

**Contact Angle Measurement for Agricultural Materials**

by

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

**JANUARY 2012**

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## **CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the  
Chemical Engineering Programme  
Universiti Teknologi PETRONAS  
in partial fulfilment of the requirement for the  
BACHELOR OF ENGINEERING (Hons)  
(CHEMICAL ENGINEERING)

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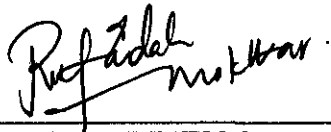
UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2012

## **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.

A handwritten signature in black ink, appearing to read 'Ruffadah Mokhtar', written in a cursive style.

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**RUFAIDAH BINTI MOKHTAR**

## ABSTRACT

The study is focused on identifying the best possible contact angle measurement method in order to determine contact angle for agricultural materials. Sugarcane (*Saccharum Officinarum*) bagasse, kapok (*Ceiba Pentandra*) and rice husk (*Oryzae Sativa*) are selected to be tested for their contact angle and then compared against the established synthetic oil sorbent, polypropylene. The objectives of this study are to measure the contact angles for agricultural materials with different types of oil (crude oil, engine oil, used engine oil) using the best identified method. The results are then compared against known value of contact angle for polypropylene. The best material for oil sorbents can be identified by measuring contact angle; the lower the contact angle of with liquid, the better the material is at absorption. This will be an indicator of the absorption ability of these agricultural materials.

Experimental works are conducted in order to achieve the set objectives. The materials are prepared accordingly before experiments are conducted to measure their contact angles. The equipment used is interfacial tensiometer which measures the contact angle based on a picture taken of the liquid drop on a solid surface. The results show that kapok has the lowest contact angles for all types of oil and it is even better than polypropylene at absorption. The results for sugarcane bagasse and rice husks vary in term of which is better at absorption. However, generally the contact angle values for agricultural materials are lower than that of polypropylene. This means that the agricultural materials are better at absorption than synthetic materials. Thus, agricultural materials are proven to be a promising alternative for the synthetic sorbent materials currently being use to absorb oil during oil spill contamination in the sea.

## **ACKNOWLEDGEMENT**

First of and foremost, the author would like to thank Allah Almighty for giving me the strength to complete this Final Year Project. The author would also like to thank the project supervisor, Dr. Mohanad El-Harbawi for giving courage and guidance in conducting this project. His continuous support, patience, enthusiasm and motivation have been valuable throughout the research and thesis-writing process.

Not to forget, many thanks to the Final Year Project Co-ordinator, Dr Lukman Ismail, for providing all the needed information to conduct the study. The author would also want to thank the lecturers and staffs of Chemical Engineering Department (especially from Ionic Liquid Centre), Universiti Teknologi PETRONAS for their utmost support, cooperation and help in the completion of this project.

Last but not least, deepest appreciation to my parents, family and friends for their kind words of encouragement and constructive comments from the start of the project right through until the end.

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# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

Contact angle is defined as:

The contact angle is the angle at which a liquid/vapour interface meets a solid surface. The contact angle is specific for any given system and is determined by the interactions across the three interfaces. Most often the concept is illustrated with a small liquid droplet resting on a flat horizontal solid surface.

“The solid-liquid contact angle is used as a measure of wettability and the surface free energy of solids in many diverse fields such as mineral and coal beneficiation, petroleum engineering, and the manufacture of pharmaceutical powders, cosmetics, pigments, paints and paper. Therefore, contact angle measurements are fundamental to many processes.” (Eratak, 2005).

There are a number of techniques used for the measurement of contact angle currently being used. The wettability of a powder is important, especially in chemical and pharmaceutical technologies when considering the manufacturing process and the properties of the final product. However, contact angle methods for powders are problematic. The common measurement techniques are the sessile drop method and the liquid penetration methods (Teipel and Mikonsaari, 2004).

The use of agricultural materials as an alternative is considered as a promising alternative for the synthetic sorbent materials currently being use to absorb oil during oil spill contamination in the sea. Some of these materials are even better than the synthetic materials in use due to its sorption qualities (Ali et al., 2010, Lim and Huang, 2007). However, this can only be determined through the measurement of its

contact angle. Contact angle measurement for natural sorbents fibres such as rice husks, sugarcane bagasse and kapok is a new field that is yet to be fully explored using some of the few methods available.

In this research work, experiments will be conducted in order to determine the best contact angle measurement methods for agricultural materials using liquid penetration method.

## **1.2 Problem Statement**

Oil removal is usually done through absorption using synthetic products such as polypropylene. This material is hazardous as it releases toxic gases upon burning. Several alternative materials from waste have been identified that have similar sorbent properties. This approach is environmentally-friendly as waste is used to treat waste.

The best material for oil sorbents can be identified by measuring contact angle; the lower the contact angle of with liquid, the better the material is at absorption. However, as of now, there is no known established standardised method for the measurement of contact angles for agricultural materials.

Direct measurement of contact angle of powders is also impossible. The direct measurement of contact angle is not applicable to small powder particles (Siebold et al., 2000).

## **1.3 Objective**

The aim of the study is to identify the best possible method for contact angle measurement of agricultural materials. The further objectives of my research are:

- To measure the contact angles for agricultural materials
- To compare the results of contact angle for natural materials with polypropylene

## **1.4 Scope of Study**

In order to accomplish the objective of the research study, a comprehensive literature review was conducted in order to gather as much information as possible from various published resources either from the internet or library. The scope of this work include to:

- Study the best method for measurement of contact angle with different liquid
- Study the measurement using many different liquid penetration methods
- Study the contact angles of different agricultural materials (kapok, sugarcane bagasse and rice husks) in comparison with synthetic materials

## **1.5 The Relevancy of the Project/Project Significance**

This project is very significant as it can help to solve the problem arising from oil spill contamination. Rapid global industrialization coupled with consumer need for energy resources have driven huge demand for fossil fuel for both industrial and end-user markets. This presents an environmental drawback in terms of possible oil spill contamination that may arise during sea transportation of petroleum products (Ali et al., 2010). Cost-effective sorbents can be utilized to remove oil spills if alternative sorbent materials can be found to replace current synthetic materials in used. Natural sorbents can replace synthetic sorbents as it is a technique of using waste to treat waste. Other than being cost effective, this is also a more environmentally-friendly approach. Therefore, this project will hopefully be beneficial to R&D Industry and produce alternative materials for oil absorption to remove oil spills.

## **1.6 Feasibility of the Project within the Scope and Time Frame**

The author has been given 2 semesters to accomplish the project under course of Final Year Project 1 for the previous semester and Final Year Project 2 for the current semester. The first semester has been utilized to document a thorough analysis of available literature and current available methods for the measurement of

contact angle for agricultural materials. Experiments to determine the best method for contact angle measurement are conducted in the second semester followed by contact angle measurement for different types of agricultural materials with different types of liquid (water, oil). The project is feasible to be accomplished at the specific time frame given.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Agricultural Materials**

Agricultural materials have been found to exhibit excellent oil sorption capabilities for diesel, crude, new engine and used engine oils (Ali et al., 2010). These provide a strong basis for the development of alternative sorbent materials to be used to clean up oil contamination in the sea.

