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Characterization of Kapok (Ceiba pentandra (L.) Gaertn.) and Its Reusability in Oil Sorption

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ABSTRACT

Natural fiber such as kapok (Ceiba pentandra L. Gaertn.) is an effective, environmentally friendly, and sustainable material for oil sorption due to its unique structure, which offers high sorption ability, good selectivity, and reusability. Kapok fiber was characterized using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and contact angle. The oil sorption characteristics of kapok fiber were evaluated across different types of cooking oil. The result demonstrated that kapok possesses excellent hydrophobic-oleophilic properties, including a hollow lumen structure of kapok fibers that enables it to absorb and retain large amounts of oil while repelling water. Additionally, a waxy substance on its surface further enhances its hydrophobic properties. Kapok fiber also exhibits a contact angle of 123.80º > 90º, proving that kapok has low water wettability. The reusability of 1g kapok fiber in oil sorption is tested by using various types of cooking oil with the following standard test method for sorbent performance of adsorbents (ASTM F726-12) across five types of cooking oil. The results show that the kapok fiber retained the oil absorbed even after being reused for 24 cycles, despite slight decreases.

Keywords: *Kapok fiber, Natural fibers, Oil sorption, Oil sorbent, Reusability, Water pollution*

1. INTRODUCTION

The increased production of household and industrial waste oils is a global issue [1]. Cooking oil waste is a by-product of the kitchen, restaurants, food processing plants, and human and animal waste that harms people's health and pollutes the environment [2]. Untreated urban domestic wastewater contains a high concentration of organic matter, such as vegetable and animal oils in the kitchen and human waste, resulting in a major municipal source of oil containing up to 36% of the oily substances that contribute to water pollution [3,4]. This oily substance in emulsion form is created when the oil comes into contact with the water [5]. Emulsions are droplet suspensions greater than 0.1 μm of two completely immiscible liquids, and one dispersed throughout the other [6]. Many methods have been used to clean up oil from polluted areas, including mechanical extraction, in-situ combustion [7], and chemical degradation. Due to the greater economic and environmental benefits, using natural oil sorbent is an effective method of concentrating, transferring, and absorbing oil [8].

Table 1 Comparison of kapok fiber reusability in oil sorption

Material	Modification	Oil used	Reusability		Reference
			No. cycle	Efficiency	
Kapok fiber, Waste cotton fiber	Kapok/cotton nonwoven using carding and needle-punching techniques	Engine oil, vegetable oil, and diesel oil	10	More than 80 % of the initial sorption capacity remaining	[9]
Kapok fiber	-	Diesel oil, new engine oil and used engine oil	15	Only 30% of sorption capacity reduction.	[10]
Kapok fiber	Kapok-derived cellulose nanofibrils foams (KNFs)	Vegetable oil and mineral oil	50	The cumulative oil release percentage declined by 11.8%	[11]
Kapok fiber	Kapok/ Microfibrillated cellulose aerogels (KCAs) modified by Vinyltrimethoxysilane (VTMO)	Vegetable oil, Gasoline oil, Ethanol, Vacuum pump oil and motor oil	10	Absorption capacity decreased by only 12.55%	[12]

Kapok fiber, a silky lignocellulose fiber with a microtubular structure (*Ceiba pentandra L. Gaertn*.), is a favourable biomaterial for adsorption [13]. Their unique features, such as those in kapok fiber-based materials, have opened up possibilities for various new applications [14]. Kapok fiber is a highly effective and environmentally friendly material for use as an oil sorbent. Its natural water resistance, known as the hydrophobic-oleophilic nature of the kapok fiber assembly, gives it a distinct oil retention property [15], high oil absorbency [16], biodegradable and reusable [17] make it an ideal choice for oil spill clean-up operations in marine and aquatic environments. **Table 1** compares the reusability of kapok fiber in oil sorption as reported by different researchers.

