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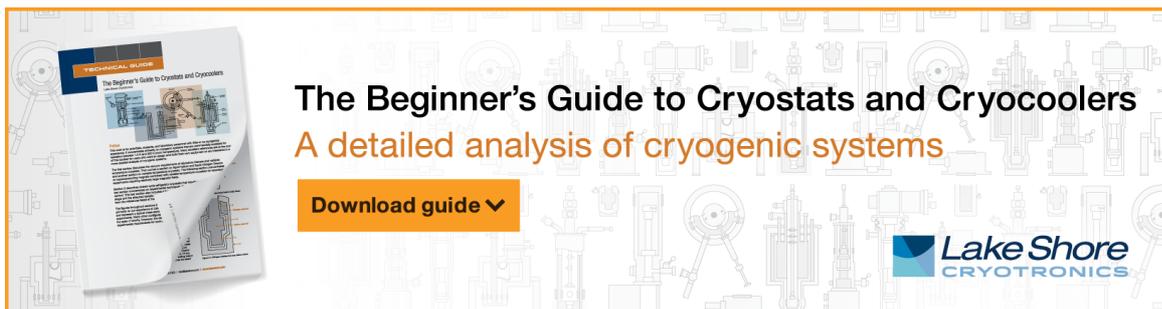


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The Oil Sorption Behaviour Investigation of Kapok (Ceiba Pentandra (L.)) Fiber

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Abstract. As oil exploration and production activities have risen globally, water contamination from oil spills and the discharge of other oily wastewaters has emerged as one of the primary environmental concerns. Thus, Kapok fiber is considered in this study as it is known as one of the most effective method for cleaning up and collecting oil spills where Kapok is a natural cellulosic fiber with unique characteristics. A critical investigation was conducted to study the potential of kapok fiber as sorbent material, also analyze the surface properties of kapok fiber for the ability of kapok fiber to absorb oil and investigate the sorption mechanisms of kapok fiber. Therefore, the surface properties of kapok fiber were analyzed using SEM, FTIR, TGA and contact angle. To investigate the selectivity nature and the sorption capacity of 5 g kapok fiber, different types of oil and different apparent viscosity were used. The types of oil used are gear oil (low viscosity), vegetable oil-based cooking oil (medium viscosity) and waste oil (high viscosity). Kapok fiber was able to absorb all types of oil, with waste oil absorbing the most about 17.88 g.g⁻¹. Scanning electron microscopy (SEM) was used to examine the morphology of raw kapok fiber. In this study, kapok fiber was shown to have a porous hollow lumen structure and a waxy coating on the surface. Other than that, for the contact angle analysis, kapok fiber had high water contact angle up to 130°. The water droplet was stood on the kapok fibers surfaces before and after absorption with contact angles ranging from 130° to 145°. In contrast, the oil droplet had disappeared from the surfaces of kapok fiber within a few seconds.

INTRODUCTION

Over the last few years, intentional and accidental oil discharges have rapidly increased during refining [1], transportation [2] and production [3]. As a result, there is a substantial detrimental impact on organisms and the ecological environment due to human activities or natural disasters, particularly in a marine zone [4]. It has the potential to get a massive impact on the environment and the economy if this happened continuously and may create a few long-term problems [5]. Therefore, it is important to develop or design a relatively efficient method to stop the potential oil pollution hazard and recover the spilt oil simultaneously.

To prevent the potential oil pollution hazard, several methods for recovering or eliminating oil from water can be used [6]. One technique that could be implemented is by using sorbent material [7]. Surprisingly, it is considered the most effective method for collecting and cleaning up the spilt oil in the water [8]. Sorbent material can be divided into three types which are synthetic organic polymer [9], inorganic mineral material [10] and organic natural material [11]. Generally, organic natural material involves many agricultural products available to absorb oil [7]. For example, straw [12], kapok fiber [13], cotton fiber [14] and sawdust [15]. However, the best that can show good oil sorption among those materials is kapok fiber [16].