The natural sorbent materials to be used in this project would include kapok, rice husks and also sugarcane bagasse due to their various characteristics and properties which include hydrophobicity, oleophilicity and biodegradability. The contact angle for these materials will be compared to each other along with other properties in order to determine the best alternative material to replace current synthetic material – the most widely used commercial sorbent being polypropylene.

The important physical properties which are related to the experiment to be conducted are the particle size in powder form, elemental compositions (C, O, Si), average density and also sorption capacities (oil and water). The effect of packing density, the oil types and solvent treatment on the sorption characteristics of kapok has been studied by Abdullah et al., (2009) and Ali et al., (2010). This will provide a strong basis for the experiments to be conducted for this project.

Rengasamy et al., (2010) has identified that with the increase in the demand for oil products there is also an increase of risk in spillage during its production, transportation, storage and usage of oil. The removal of crude oil and petroleum products that are spilled at sea is a serious problem of the last few decades. There are some oil spill remediation products currently available including but not limited to

dispersants, absorbents, bioremediation agents and other miscellaneous products such as surface cleaners, gelling agents, demulsifiers, solidifiers and many more that can be used to clean up oil spill.

There are a few methods of control for oil spill contamination in the sea. The present methods for oil spill removal are numerous. However, it is usually divided into three main categories; physical methods, chemical methods and also biological methods (Bordosern, 2004). Physical methods involved the use of booms, skimmer and also absorbent. One of the most economical and efficient methods for removing oil contamination is oil sorption by sorbents. Oil sorbents can concentrate and transform liquid oil to the semi-solid or solid phase, which will then be removed from the water body and handled without significant oil draining out. Preferably, the sorbent materials must demonstrate fast oil sorption rate, high oil sorption capacity (oleophilicity or lipophilicity), low water absorption, high oil retention capacity during transfer, high recovery using simple methods of the absorbed oil, good reusability, high buoyancy, and excellent physical and chemical resistances against deformation, photodegradation and chemical attacks (Lim and Huang, 2007).

The sorbent materials can be further divided into three major classes: inorganic mineral products, organic products and also organic vegetable products. Mineral products used include perlite, vermiculites, sorbent clay, graphite and also diatomite. One of the most widely used commercial synthetic sorbent is polypropylene due to its sorption capabilities and physical properties. However, polypropylene is not biodegradable, and hence possesses environmental problems as they degrade very slowly as compared to vegetable or natural-based products. Thus, the agricultural fibre-based oil sorbents could be an interesting alternative to the synthetic oil sorbents as apart from being environmentally-friendly, they are also inexpensive and readily available. Such agricultural products as rice husks and sugarcane bagasse are waste from industry and hence, the utilisation in this manner will results in savings in terms of disposal and treatment fee.

### 2.1.1 Kapok

Kapok fibre is an agricultural product which is very high in oil absorbency characteristics. The kapok fibres typically comprise of 64% cellulose, 13% lignin and 23% pentosan (Kobayashi et al., 1977). The product also contains waxy cut on the surface which makes the material to become water-repelling, meaning that the material water absorbency is quite low.

Abdullah et al., (2009) studied the effect of packing density, the oil types and solvent treatment on the sorption characteristics of kapok. The physiochemical and sorption characteristics of Malaysian kapok with diesel and engine oil were also studied. Kapok exhibited high hydrophobic-oleophilic characteristics attributed to hollow lumen and its waxy surfaces. This is especially useful as it can be used to absorb oil but not water during oil spillage in the sea. The sorption capacities of kapok for three types of oils were significantly higher than those of polypropylene (Lim and Huang, 2007).

Malaysian kapok is said to have great potential as effective natural oil sorbent due to its high sorption and retention capacity, structural stability and high reusability. As kapok and milkweed fibre have low cellulose content, slow degradation is an advantage in fresh and marine water. According to Ali et al., (2010), both kapok and sugarcane bagasse exhibit excellent oil sorption capabilities for diesel, crude, new engine and used engine oils which can be used for alternative to the synthetic materials in use. This is supported by the Figures 2.1-2.3 on the water and oil sorption capabilities of these materials.



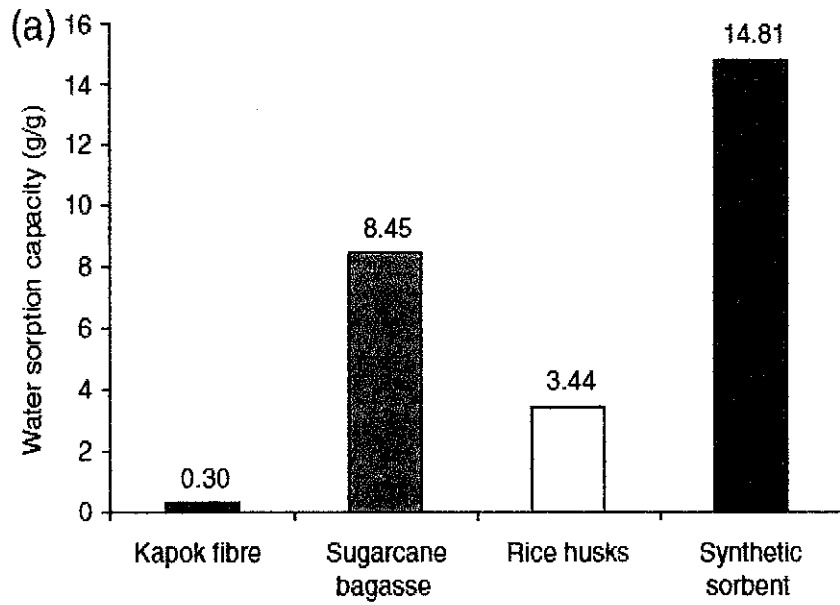


Figure 2.1: Water sorption capacities of materials (Ali et al., 2010)

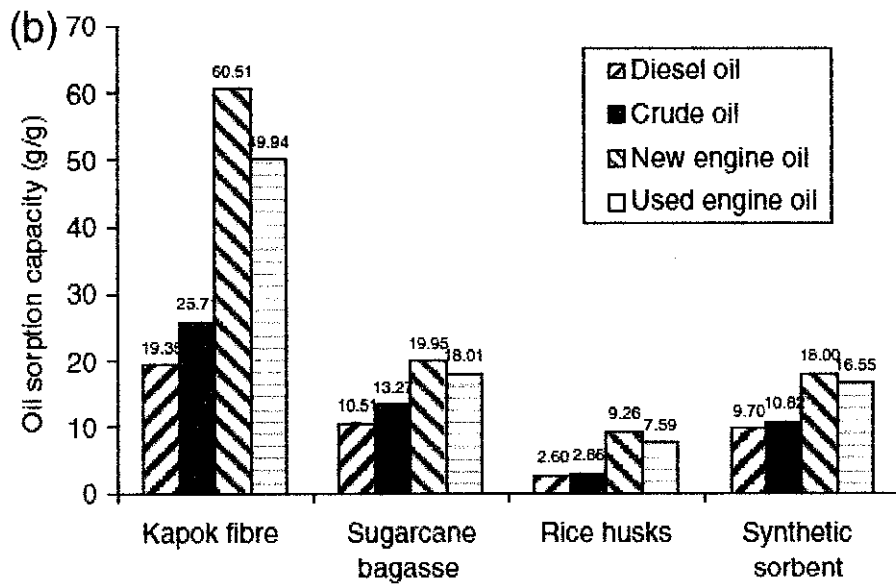


Figure 2.2: Oil sorption capacities of materials (Ali et al., 2010)