The overall sorption qualities of kapok suggest that Malaysian kapok is a suitable lignocellulosic material for use as an oil sorbent, with high oil sorption and retention characteristics that remain stable over extended periods. This stability is demonstrated in a study [10], where only 30% of the sorption capacity has been reduced after 15 reuse cycles. However, the oil retention capacity after the sorption/desorption cycle of kapok fiber can also be increased by various modifications, where the oil retention capacity of modified kapok is slightly higher than that of the natural kapok fiber [9–12].

In this work, the reusability of kapok fiber was tested with five different types of cooking oil following the standard test method for sorbent performance of adsorbents (ASTM F726-12) for 24 cycles—the total amount of oil the adsorbent can hold after each saturation cycle was evaluated. The contact angle, SEM, and FTIR analysis were also used to characterize the kapok fiber.

2. MATERIAL AND METHODS

2.1. Material

In this study, raw kapok fibers have been utilized as the main material. Kapok fiber had been purchased from suppliers Mutiara Zulfikar Sdn. Bhd. Resided at Kulim, Kedah. The oils used for the reusability test were corn oil (Brand: *Daisy*), palm oil (Brand: *Alif*), coconut oil (Brand: *Akasa*), blend oil (Brand: *Knife*), and used cooking oil. The corn oil (CNO) and Blend oil (BO) (consisting of refined palm olein, peanut oil, and sesame oil) were produced by Lam Soon Edible Oils Sdn. Bhd., palm oil (PO), a product from Sime Darby Plantation Berhad, and coconut oil (COO), a product from Kara Marketing Sdn. Bhd. The used cooking oil (UO) was collected from the Barracuda café at the UTHM Pagoh residential college.

2.2. Characterization of Kapok Fiber

The phase and structure analysis was done using Fourier-transform infrared spectroscopy (FTIR)using Fourier-transform infrared spectroscopy (FTIR) to identify the functional groups present at the kapok fiber surface. Kapok fibre morphology was characterized using a Scanning Electron Microscope (SEM). The surface interaction between the water and kapok fiber was tested using contact angle measurement.

2.2.1. FTIR

Fourier Transform Infrared Spectroscopy, FTIR (*Agilent Technologies*), was used to analyze the infrared absorption spectrum of kapok fiber. The test samples for FTIR analysis were prepared by cutting a piece of kapok fiber (loose form) and using it as a sample. The sample was placed inside the FTIR spectrometer and bombarded with infrared beams for qualitative analyses. The sample, however, must be thin enough to allow the infrared beam to pass through. At a resolution of 8 cm^{-1}, two different scans were performed at wavelengths ranging from 4000 cm^{-1} to 650 cm^{-1}.

2.2.2. SEM

The morphology of the kapok was analyzed using scanning electron microscopy (SEM) (*Oxford Instruments*). For SEM analysis, the samples were adhered to a rectangular stainless-steel sample holder with conductive double-sided sticky tape. The samples were sputter-coated with gold using a rotary pumped coater (*Quorum Q150R S*) to create a conductive coating that improves the micrographs under SEM. The surface morphology was evaluated using a 20 kV accelerating voltage.

2.2.3. Contact Angle

Contact angle analysis was performed on a contact angle machine (*VCA Optima*) to classify the interactions between solids and liquids. The sample was squeezed with a glass film to flatten the surfaces for a few days. A droplet of water was positioned on the sample surface as soon as it was placed. The *VCA Optima* took a static or moving image of the droplet. Using a high-resolution camera and advanced computer technology, they identified tangent lines as the foundation for measuring contact angles. The test liquid was dispensed using a syringe. Computerized operations eliminated line-drawing errors, and dynamic images were captured for further analysis.