Generally, kapok fiber is a type of natural plant fiber which produce by the fruits of the silk-cotton tree [17]. Based on the kapok fiber's characteristics, kapok fiber has received a lot of attention as an oil absorptive material in the recent year [18]. It has low density, huge hollowness and good buoyancy [19-21]. It also has high sorption capacity and biodegradability [22]. Furthermore, due to a small amount of waxy coating also covering the fiber surface, it becomes much more hydrophobic [23]. Therefore, kapok fiber has a higher oil sorption ability than other natural materials because of these features and characteristics [24].

This study was focused on kapok fiber to investigate the oil sorption behaviour of kapok fiber. However, even kapok fiber can be an alternative way of oil pollution control, the surface properties, interactions and sorption mechanisms of kapok fiber still cannot be well-identified. Therefore, the raw kapok fiber was immersed with different kinds of oil in the current project. At the same time, the raw kapok fiber is also analyzed by some standard methods to perform the test such as Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and Thermogravimetric Analysis (TGA) and Contact Angle.

MATERIAL AND METHODS

Material

The kapok used in this research was a product of Malaysia. All dust and lumps were removed from the kapok fibers before it was used in absorption tests. Other than that, no further treatment and modification were performed on the kapok fiber to ensure its freshness.

Three types of oils, namely used cooking oil, used gear oil and wasted oil, were used in the study to investigate the oil sorption characteristic of kapok fiber. The three types of oil were chosen to represent different viscosity which were used gear oil for low viscosity oil, used cooking oil for intermediate viscosity, and wasted oil for high viscosity.

Oil Absorption Experiment

The experiment starts with 1 L of oil and 5 L of water were poured into a white container. After the water and oil achieved a steady condition, 12 samples of kapok fiber were immersed in the solution for 30 minutes to ensure that the sorption reached equilibrium. Each sample of kapok fiber was 5 g. All samples of kapok fiber were flipped to ensure all the surfaces of kapok fiber were exposed to the oil in the container in the first 15 minutes. After 30 minutes, all the samples of kapok fiber were taken out and drained for 15 minutes. Then the weight of each sample were taken. This method was repeated three times with different oil, respectively.

Oil Sorption Capacity Measurement

To measure the oil sorption capacity, each sample was weighed before and after sorption. Besides, all oil sorption capacity experiments were repeated three times to obtain an average value of weight. The sorption was calculated by the following formula:

$$\text{Oil sorption capacity} = (m_1 - m_0) / m_0 \quad (1)$$

The oil sorption capacity was calculated as grams of oil per gram of sample. For m_1 is the final weight (g) of the kapok fiber and m_0 is the initial weight (g) of the kapok fiber. In addition, the method of oil-absorbing measurement was followed in a research study by Wang (2018) whereby cotton fiber was used for oil-absorbing measurement [25].

CHARACTERIZATION OF KAPOK FIBER

Surface Morphology

The surface morphology of raw kapok fiber was evaluated by Scanning Electron Microscope (SEM) using JEOL JSM-6380LA. The sample was cut into small portions and coated with a platinum coating to improve electrical conductivity. Then the sample was transferred into the specimen stage and viewed under SEM operating with an accelerating voltage of 10 kV under low pressure at 8 Pa.

Thermal Behavior

Thermogravimetric Analysis (TGA) was used to determine the thermal behavior of kapok fiber before and after the absorption test using Linseis L81/1550 DTA-TGA. The kapok fiber samples were prepared in the form of a small lump. About 1.4 – 8.0 mg of kapok fiber was heated in the thermal gravimetric furnace from 30 °C to 700°C at the heating rate of 10°C/min under a nitrogen atmosphere.

Surface Properties

Contact Angle analysis was used to investigate the surface properties of kapok fiber using VCA Optima, AST Products, Inc. Tap water was used to determine the contact angles formed on the surface of kapok fiber. After dropping a single liquid randomly on the surface of kapok fiber, the probe liquids were allowed to equilibrate for 20 seconds before the image of the contact angles was acquired. The sample were then measured between 0° to 180° by using the same machine.