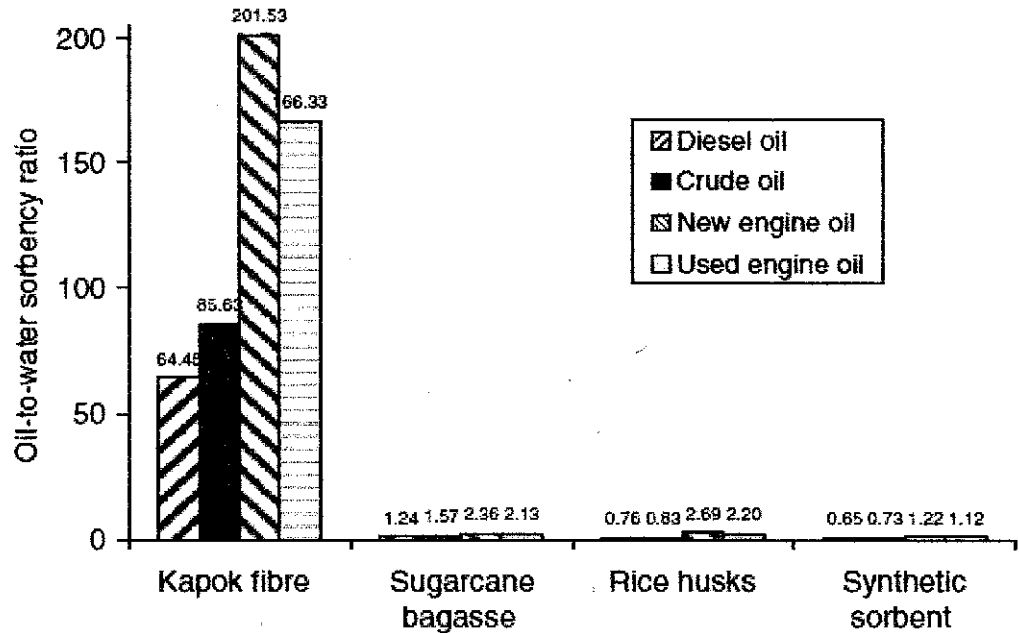


Figure 2.3: Oil to-water sorbency ratios of materials (Ali et al., 2010)

### 2.1.2 Sugarcane Bagasse

Sugarcane bagasses are readily available and can be easily found in Malaysia. The production of sugar from sugarcane is one of the main agricultural industries in Malaysia. Sugarcane bagasse is a waste produced in large quantities by the sugar industries. Generally, 1 tonne of sugarcane generates around 280kg of bagasse which is about 28% of the total weight (Sun et al., 2004). The fibrous by-product remaining after sugar extraction from sugarcane contributes to a large number of wastes from the industry. Currently, the utilisation of the bagasse is still limited to be used as animal feed. There is no other option to further utilise the waste into other useful products.

Sugarcane bagasse is a low density and fibrous material with a very wide range of particle size and high moisture content (10-15%). It is naturally consisting of three components which are; pith, fibre and rind mixed in different proportions. There is a significance difference in shapes and sizes of these three components. The rather regular shape of spongy pith particles with a near unity length/width ratio can be approximated by a spherical shape. The shape of fibres with high length/width ratios

can be modelled by cylinders. The large rind material roughly comprises rectangular particles with high length/width ratios. Some fibres are often adjacent to the inner wall of the rind particles (Rasul et al., 1999).

### 2.1.3 Rice Husks

Another type of common waste in Malaysia is rice husk. It is a waste product of the agricultural activity in most countries in Southeast Asia. Rice husks have posed a major problem of disposal to the rice milling industry in Malaysia, and also throughout the world. Efforts have been made since the last 20 years in finding ways to utilise the waste. The special structure-skeleton of cellular cell of rice husk means that it can be used to absorb oil (Lim and Huang, 2007).

## 2.2 Methods for Contact Angle Measurement

Contact angle,  $\theta$ , is a quantitative measure of the wetting of a solid by a liquid. It is defined geometrically as the angle formed by a liquid at the three phase boundary where a liquid, gas and solid intersect. The equilibrium contact angle is specific for any given system and is determined by the interactions across the three interfaces. Most often the concept is illustrated with a small liquid droplet resting on a flat horizontal solid surface. The shape of the droplet is determined by the Young-Laplace equation (2.1), with the contact angle playing the role of a boundary condition as shown in Figure 2.4 below:

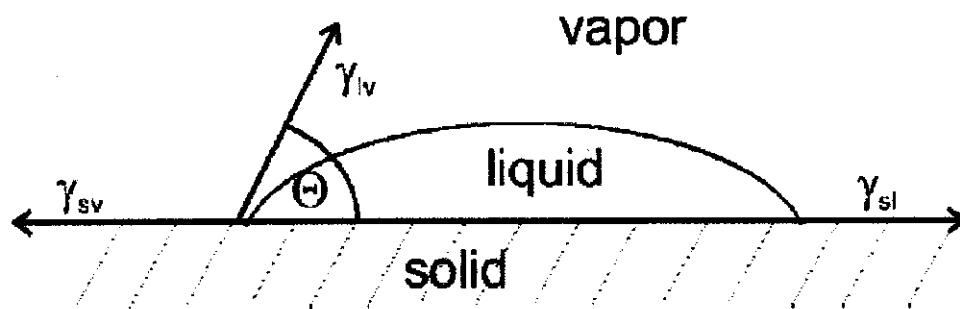


Figure 2.4: Wetting of a solid with a liquid (Teipel and Mikonsaari, 2004)

$$\gamma_{sl} = \gamma_{sv} - \gamma_{lv} \cdot \cos\theta \quad (2.1)$$

where  $\theta$  is the contact angle,  $\gamma_{sl}$  the surface tension (solid/liquid),  $\gamma_{sv}$  the solid surface tension (solid/vapour) and  $\gamma_{lv}$  the liquid surface tension (liquid/vapour) as shown in Figure 2.4.

The contact angle is not limited to a liquid/vapour interface; it is equally applicable to the interface of two liquids. Two procedures are available for studying contact angles for the three-phase contact line of a solid surface/water/air: a drop of liquid can be placed on a solid surface as shown in Figure 2.5 (left); alternatively, a submerged air bubble can be used to measure the contact angle of a solid immersed in water as shown in Figure 2.5 (right).

