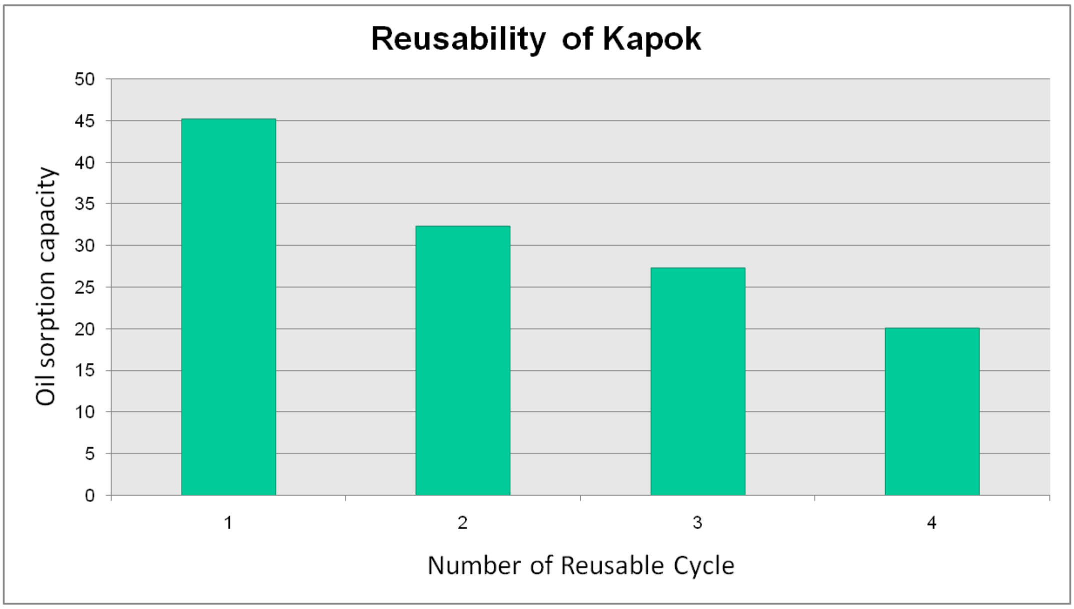



Figure 21: Graph of oil sorption capacity versus cycle of reusability

Base on Figure 9, it can be conclude that the sorption capacity of kapok decreased with number of cycles. The finding proves that kapok can function efficiently up to 3 cycles only. Even though some literature proves that it can goes up to 4th cycles (Choi,1992), due to difference in drying techniques and treatment of saturated sorbent, this variation happened. To enhance the reusability, drying with the aids of chemical is normally used.

# CHAPTER 5

## CONCLUSION AND RECOMMENDATION

In general, this research was done to assist the oil spill cleanup technology and provide the best solution and sorption parameters for new biodegradable sorbent, raw kapok. Kapok, base on the characterization, contains fibrous cellulose that can absorb oil efficiently. A series of test had been strictly conducted in a simulation scale. It is found that raw kapok, without any chemical modification, has the capacity to absorb up to 45% of its original weight as average. This value is comparable to the commercialized synthetic sorbent.

It was also found that, the sorption capacity of sorbent will increase with its weight. The constraint of this sorbent is sorption time. The optimum sorption time for kapok fiber is 15minutes, longer than the allocation could leads to secondary pollution and sinusoidal trend. Besides, during the oil recovery process, the retention behavior of saturated kapok had also been observed. Rapid dripping occurs at the early stage after saturation, thus control on sorption time is important to avoid secondary pollution and huge escape of absorbed oil. This research also had successfully proves that kapok has the capacity to be reuse up to three times.

For future recommendation, this paper has suggested the modification of kapok porous structure via chemical mean. Chemical like sodium hydroxide might be effective to enhance the capillary wall and enlarge the pore opening of kapok fiber. The drying method also can be improved with a better aids and technology. In short, kapok has a great capacity to become a sorbent material that is also natural and biodegradable. The objective of this research is verified.

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# APPENDICES

## Appendix A : Calculation of oil sorption capacity

| Samples | wt dry(g) | wt wet(g) | sorption capacity |
|---|---|---|---|
| 1st trial | 1.00 | 45.855 | 44.855 |
| 2nd trial | 1.00 | 46.185 | 45.185 |
| 3rd trial | 1.00 | 45.899 | 44.899 |
| 4th trial | 1.00 | 46.598 | 45.598 |
| 5th trial | 1.00 | 46.623 | 45.623 |
| | | | 45.232 |

$$Oil\ sorption\ capacity = \frac{Wt\ of\ wet\ sorbent(g) - Wt\ of\ dry\ sorbent(g)}{Wt\ of\ dry\ sorbent(g)}$$

$$= \frac{Wt\ of\ oil\ adsorbed(g)}{Wt\ of\ sorbent(g)}$$

$$= \frac{45.855g - 1.00g}{1.00g}$$

$$= 44.855\ \frac{g\ of\ oil}{g\ of\ sorbent}$$

**Appendix B: Calculation on dynamic oil retention**

| Time (min) | Weight of Oil Recovered (g) | Percent of retention (%) |
|---|---|---|
| 1 | 12.70 | 100% |
| 2 | 14.39 | 88% |
| 3 | 15.65 | 81% |
| 4 | 17.24 | 74% |
| 5 | 18.40 | 69% |
| 10 | 19.21 | 66% |
| 15 | 19.56 | 65% |
| 20 | 19.79 | 64% |
| 25 | 20.62 | 62% |
| 30 | 21.12 | 60% |

$$Dynamic\ oil\ retention = \frac{Wt\ oil\ at\ the\ 1st\ minute}{Wt\ of\ oil\ at\ other\ particular\ minutes} \times 100$$

$$= \frac{12.70g}{14.39g} \times 100$$

$$= 88\%$$