Element And Functional

Fourier Transform Infrared Spectroscopy (FTIR) was used to identify the existence of elements and functional groups in kapok fiber before and after the absorption test. All samples were analyzed using FTIR 100 series, Perkin –Elmer. Each spectrum was scanned 32 times in the range 4000 – 400 cm^{-1} wavelengths with a maximum resolution of 0.09 cm^{-1} in transmission mode.

RESULTS AND DISCUSSION

Oil sorption capacity

The oil absorbency of raw kapok fiber on used cooking oil, used gear oil and wasted oil was investigated to determine the oil sorption capacity of kapok fiber. Figure 1 shows the oil sorption capacity of each sample. As we can see, kapok fiber was effective as an oil sorbent and able to absorb oil due to the presence of a penetrable lumen network with inter-fiber pores in the kapok microstructure [25]. The presence of a lumen structure has been considered as a factor in oil storage after it has been obtained [16]. Figure 2 shows the condition of kapok fiber after the absorption experiment.

Based on the result, kapok fiber had the highest sorption capacity for waste oil and the lowest capacity for used gear oil. Meanwhile, used cooking oil has the second-highest sorption capacity. Within the same unit volume, wasted oil was heavier than used cooking oil and used gear oil, resulting in most sorption capacity of oils in this study. According to the research study from Kartina et al., the higher viscosity of the wasted oil was assumed to be the factor for the considerably huge sorption by the kapok fiber [26]. However, due to the wasted oil had a higher viscosity, it formed a thicker film layer coating on the wall of the flow channels. As a result, the effective size of the flow channels was reduced.

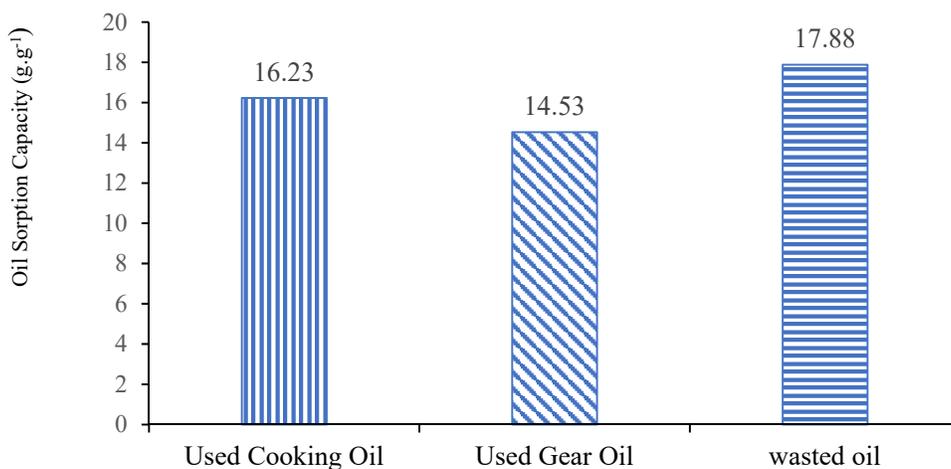


FIGURE 1: Oil sorption performance of absorption experiment.