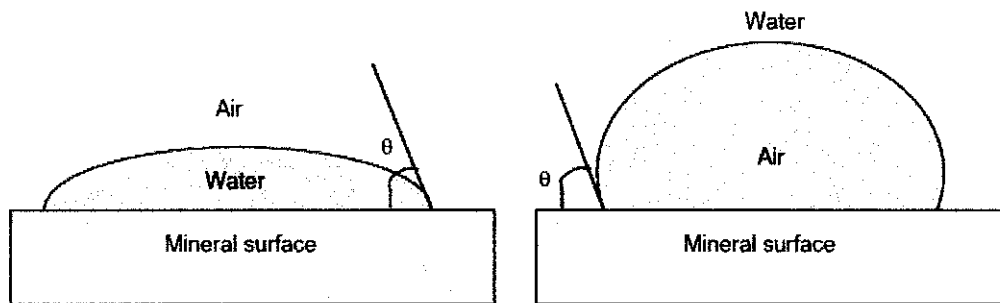


Figure 2.5: Contact angle ( $\theta$ ) formation of water droplet in air (left) and air bubble in water on a solid surface (right) (Chau, 2009)

In considering a liquid drop on a solid surface, there are two possible states of the solid. If the liquid is very strongly attracted to the solid surface (for example water on a strongly hydrophilic solid) the droplet will completely spread out on the solid surface and the contact angle will be close to  $0^\circ$ . Less strongly hydrophilic solids will have a contact angle up to  $90^\circ$ . On many highly hydrophilic surfaces, water droplets will exhibit contact angles of  $0^\circ$  to  $30^\circ$ . If the solid surface is hydrophobic, the contact angle will be larger than  $90^\circ$ .

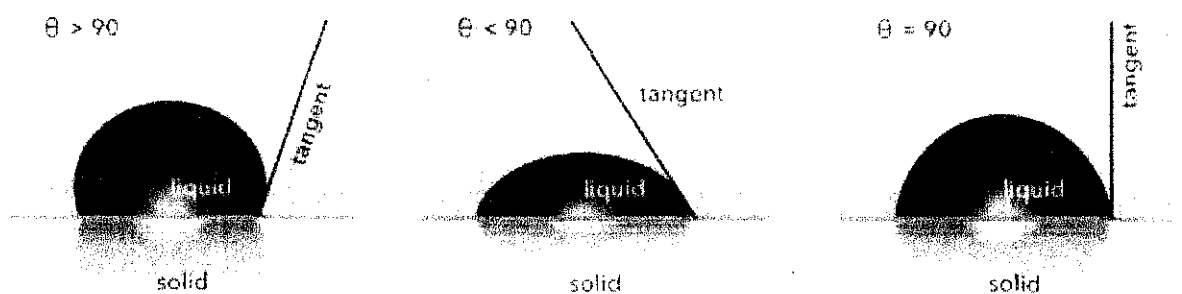


Figure 2.6: Different contact angles

It can be seen from Figure 2.6 that a low value of contact angle indicates that the liquid spreads, or wets well, while high contact angle value indicates poor wetting. If the angle is less than  $90^\circ$ , the liquid is said to wet the solid. If it is greater than  $90^\circ$ , it is said to be non-wetting. In the case of zero contact angle, this represents a complete wetting condition.

According to Eratak (2005), contact angle measurements on finely divided solids are much more difficult than those on moderately large, uniform solid surfaces, but the former is often more desired and more important since many industrial applications involve processing particulate solids.

The methods which are most frequently used for the solid surface free energy determination rely on wetting contact angle measurements. However, interpretation of the measured values is still debatable, especially for powdered solids (Chibowski and Perea-Carpio, 2002). In the case of powdered minerals, it can be said that there are two main groups of techniques for contact angle measurements; one group is applicable to flat and smooth surfaces while the other one has been especially developed for non-ideal surfaces or particles (Chau, 2009).

The resulting contact angles for these two types of surfaces will be very different due to the condition of the surfaces. The “intrinsic contact angle” as shown in Figure 2.7 (left) is expected for an ideal solid surface. The usual optical methods for measuring contact angles will yield the “apparent contact angle”. This is the angle between the direction of the tangent to the “smoothed” solid surface and the direction of the tangent to the air-water interface as shown in Figure 2.7 (right). On smooth

solid surfaces, the apparent contact angle is identical with the actual contact angle. On real uneven surfaces, those two values may be very different (Chau, 2009).

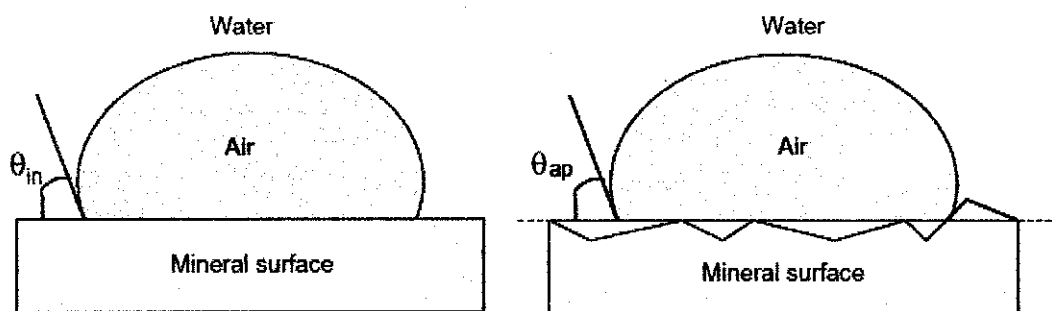


Figure 2.7: Differentiation between contact angle values: the intrinsic contact angle (left) and the apparent contact angle (right) (Chua, 2009)

In general, there are two methods currently in use to determine powder contact angles: (i) the Washburn equation (or dynamic) method which is based on the rate of liquid flow into a packed bed or porous plug of particulate solids; (ii) the Bartell (or static) method which is based on equilibrium measurements of the capillary pressure rise required to prevent liquid from penetration the packed bed (Adamson, 1967). The principles of the measurements are simple. However, both methods suffer some experimental and fundamental difficulties. One of these is that both methods in their simplest form require the visual observation of the wetting liquid from inside the porous bed. This may not be accurate in the case of irregularly shaped polydisperse particles or its exact position may not be clearly visible due to wall effects of the enclosing glassware (Eratak, 2005).

Modified versions to avoid difficulties of the two general measurement methods have been reported in the literature. Good et al., (1993) developed the thin layer wicking method to measure the rate of advance of wetting liquids through a thin layer of solid particles deposited onto a glass slide. This method uses the Washburn equation to determine the cosine of the contact angle and requires calibration tests with a perfectly wetting liquid to calculate the effective interstitial pore radius of the thin layer. Chibowski and Perea-Carpio (2001) developed a technique involving the measurement of the weight of liquid penetrating into a powder bed, instead of monitoring the movement of the liquid front, for the determination of the solid

surface free-energy components, but did not propose to derive the contact angle from such data.

The powder contact angle device of Dunstan and White (1986) and that of Diggins et al. (1990) both used the Bartell concept; however, rather than applying an external pressure difference to prevent capillary rise, the penetrating liquid was allowed to rise causing a gradual increase in the pressure of air enclosed above the wetting front. The capillary pressure was calculated by measuring the air pressure to stop the rise of liquid up the packed bed and subtracting any hydrostatic head, if present.

Teipel and Mikonsaari (2004) stated that standard experiments like direct contact angle observations cannot be used when dealing with finely dispersed solid materials. Usual investigation methods in that case are inverse gas chromatography, the sessile drop method and the capillary penetration technique. In their study, the capillary penetration method is investigated, in particular with the sampling procedure and sample preparation.