2.3. Reusability Test

The dry adsorbent sample (So) was weighed within 1.0 ± 0.02 grams, then saturated, drained, and reweighed. The saturated test followed the ASTM F726-12 (short-test) procedure. A quantity of 400 mL cooking oil was chosen, and a 1000 mL beaker was used as a test cell so that the adsorbent could float freely inside the mesh basket. Then, the kapok fiber sample was weighed and placed inside the wire-mesh basket, as shown in **Fig. 1**. The kapok fiber with mesh basket was immersed inside a beaker filled with cooking oil and floated freely for 15 minutes ± 20 seconds. After that, the kapok fiber was removed manually in a vertical orientation and left to drain for (30 ± 3) seconds. A tared weighing pan was placed under the kapok sample to catch any additional drips, and the kapok was immediately transferred into the pan. The sample weight after saturation was recorded.

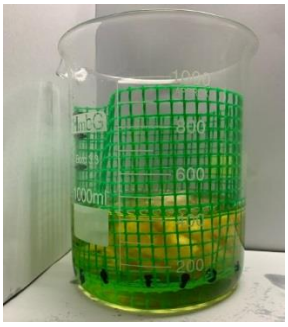

Figure 1. Setup for ASTM F726-12 (short-test) procedure.

After saturation, the sample weight was subtracted from the dry adsorbent weight to obtain total oil adsorbed (O_S). An adsorbent sample was placed on the porous cover, and a stiff plate (metal) of 5kg weight and a flat wooden plate was placed between the kapok and weight for equal distribution force as **Fig. 2**.

The adsorbent was extracted for (15 ± 2) seconds. After removing the weights and plate, the sample was placed into the fresh tared weighing pan and reweighed to within ± 2%. After extraction, the sample weight was subtracted from the dry adsorbent weight to obtain the remaining net oil (O_N). This procedure was repeated until the data for 24 cycles. The data was recorded as O_{S1}, O_{S2}, O_{S3}, O_{S4}, $O_{S5...}$, O_{S24} for each oil adsorbed cycle. All tests were triplicated.

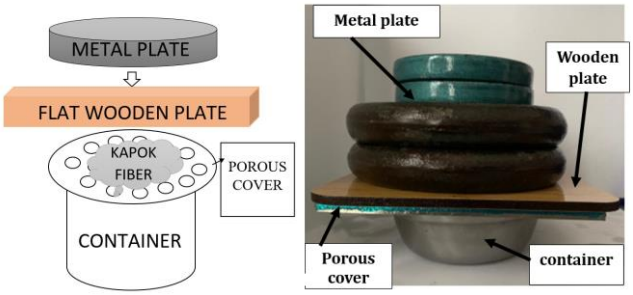

Figure 2. The setup for extracting saturated kapok fiber.

2.3.1. Calculation

The total amount of oil the adsorbent can hold after each saturation cycle is a measure of the degree of deterioration and shall be reported as the absorbency ratio by weight. The absorbency ratio by weight for each cycle was calculated based on the total oil adsorbed as given by **Eq. (1)**.

$$M_x = \frac{O_{sx}}{S_{ox}} \quad (1)$$

Where
M_x = Oil absorbency
S_{Ox} = initial adsorbent weight at the beginning of cycle "x",
O_{STx} = weight of adsorbent samples at the end of cycle "x", and
O_{Sx} = ($-_{Ox}$ − O_{STx}) net oil adsorbed per cycle

3. RESULTS AND DISCUSSION

3.1. Characterization of Kapok Fiber

According to the FTIR spectra of raw kapok fiber in **Fig. 3,** all components of Malaysian kapok are lignocellulosic materials with hydrophobic waxy coatings [10]. Kapok fibers typically comprise 64% cellulose, 13% lignin, and 23% pentosan, similar to cotton [18]. The O-H stretching vibration and aliphatic C-H stretching between 3400-3200 cm^{-1} and 2937-2929 cm^{-1}, which represents mainly the hydroxyls group of cellulose and lignin [19–21]. A peak between 1260-1180 cm-1 indicates the-O stretching vibration band of hemicellulose and lignin [19]. The C=O

stretching vibration of ketones, carboxylic groups, and esters found in lignin and acetyl ester groups is the source of the absorption peak observed at 1680-1642 cm^{-1} [9]. Thus, the key components of lignocellulosic fibers were identified: ellulose, hemicellulose and lignin [19].