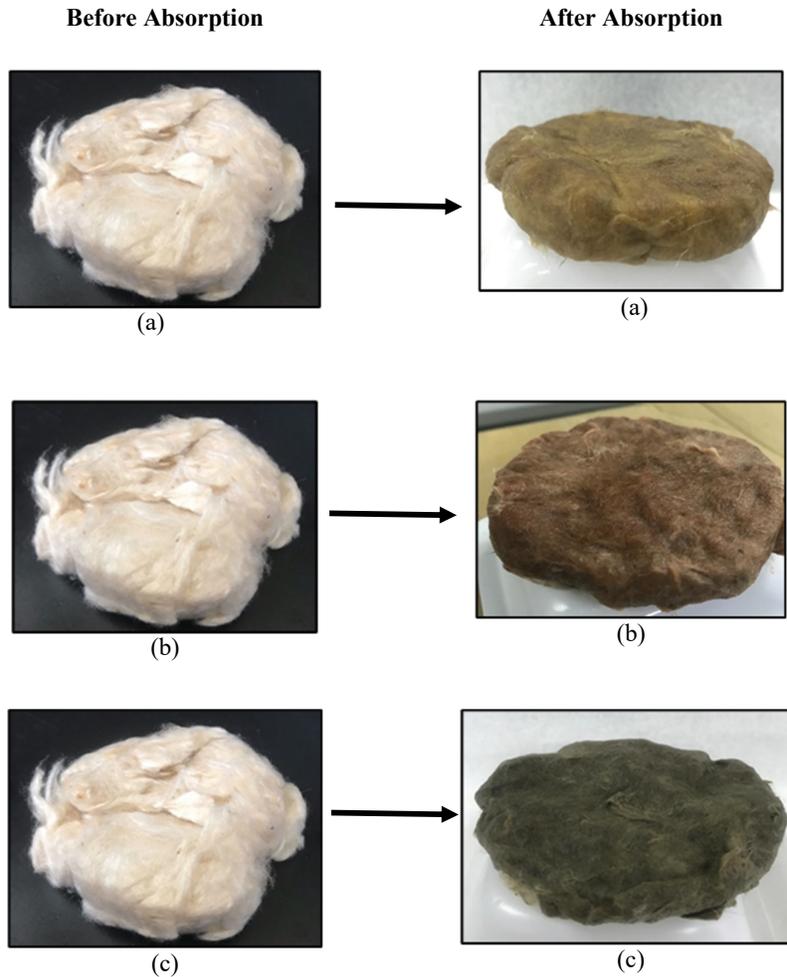


FIGURE 2: Condition of kapok fiber before and after absorption with different oil (a) cooking oil (b) gear oil (c) waste oil.

Surface Morphology

Raw kapok fiber morphologies were analyzed using Scanning Electron Microscopy (SEM). Figure 3 shows the SEM image of surface morphologies of raw kapok fiber. It can be seen that raw kapok fiber has a silky appearance and smooth surface due to the plant wax's covering. Therefore, this phenomenon was similar to the research study by Jamat et al. [27]. Figure 3 (f) shows that there was more wax on the kapok surface to make the kapok fiber smooth, non-absorbent from water, difficult to tangle and insect-resistant. As a result of this feature, kapok fiber becomes highly water resistant.

On top of that, the average diameter of raw kapok in this study is $27.4 \mu\text{m}$, slightly larger than the $17.59 \mu\text{m}$ [18]. Figure 3 (d) illustrates the kapok fiber has a spherical shape and smooth surface. It also has a hollow structure with a large lumen filled with air and a thin fiber wall. The physical look of raw kapok fiber in this study was similar to the research study from Purnawati et al. [18]. In addition, due to having unicellular cellulosic fibers, oval or cylindrical, long with pointed ends and thin cell walls with ample lumens, kapok fiber has low fiber density and high bulkiness, good oil absorptivity and water-repellent nature [19].