### **2.2.1 Direct measurements of contact angle**

It is observed that in most cases when a liquid is placed on a solid surface, it will not wet but remained as a drop having specific particular angle of contact between the liquid and solid phases (Adamson, 1967). The direct measurement of the contact angle can be applicable for large sample of solids. The tilting plate method has given the most reproducible and probably the most accurate contact angle values. A several centimetre wide plate of the solid dips into the liquid, and its position is altered by means of an adjustable mount until the angle such that the liquid surface appears to remain perfectly flat right up to the surface of the solid (Adamson, 1967).

The other technique for measuring the contact angles directly is the sessile drop method. Sessile drop technique is a widely used for measuring the direct contact angle. For this measurement the surface of the solid must be smooth and clean before the solid is dipped into the liquid and on the surface a bubble is formed, the angle between sessile drop and solid can be read from goniometer.

### 2.2.2 Column wicking method

The contact angle of fine particles can also be measured by using column wicking method. This method is based on the penetration of liquid into the porous structure by measuring the change of surface energy. Powdered solids are packed into a capillary tube in this technique and it is then immersed in a liquid of known surface tension. The rise of liquid into the powdered solids can then be observed. The contact angle can be calculated from the height of the liquid as a function of penetration time based on the Poiseuille's law.

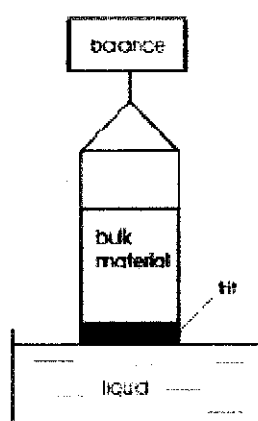


Figure 2.8: Schematic diagram of the capillary penetration method

### 2.2.3 Thin layer wicking method

Thin layer technique is based on the phenomena of a liquid penetration (wicking) into a solid porous layer deposited on a glass plate, e.g. microscope slide. The surface free energy components are then calculated from the proper form of the Washburn equation (Teixeira et al., 1998).

For the thin layer wicking method, the powdered solid is deposited on a microscopic slide in the form of aqueous slurry before the sample is dried and one side of the slide is immersed in a liquid in the vertical position and the liquid penetrates into the solid slowly. This thin layer wicking method also uses the Washburn equation.



However, the methods which are based on Washburn equation give only advancing contact angles rather than equilibrium contact angles.

Another method to measure the contact angle is by compressing the powders into pellets. This method is not recommended, however, as the surface properties such as surface roughness, liquid adsorption and porosity can change in the compressing phase and so the measurement of contact angle using pellets will be made mostly on assumed values. This will not results in accurate contact angle values.

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 Research Process**

The research process starts with stating the hypothesis after choosing the problem. Then, the author has to formulate the research design which is mainly related to laboratory experiment in the synthesis period. After the synthesis period, the product will proceed to be characterized under specific parameters. After that, available data and information are collected from reliable sources, followed by analysis and interpretation of result obtained from the characterization.

#### **3.2 Project Activities**

##### **3.2.1 Literature Review**

Literature review is a formal survey of professional literature and review theories and research already done on the topic. A thorough literature search which covers the background studies of contact angle measurement method and agricultural materials is conducted in order to provide a strong basis for the experimental work of the project.

##### **3.2.2 Materials for Experiment**

The agricultural materials to be used in this research experiment need to be grinded to small particle size due to their uneven and non-uniform physical structure (Constanzo et al., 1995).

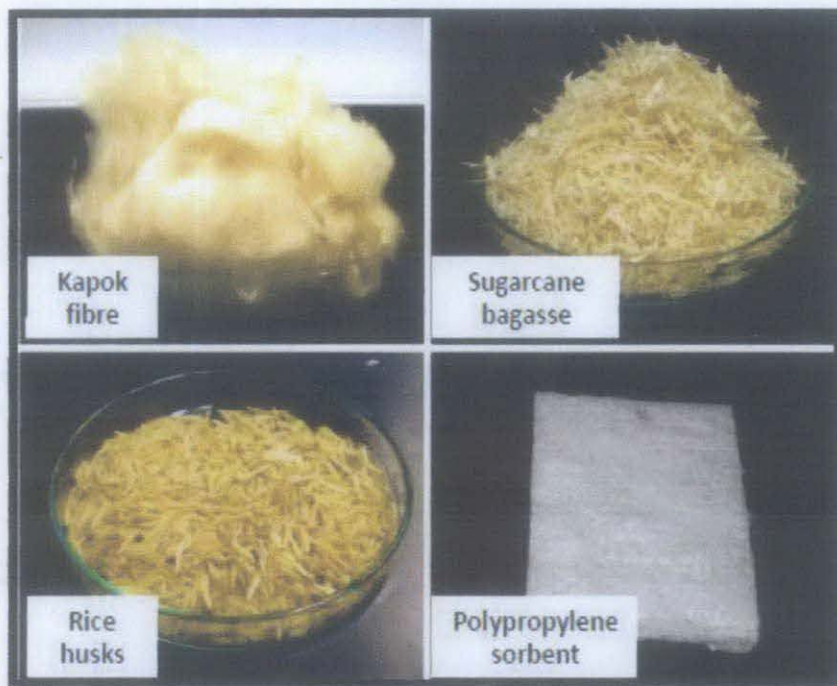


Figure 3.1: Materials used

These solid materials will be tested with many different liquid which are crude oil, engine oil, used engine oil and also diesel.



Figure 3.2: Different types of oil for test

### 3.3 Process Flow

The flow for the experimental procedure of the project will be conducted as the following:

