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Study the Hydrophobicity/Oleophilicity Properties of Kapok Fibre and Its Performance in Diesel/Water Filtration

Adam Danial[1], Rosniza Hussin[1]*

[1]Department of Mechanical Engineering Technology, Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia, 84600 Pagoh, Johor, MALAYSIA

*Corresponding Author Designation

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Abstract: Kapok fibre is harvested from the seed of Ceiba pentandra. Kapok exhibit some unique characteristics that favors it to be the study subject for various researchers. Thus, study was conducted to find out the plausible of kapok fibre as a sorbent for diesel spill. The objectives of this study are to study the surface properties of kapok fibre and to evaluate the interaction mechanism of kapok fibre in diesel/water separation performance. Packing density of kapok fibre has been the parameter in this study and the determined packing densities to find the oil sorption capacity are 0.0506 gcm^{-3}, 0.0759 gcm^{-3} and 0.1012 gcm^{-3}. Packed kapok fibre was placed on the surface of diesel/water mixture for 30 minutes. The oil sorption capacity for packing densities of e 0.0506 gcm^{-3}, 0.0759 gcm^{-3} and 0.1012 gcm^{-3} are 15.51 gg^{-1}, 10.83 gg^{-1} and 8.75 gg^{-1} respectively. Diesel oil is successfully absorbed by kapok.

Keywords: Oil Sorption Capacity, Scanning Electron Microscope, Kapok Fibre

1. Introduction

For the past few years, there are a lot of incidents involving diesel spill into ocean. For example, diesel spill which happened in Norilsk and Taimyr, Russia due to the collapse of the fuel tank on May 2020 [1] Diesel are used to operate heavy machine, fuel for heavy vehicle and fuel to light up oil lamp in rural area also during festive celebration. Oil spill pollution in the marine environment can aggravate human health and disrupt sustainability of marine resources [2]. There are many different ways to clean up oil spill and are grouped by categories. The first group is devoted to physical techniques such as adsorbents [3] booms [4] and skimmers [5]. The second classification entails the biological methods like bioremediation processes that employ specific bacteria while the third group is chemical method involving chemical coagulation and electrocoagulation [6]. Among all of the oil spill clean-up method, sorbent is considered the most efficient [7].

Sorbents are material with high attractions for oil and repellent for water [8] which works by absorption [9] and adsorption [10]. This study focusses about how natural fibre, specifically kapok fibre

**Corresponding author: rosniza@uthm.edu.my*

is a good material to recover oil as a sorbent. Kapok is known to have characteristics which made it a good sorbent material to be used in the industry besides other popular sorbents such as saw dust [11], feathers [12], zeolite [13] and other readily available carbon-based product.

The objectives of this study are to analyse surface properties of kapok fibre and to evaluate the interaction mechanism of kapok fibre in oil/water separation performance.

2. Materials and Methods

The materials and methods section, otherwise known as methodology, describes all the necessary information that is required to obtain the results of the study.

2.1 Materials

Kapok purchased was from Malaysia. Seeds other non-fibre were removed before packing the kapok into the desired packing density. The determined packing density are n 0.0506 g cm^{-3}, 0.0759 g cm^{-3} and 0.1012 g cm^{-3}. Kapok are packed into petri dishes and then were tighten by strings.

Diesel was purchased from local petrol station and wastewater were collected from nearby drain.

2.2 Oil Adsorption/Absorption

Diesel was measured at 1000 ml and mixed with wastewater measuring at 4000 ml. After oil and water has separated completely, the height of wastewater and diesel inside the container were measured before immersing kapok in the mixture. Weight of kapok were measured first then 12 cluster of kapok with same packing density are placed on the surface of diesel – water mixture. After the first 15 minutes, each of the kapok cluster were flipped to ensure the maximum oil sorption. After the second 15 minutes has reached, the kapok was lifted using wire gauze and let rested for the excess diesel to drip into the container back. After 15 minutes resting the kapok, the weight of each cluster was measured to get the average weight.

Experiment was repeated using kapok with packing density of 0.0759 g cm^{-3} and 0.1012 g cm^{-3}.