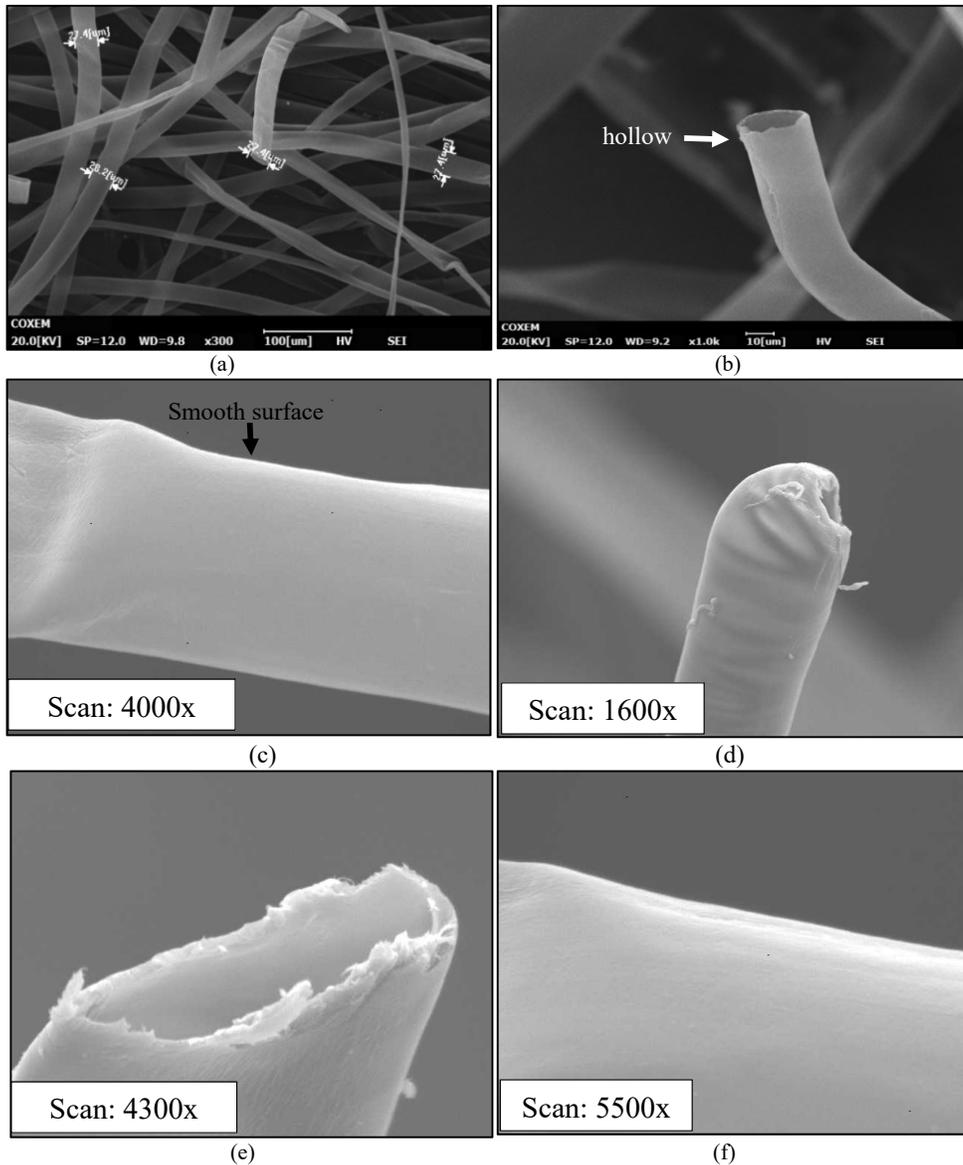


FIGURE 3: SEM image for kapok fiber (a) average diameter (b) large lumen and hollow structure (c) silky appearance and smooth surface (d) spherical shape (e) thin wall (f) wax surface.

Thermal Behavior

For the kapok fiber before the absorption test that was raw kapok fiber, the degradation temperature was identified to be 140.2 °C until 361.7 °C. The apparent peak at 250 °C to 300 °C was typically attributed to hemicellulose thermal degradation in an inert atmosphere [28]. Therefore, this behavior was quite similar to the result of this research, whereby hemicellulose degradation occurs at temperatures between 140.2 °C to 361.7 °C. Meanwhile, at this stage, cellulose, polysaccharides and hemicelluloses began to degrade and continued until they reached the decomposition temperature [29]. As we can see in Figure 4 the initial weight loss for raw kapok fiber occurs. As for the kapok fiber after absorption test, the initial weight loss for used cooking oil samples occurs between 50 °C and 150 °C. Both of these conditions occurred as a result of the sample's moisture content evaporating, [29].