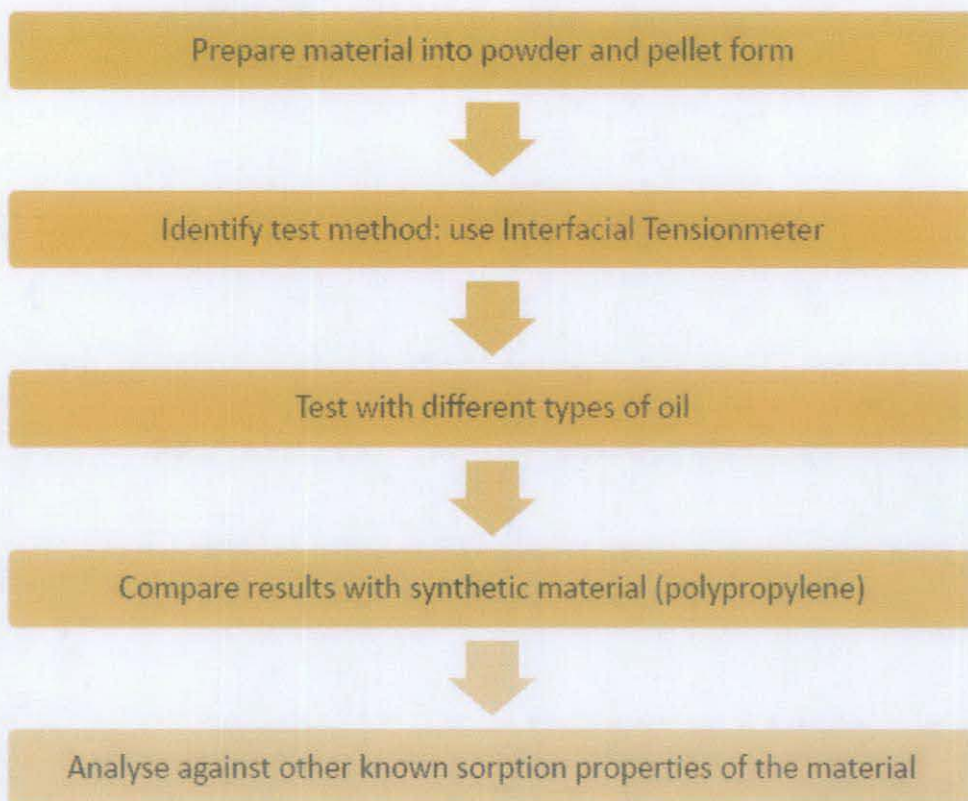


Figure 3.3: Process flow

### 3.3.1 Material Preparation

The materials would have to first be prepared in powder form of specific diameter ranging from  $63\mu\text{m}$  to  $125\mu\text{m}$ . This is done by using a grinder and the powder are then filtered using an electronic sieve according to its specific size which is needed.



Figure 3.4: Rice husks in different size



Figure 3.5: Sugarcane bagasse in different size



Figure 3.6: Kapok after being grinded

The powdered solids are then compressed into pellet forms to ensure that the surface of solid for liquid to be dropped is smooth and uniform. This is done by using manual compressor up to a pressure of 10,000 psi.



Figure 3.7: Apparatus used to fill in powder into metal casing



Figure 3.8: Filling in powder



Figure 3.9: Manual compressor

The resulting agricultural materials in pellet form are much easier to handle and so the experiment can be conducted more accurately.



Figure 3.10: Pellet form of agricultural materials

### **3.3.2 Experimental Procedure**

The chosen identified method to measure the contact angles of the agricultural materials is by dropping a liquid on to the solid pellet. The contact angle will be calculated using interfacial tensiometer- Optical Contact Angle Measuring Device: OCAH 200.

1. The pellet is first placed on a clean glass plate to ensure that it is free from contamination.
2. Oil is filled into syringe with a volume of 1 ml.
3. The syringe with precision tips of internal diameter 1.54 mm is placed at its holder at the equipment.
4. Ensure that the camera alignment is correct in order to capture the best image of the liquid drop on the solid.
5. The equipment is connected to a computer. Ensure that the software is running.
6. Drop the oil from the syringe by using the dispense unit in the software. Alternatively, this can be done manually using the hand.
7. Once the liquid drop on to the solid surface, capture the image.
8. Run the calculation for contact angle measurement.
9. Record the value of contact angle obtained.

This procedure is then repeated for different types of oil and also with different types of solid including polypropylene. The results can then be compared between the agricultural materials and the synthetic sorbent material – polypropylene.



Figure 3.11: Optical Contact Angle Measuring Device: OCAH 200

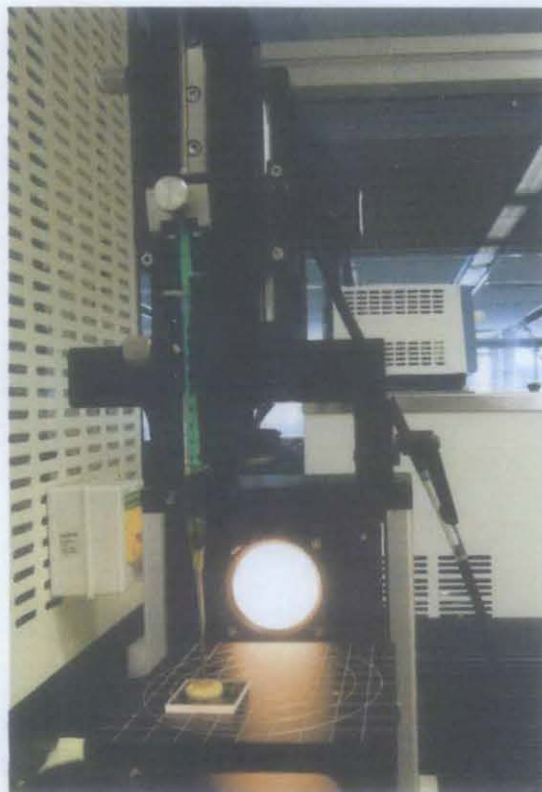


Figure 3.12: Equipment setup



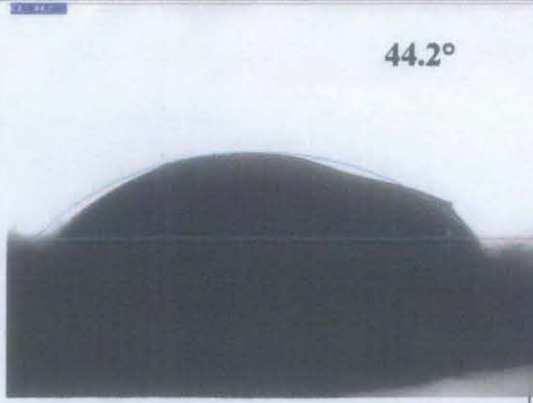


## CHAPTER 4







### RESULTS AND DISCUSSION

#### 4.1 Results

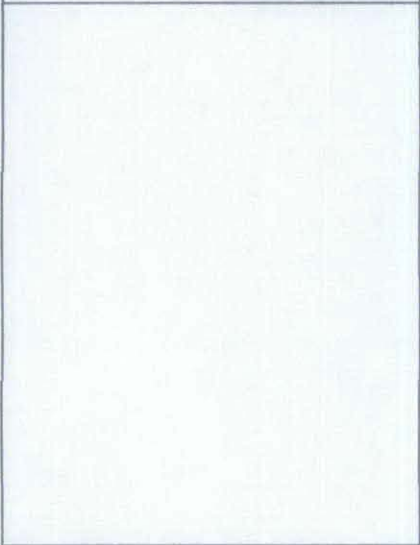
The values of contact angle obtained are based on the image captured of the liquid drop on the solid surface. In some cases where the absorption rate is too fast, the absorption will be recorded and a snapshot of the frame when the liquid first drop on to the solid pellet is taken as the result.

Table 4.1: Results of contact angles for different oil

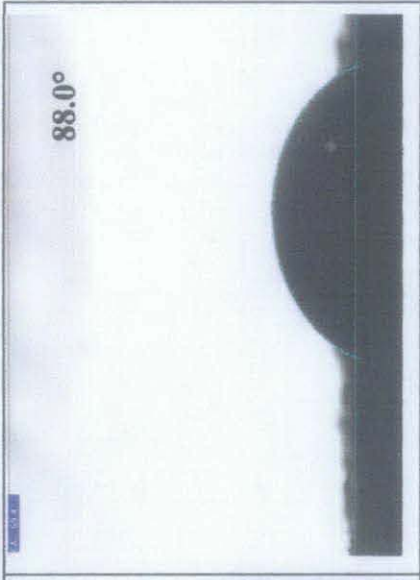
Materials	Crude Oil	Used Engine Oil	New Engine Oil
Kapok	 44.2°	 77.2°	 34.1°

<p>Sugarcane Bagasse</p>	<p>79.0°</p> 	<p>92.6°</p> 	<p>55.5°</p> 
<p>Rice Husk</p>	<p>70.7°</p> 	<p>80.3°</p> 	<p>69.4°</p> 

Polypropylene



93.0°



88.0°

The values of contact angles are presented in the following graphs Figure 4.1 to Figure 4.2.