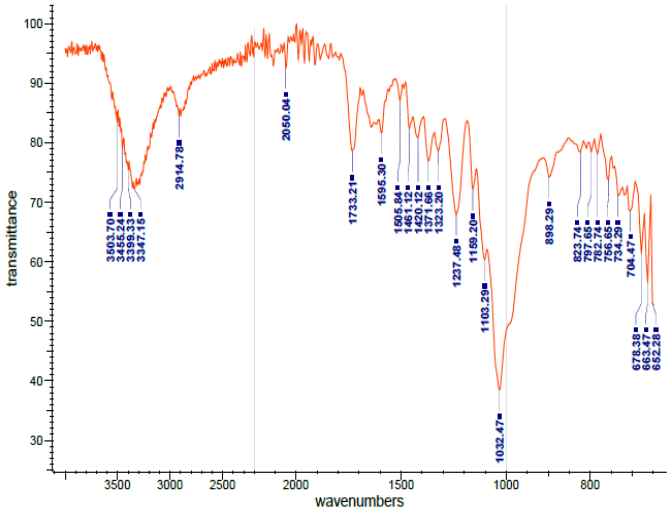

Figure 3. FTIR spectra of raw kapok fiber.

The SEM analysis of kapok microstructures in **Fig. 4** reveals the hollow structure with a thin fiber wall and a large lumen filled with air. Kapok fiber was reported to have a larger lumen size than cotton and comparable to milkweed, affecting oil sorption capacities attributed primarily to target, non-collapsing hollow lumens, thus increasing capillary action [22]. T.T.Lim & X. Huang, stated that the kapok hollow lumen has an external diameter of 16.5 ± 2.4μm and an internal diameter of 14.5 ± 2.4μm, which indicates that the lumen made up 77% of the fiber volume [23]. The analysis also shows that the kapok had an exceptionally smooth surface with no ripples or coarse structure. The smoothness of the surface is due to the wax that has been adhered to the fiber [24].

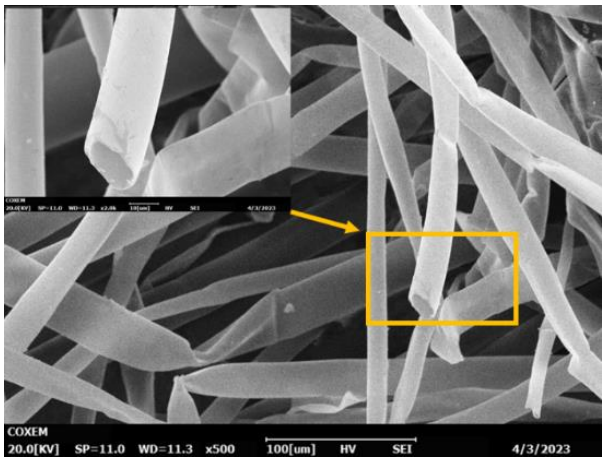

Figure 4. SEM images of raw kapok fiber.

The contact angle reported in this study was 123.80°, as displayed in **Fig. 5,** which exceeds 90°, suggesting that wax on the fiber surface makes kapok fibers hydrophobic and imparts low wettability qualities [25]. The contact angle of kapok fiber has also been observed to be 138.6° and 151.2°, as reported in other studies [25–27]. The result is more than sufficient to demonstrate how hydrophobic kapok fiber is. Nonetheless, depending on the point of origin of the kapok, the contact angle may vary [26].