However, the initial weight for used gear oil and wasted oil immediately increases. According to the research study from Xu et al., this phenomenon happened due to the viscosity of used gear oil and wasted oil was decreased when the temperature was raised [14].

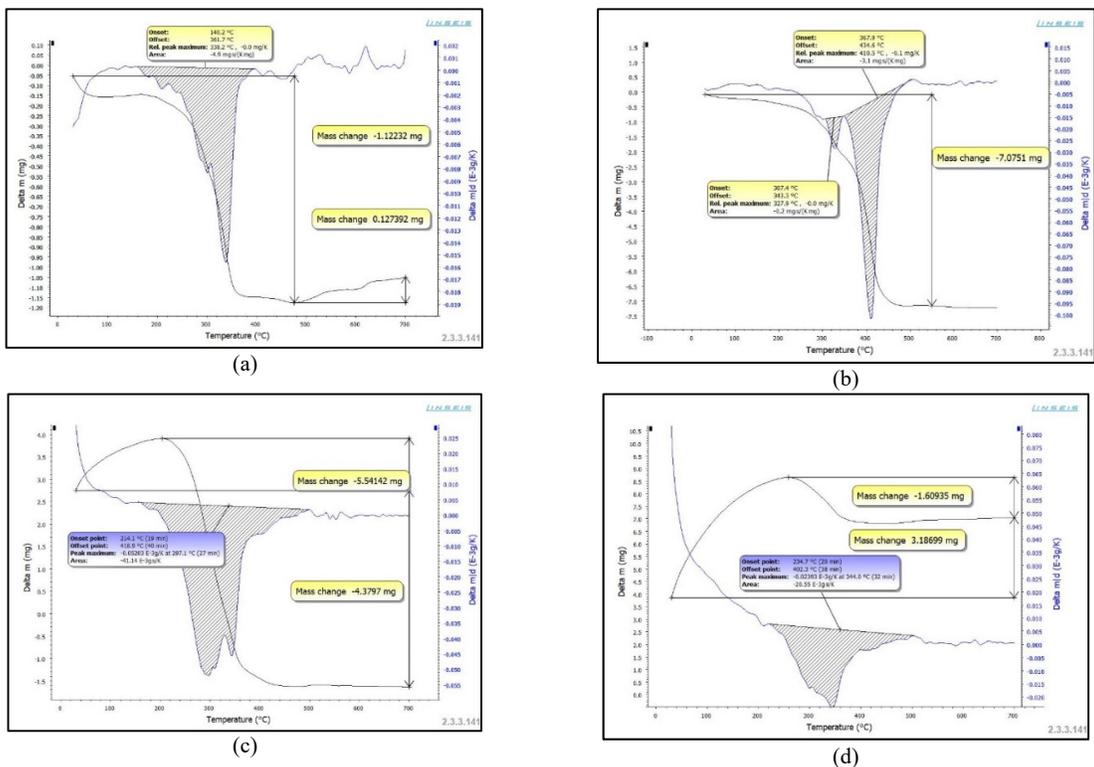


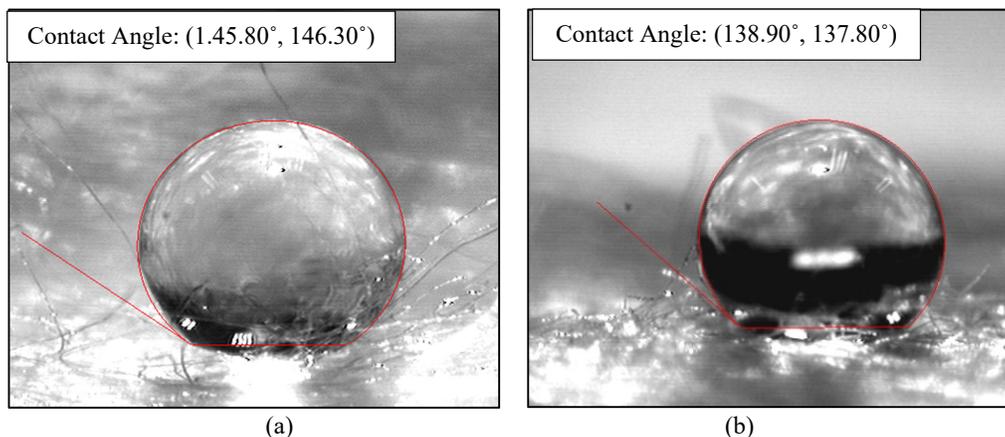
FIGURE 4: Thermal analysis of kapok fiber (a) raw kapok (b) used cooking oil (c) used gear oil (d) wasted oil.