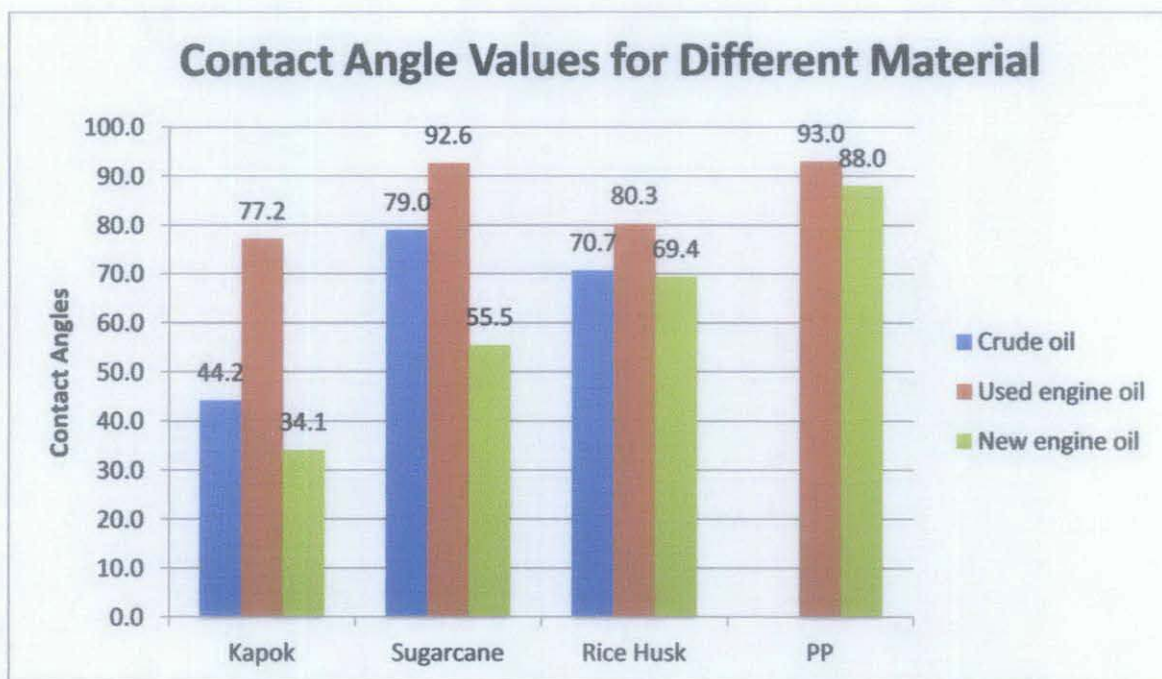


Figure 4.1: Contact Angle Values for Different Materials

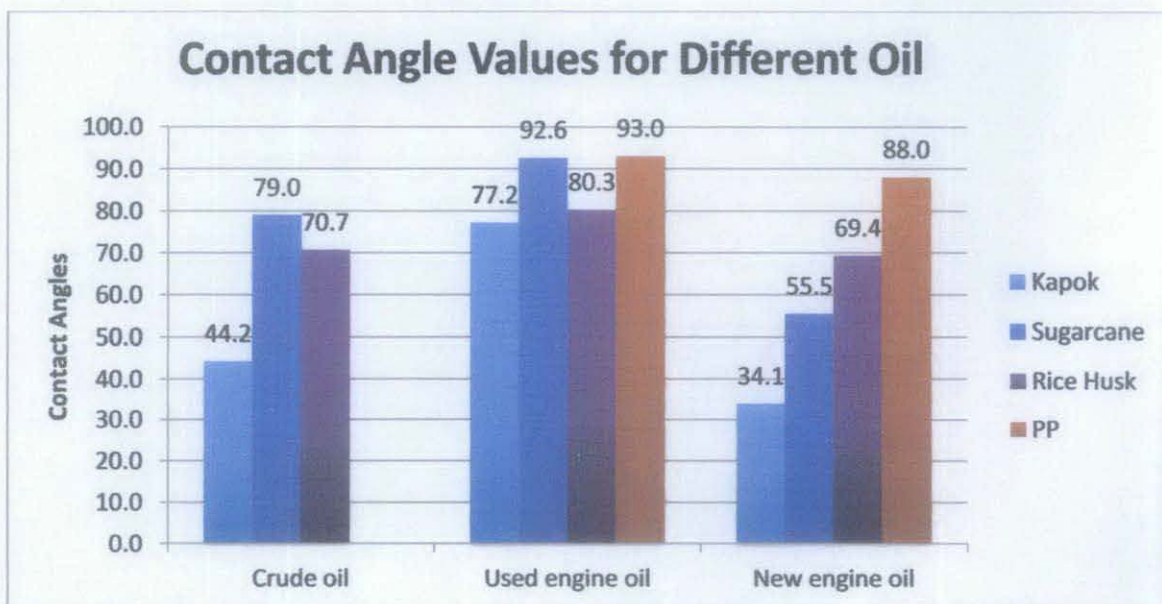


Figure 4.2: Contact Angle Values for Different Oil

The values for diesel cannot be measured because the density for diesel is the lightest out of all the oil (refer Table 4.2) which means that it is easily absorbed by the solid

pellet. The absorption rate is too fast that the liquid droplet on the solid surface cannot be captured by the camera of the interfacial tensiometer.

Table 4.2: Properties of liquid at 25°C (Ali et al., 2010)

<b>Liquid</b>	<b>Density at 25°C (g/cm<sup>3</sup>)</b>	<b>Liquid air surface tension at 25°C (MN/m)</b>	<b>Oil water surface tension at 25°C (MN/m)</b>
Water	1.00	68.50	-
Diesel oil	0.83	26.77	18.85
Crude oil	0.84	27.72	15.35
New engine oil	0.85	28.25	17.60
Used engine oil	0.93	27.35	7.52

## 4.2 Discussions

From Figure 4.1, it can be seen that generally kapok has the lowest contact angles value for all types of oil. Polypropylene, on the other hand, resulted in the highest contact angle values for every type of oil. This means that the sorption capacities of the agricultural materials are much better than the synthetic materials. This is also in line with study on sorption capacities done by Ali et al. (2010) as shown in Table 4.3 below:

Table 4.3: Oil sorption capacities of biomass (Ali et al., 2010)

<b>Sorbent</b>	<b>Type of oil</b>	<b>Sorption capacity (g/g)</b>
Kapok fibre	Diesel oil	19.35
Sugarcane bagasse	Diesel oil	10.51
Rice husks	Diesel oil	2.60

The values from Table 4.3 also give support that the values of contact angle for diesel would also follow the same pattern from the smallest value to the highest value: kapok < sugarcane bagasse < rice husk < polypropylene.