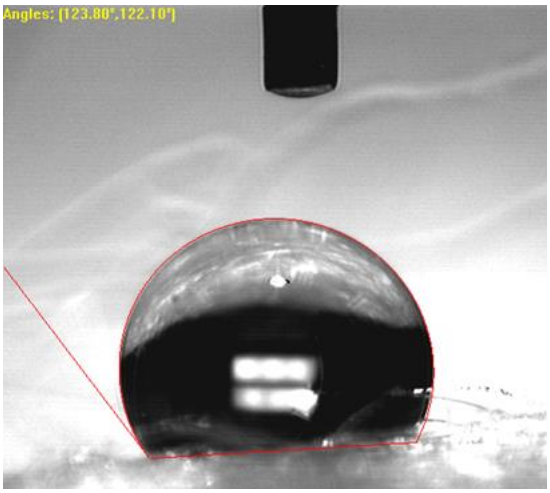

Figure 5. The contact angle of raw kapok fiber.

3.2. Reusability of Kapok Fiber

In each cycle, the kapok fiber was exposed to different types of oils, including blend oil (BO), corn oil (CNO), palm oil (PO), coconut oil (COO), and used oil (UO) (oil that has already been used and might contain contaminants). For each type of oil, the sorbent material absorption capacity was measured for 24 cycles. The initial dry weight was constant at 1g. The absorption capacity was reported as the amount of oil absorbed per gram of the sorbent material, as shown in **Table 2**. On average, kapok fiber absorbed the most palm oil (52.3 g/g), followed by used oil (50.5 g/g), corn oil (48.8 g/g), coconut oil (48.7 g/g) and blend oil (45.9 g/g). The standard deviation values indicate the variability in the absorption capacity across the different cycles for each type of oil. A higher standard deviation suggests greater variability in the absorption results, meaning that the values are spread over a wider range.

In comparison, a lower standard deviation indicates that the values are closer to the mean. Overall, the data suggest that the kapok fiber has varying absorption capacities for different types of oils. The absorption capacity is influenced by factors such as oil viscosity [10], chemical composition [28], and interactions with the sorbent material surface [10]. The speed at which oil infiltrates through capillaries is inversely related to the viscosity of the oil [10]. The relatively consistent absorption capacity values for each type of oil across 24 cycles of reuse indicate the high reusability of the kapok fiber [29].

Table 2 Total oil absorbed, Os for 24 cycles of 5 different types of oil

Cycle	Total oil absorbed, Os (g/g)				
	Blend oil	**Corn oil**	**Palm oil**	**Coconut oil**	**Used oil**
1	42.8	50.0	48.5	49.9	43.2
2	47.0	47.5	52.2	49.5	52.8
3	43.4	48.7	54.9	50.6	54.1
4	45.8	49.5	52.9	52.6	53.7
5	45.6	48.8	52.8	53.6	51.7
6	46.4	48.3	51.5	50.0	53.7
7	43.9	50.5	54.0	49.4	50.4
8	47.7	50.1	52.9	50.2	52.4
9	45.6	49.5	54.9	51.5	52.9
10	47.6	47.6	53.0	50.8	53.5
11	47.2	51.5	53.9	51.1	50.6
12	43.4	50.2	54.7	50.9	52.9
13	47.2	47.0	53.7	47.5	43.0
14	47.2	48.8	52.7	48.0	48.7
15	46.3	48.9	52.3	48.6	50.1
16	45.6	48.9	52.3	49.3	49.8
17	43.8	50.0	51.9	49.8	47.9
18	46.6	47.4	48.9	49.5	46.1
19	47.4	47.3	50.5	42.7	50.0
20	46.6	46.8	48.9	45.0	51.0
21	44.7	49.2	50.8	43.9	50.8
22	46.2	49.5	52.7	45.4	51.6
23	45.9	47.7	53.6	44.7	51.2
24	48.6	48.1	50.8	45.1	50.8
Average	45.9	48.8	52.3	48.7	50.5
Standard deviation	1.5429	1.2248	1.8230	2.8609	2.9925

The oil adsorbency ratio by weight indicates the amount of oil a sorbent can absorb relative to its weight. As a sorbent deteriorates, its ability to effectively adsorb oil may decrease, resulting in a lower oil absorbency ratio. This deterioration can be caused by factors such as saturation, exposure to environmental conditions, and chemical interactions with absorbed substances. So, a lower oil adsorbency ratio often corresponds to a higher degree of deterioration of the sorbent [30].