Surface Properties

The contact angle was performed to evaluate the surface properties of kapok fiber. Figure 5(a-d) shows the contact angle measurement of raw kapok fiber and kapok fiber with three different oils. The surface contact angle of raw kapok fibers was 145.80° and kapok fibers with three different oils decreased to 124.90° for used cooking oil, 138.90° for used gear oil and 134.20° for wasted oil. This phenomenon was related to a study by Sun [30], whereby the kapok fibers became more hydrophilic due to the oil and wax layer of the kapok fibers interacting together [30].

In general, the water contact angles of kapok fiber ranged from 120° to 145°, which was reported in a research study [31]. In this research, the contact angle of raw kapok fiber was 145.80°. The result was more than enough to prove that kapok fiber is highly hydrophobic. The smooth wax coating was responsible for hydrophobicity [32]. Other than that, the high-water contact angles of each sample of kapok fiber demonstrated that it has excellent hydrophobic and oleophobic characteristics. As a result, the higher water contact angles of kapok fiber indicated their better water repellent abilities [31].

In addition, due to establishing an excellent contact angle (>90°) between water and kapok fiber, water is a non-wetting liquid for kapok fiber [16]. Therefore, the enormous lumen of kapok fiber is not accessible to water [33]. Hence, the contact angle of kapok fiber with water was constant as time flew.



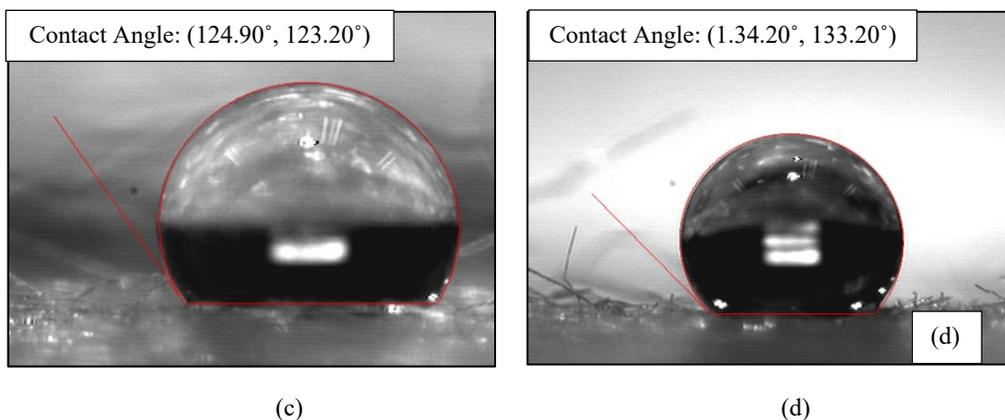


FIGURE 5: Contact angle of kapok fiber (a) raw kapok (b) used gear oil (c) used cooking oil (d) wasted oil.

Element And Functional Group

Figure 6 show FTIR results for all samples. The kapok fiber before the absorption test, which was raw kapok fiber, several major transmittances and primary absorption peaks, were identified. The broadest peak can be seen at 3342 cm^{-1} and it corresponded to the non-free O-H stretching vibration [22]. Other than that, the next well-pronounced peak was at 2918 cm^{-1} and was assigned to asymmetric and symmetric aliphatic CH_2 and CH_3 stretching vibration similar to the research study where, this was believed to be related to the presence of plant wax which generally consists of fatty acids, ketones, aldehydes, esters, long-chain alkanes and alcohols [16]. Therefore, the FTIR result for raw kapok fiber in this study indicate that the kapok fiber was lignocellulosic with a hydrophobic waxy coating.