However, there are some conflicting values of contact angles for sugarcane bagasse and rice husks as shown in Figure 4.2. For crude oil and used engine oil, the values of contact angle for sugarcane bagasse are supposed to be lower than that of rice husk as proven in other literatures. The plausible explanation for this result is that there might be some pores between the particles of rice husks in the pellet. This would allow the test liquids to penetrate the pores, invariably resulting in unstable and unreliable contact angles (Constanzo et al., 1995).

Looking at the structure of the materials from SEM images at 20  $\mu\text{m}$  magnification, it is apparent that the characteristic of the material could also be a contributing factor for this result.

(a)



(b)



(c)

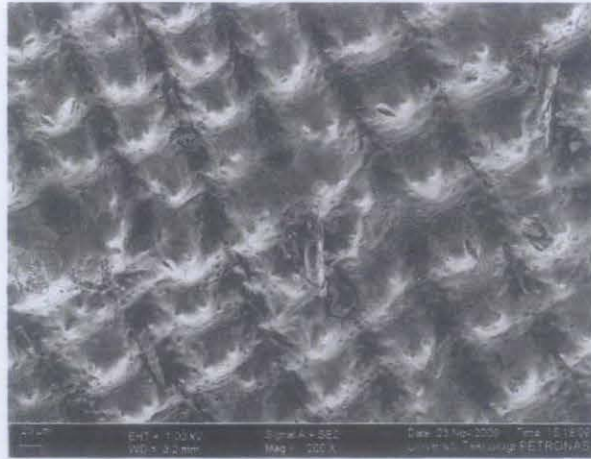


Figure 4.3: Micrographs of (a) kapok fibre, (b) sugarcane bagasse and (c) rice husks (Ali et al., 2010)

The waxy and spaced-out particles of kapok fibres mean that liquid samples are able to penetrate faster into the material. This means that the sorption rate of oil into kapok will be much faster than the other two materials. Sugarcane bagasse surface is also suitable for absorption as compared to that of rice husks as rice husks is made up of silica (Si) as part of its elemental composition. Kapok fibre and sugarcane bagasse on the other hand is not made out of Si which means that they are able to absorb liquid better than rice husks (Ali et al., 2010).

From the findings, it is apparent that the use of agricultural materials to replace synthetic materials as oil sorbent is a more promising alternative (refer Appendix for suitability matrix for usage of agricultural materials for oil removal).

## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

#### **5.1 Conclusion**

The contact angles for some natural materials are currently unknown although this is one of the most important properties which are needed in order to analyse the effectiveness of the materials as a sorbent in water. This is because there is no specific method currently available specialised in measuring the contact angles of such materials. The physical characteristics of these materials vary between one material to another which makes it even more difficult to identify a standard and specific method for contact angle measurement.

From this project, the contact angle of such agricultural materials can be determined through a specific sessile drop method by dropping a liquid sample on to a solid pellet made from agricultural materials powdered solid. By identifying their contact angles, the suitability of the natural materials as sorbent can then be further evaluated along with their other characteristics and properties.

The study has achieved its objectives in measuring the contact angles for agricultural materials with different types of oil sorbent using the best identified liquid penetration method. From the values obtained, it can be observed that out of the three agricultural materials kapok exhibit the best contact angle value for oil sorption. In comparing the results of contact angle for agricultural materials with polypropylene, it can also be concluded that these agricultural materials can be considered as a promising alternative for the synthetic sorbent materials currently being used.



## **5.2 Recommendations and Future Work**

The recommendations and future work for the study include the following aspect:

### **1. Material preparation**

The pellet form preparation of the powdered solids must be done in a clean environment to ensure that there is no contamination from the equipment used to prepare the pellets. The contamination could largely affect the results of contact angle measurement obtained.

### **2. Experiment procedure**

Capillary penetration method could be utilised in order to observe the effect of liquid penetration into powdered solids packed into a capillary tube. This might give support to the current results already obtained.

### **3. Available resources**

With the conclusion that kapok is the best suited material to replace the synthetic material currently in use, it is necessary to analyse on the availability of kapok trees in the country and whether the numbers are enough to meet future demands. Another alternative would be to synthesise material which is similar in properties to kapok fibre which could provide enough supply to meet the demand should kapok fibre become a viable alternative for the hazardous material currently being used as oil sorbent.

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## APPENDICES

Table A: Elemental composition and average density of kapok fibre, sugarcane bagasse and rice husks (Ali et al., 2010)

	<b>C (%)</b>	<b>O (%)</b>	<b>Si (%)</b>	<b>Average Density (g/cm<sup>3</sup>)</b>
<b>Kapok fibre</b>	53.79	46.21	-	0.7358
<b>Sugarcane bagasse</b>	50.92	49.08	-	0.9866
<b>Rice husks</b>	24.01	51.03	24.94	1.1575

Table B: Suitability matrix for usage of kapok fibre, sugarcane bagasse, rice husks and synthetic sorbent for oil removal (Ali et al., 2010)

	<b>Kapok fibre</b>	<b>Sugarcane bagasse</b>	<b>Rice husks</b>	<b>Synthetic sorbent</b>
Removal of diesel or crude oil from oil-water mixture	<ul style="list-style-type: none"> <li>Highly recommended</li> </ul>	<ul style="list-style-type: none"> <li>Moderately suitable</li> <li>Reduced capacity due to water sorption</li> </ul>	<ul style="list-style-type: none"> <li>Not suitable</li> <li>O/W ratio &lt; 1</li> </ul>	<ul style="list-style-type: none"> <li>Not suitable</li> <li>O/W ratio &lt; 1</li> </ul>
Removal of engine oil from oil-water mixture	<ul style="list-style-type: none"> <li>Highly recommended</li> </ul>	<ul style="list-style-type: none"> <li>Moderately suitable</li> <li>Reduced capacity due to water sorption</li> </ul>	<ul style="list-style-type: none"> <li>Moderately suitable</li> <li>Reduced capacity due to water sorption</li> </ul>	<ul style="list-style-type: none"> <li>Moderately suitable</li> <li>Reduced capacity due to water sorption</li> </ul>
Removal of diesel or crude oil from clearly stratified oil/water layers	<ul style="list-style-type: none"> <li>Highly recommended</li> </ul>	<ul style="list-style-type: none"> <li>Suitable for relatively stagnant oil layer</li> </ul>	<ul style="list-style-type: none"> <li>Not suitable</li> <li>Diesel/crude oil sorption capacity &lt; 5 g/g</li> </ul>	<ul style="list-style-type: none"> <li>Suitable for relatively stagnant oil layer</li> </ul>
Removal of engine oil from clearly stratified oil/water layers	<ul style="list-style-type: none"> <li>Highly recommended</li> </ul>	<ul style="list-style-type: none"> <li>Very suitable for relatively stagnant oil layer.</li> <li>O/W ratio &gt; 2</li> <li>Engine oil sorption capacity &gt; 10 g/g</li> </ul>	<ul style="list-style-type: none"> <li>Suitable for relatively stagnant oil layer</li> </ul>	<ul style="list-style-type: none"> <li>Suitable for relatively stagnant oil layer</li> </ul>