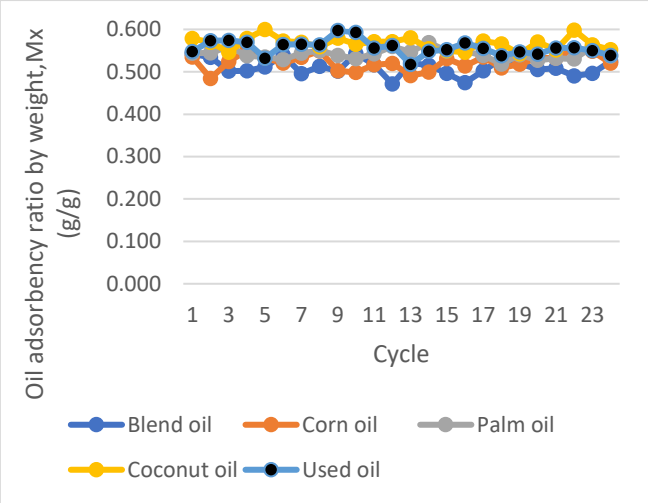

Figure 6. Oil Adsorbency Ratios of Different Oils by Weight (Mx) in g/g.

The cycles represent the repeated use of kapok for oil spill clean-up. Based on **Fig. 6**, the Mx values vary slightly across different cycles but do not show a clear trend of increasing or decreasing over time. This suggests that kapok fiber has good durability and reusability for oil absorbency. The Mx values also vary slightly across different types of oils but are generally within the range of 0.47 to 0.60 g/g. This indicates that kapok has good compatibility and selectivity for oil absorbency, regardless of the oils' viscosity, density, or polarity.

The highest Mx value is 0.600 g/g for coconut oil in cycle 5, while the lowest is 0.471 g/g for blend oil in cycle 12. The average Mx values for each type of oil are as follows: blend oil (0.510 g/g), corn oil (0.522 g/g), palm oil (0.541 g/g), coconut oil (0.569 g/g) and used oil (0.555 g/g). Based on these averages, it can be seen that coconut oil has the highest average oil absorbency ratio by weight, followed by used oil, palm oil, corn oil, and blend oil. This could be related to the molecular structure and composition of the oils, such as their chain length, degree of saturation, and presence of impurities. For example, coconut oil has a high content of medium-chain fatty acids [31], which may make it more compatible with kapok fiber than other oils with longer or shorter chains. Used oil had a higher absorbency ratio than fresh oils because it contains more contaminants and oxidized compounds, which increase its polarity and affinity with kapok.

4. CONCLUSION

In summary, kapok fiber is renowned for its conventional use in creating plush fillings for items like pillows and mattresses. However, it also exhibits promising attributes that make it suitable as an efficient and ecologically sound oil sorbent. These characteristics underscore its potential in this alternative application, including its notable hollow structure with a thin fiber wall and a large lumen filled with air, hydrophobic nature, and reusability. Each of these characteristics is supported by the result obtained. For example, SEM results show that kapok has a large percentage of the hollow lumen, contributing to its excellent oil absorbency and retention capacity. FTIR results demonstrate the hydrophobic nature of kapok due to it being a lignocellulosic fiber (presence of lignin). The contact angle results also confirm kapok's hydrophobicity, as the contact angle exceeds 90°. Lastly, the reusability test shows that kapok maintains high reusability after 24 cycles of sorption/desorption. Continued research and refinement could unlock further potential for utilizing kapok fibers as oil sorbents while addressing inherent constraints.

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