Meanwhile, for the kapok fiber after absorption test, it show that the absorption bands at 3342 cm^{-1} for wasted oil and used cooking oil were increasing. This happened due to the breaking of hydrogen bonds, which increased cellulose hydroxyl groups in fiber walls [23]. Other than that, all the sample of kapok fiber after absorption test were increase in the characteristic absorbance peak at 2918 cm^{-1} . According to the research study from Lim, this phenomenon happened due to the kapok fiber had lost their smooth and waxy surface after absorption test [21].

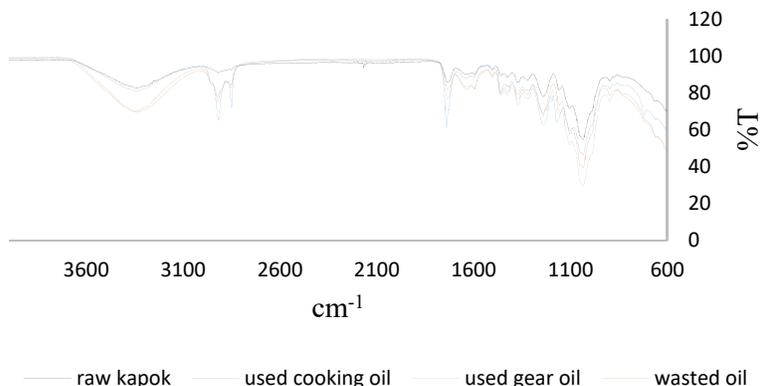


FIGURE 6: FTIR result for all samples.

CONCLUSION

As the conclusion, the kapok fiber has a unique characteristic and effectively as an oil sorbent material. Kapok fiber had the highest sorption capacity for waste oil and the lowest capacity for used gear oil. Meanwhile, used cooking oil has the second-highest sorption capacity. Based on a previous research paper review, the different viscosity of the oil was assumed the factor for the considerably different sorption capacity by the kapok fiber [34]. For the surface morphology, the average diameter of raw kapok in this study was $27.4\text{ }\mu\text{m}$, slightly larger than the $17.59\text{ }\mu\text{m}$ other than that, raw kapok fiber has a silky appearance and smooth surface due to the plant wax's covering. It also has more wax on the surface to make the kapok fiber non-absorbent of water, difficult to tangle and insect-resistant. As conclusion of this feature, kapok fiber becomes highly water resistant. For the thermal behavior of kapok fiber before the absorption test, the raw kapok fiber apparent peak at $250\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$ was typically attributed to hemicellulose thermal degradation and weight loss happened at this stage due to the evaporation of moisture content in the sample. However, the initial weight of kapok fiber after absorption that was wasted oil and used gear oil increased due to the viscosity of used gear oil and wasted oil was decreased when the temperature raised.

The surface contact angle of raw kapok fibers was 145.80° and kapok fibers with three different oil decreased to 124.90° for used cooking oil, 138.90° for used gear oil and 134.20° for wasted oil. This phenomenon happened due to the

kapok fibers becoming more hydrophilic due to the oil and wax layer of the kapok fibers interacting together. However, the water contact angles of each sample ranged from 120° to 145°. Therefore, it demonstrated that kapok fiber has excellent hydrophobic and oleophobic characteristics. For the element and functional group from FTIR analysis, the peak at 2918 cm⁻¹ for raw kapok fiber was assigned to asymmetric and symmetric aliphatic CH₂ and CH₃ stretching vibration. This was believed to be related to the presence of plant wax. However, for the kapok fiber after the absorption test, the absorbance peak at 2918 cm⁻¹ were increased. This happened due to the loss of a smooth and waxy surface after the absorption test.

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