2.3 Equations

Kapok was weighed before packed into individual cluster. To calculate the packing density, Eq. 1 is used. The packing density was calculated in gram per centimeter cubic (cm^{-3})

$$packing\ density = \frac{weight\ of\ kapok\ (g)}{voume\ of\ container\ (cm-3)} \qquad Eq.\ 1$$

The volume of the petri dish used as the container for kapok has a volume of 79.05 cm^{-3}.

Oil sorption capacity can be calculated using Eq. 2.

$$oil\ sorption\ capacity = \frac{mv - mi}{mi} \qquad Eq.\ 2$$

The unit of oil sorption capacity is in grams per gram (g g-1). mv in the equation is the final weight of kapok which is the average of kapok after immersion in diesel and mi is the initial weight of kapok.

2.4 Characterization of Kapok Fibre

2.4.1 Surface Morphology of Kapok Fibre Using SEM

Kapok's surface morphology was analyzed using Scanning Electron Microscope (SEM) from Jeol, Japan, the JSM-IT800. Kapok were dried according to the standard procedure which is 110 °C for 24 hours. Kapok was coated in gold to increase the conductivity. Sample of kapok was placed on steps using sticky carbon steps. The camera position was adjusted using two knobs that control the x-axis movement and y-axis movement.

2.4.2 Thermal Analysis of Kapok Fibre Using TGA

Thermal stability of kapok fibre have been studied using a thermogravimetric analyzer from Linseis, L81-I STA. Before using the machine, calibration on the machine must be done to prevent any error. The calibration included balance calibration, furnace calibration and temperature calibration. For raw kapok fibre, the temperature was set from 30 °C to 700 °C or until it completely degrades. The heating rate was set at 10 °C/min under oxygen atmosphere. Curve indicating the thermal stability were shown on the screen. Small pieces of kapok weighing 2.8 mg is prepared and placed inside the crucible. The process is then started.

2.4.3 Surface Analysis of Kapok Fibre Using Contact Angle

The contact angle of kapok fibre were studies using the sessile drop method where a drop of liquid is placed on the surface of material. This was done by using contact angle analyzer from VCA Optima. A drop of water and a drop of diesel oil was placed on the surface of kapok fibre by inserting the amount of liquid that is needed in the software. The liquid droplet was placed automatically by the needle of the machine. The contact angle of the liquid was shown on desktop. Image of the droplets on kapok fibre were taken by the analyzer.

2.4.4 Fourier Transform Infrared Spectroscopy

The stage of the FTIR machine is cleansed with alcohol to remove impurities. Small piece of kapok is placed on the stage. Wavelength ranging from 4000 cm^{-1} – 400 cm^{-1} was set and for each spectrum 32 tests were run.

3. Results and Discussion

3.1 Oil Sorption Capacity

The height of diesel inside the container with the dimensions of 37.0 cm x 29.0 cm x 12.0 cm is 1.3 cm while the height of water is 4.4 cm. The average weight of oil and water before immersion of kapok is 739.11 g and 3964.33 g respectively.

After immersion of kapok, the height of water and diesel residue is 4.4 cm for all packing density while the average weight after immersion for kapok fibre with packing density of 0.0506 g cm^{-3}, 0.0759 g cm^{-3} and 0.1012 g cm^{-3} are 3947.00 g, 3895.33 g and 3917.33 g respectively.

Kapok with packing density of 0.0506 g cm^{-3} have 4 g of raw kapok, packing density of 0.0759 g cm-3 contains 6 g of raw kapok and packing density of 0.1012 g cm^{-3} contains 8 g of kapok. The average weight of each of the kapok's cluster for kapok with packing density of 0.0506 g cm^{-3} is 66.03 g, kapok with packing density of 0.0759 g cm^{-3} have average weight after immersion 70.97 g and average weight of 78.000 g after immersion for kapok with packing density of 0.1012 g cm^{-3}.

The oil sorption for each packing density are shown in Table 1 and Figure 1:

Table 1: Oil Sorption Capacity

Packing Density (g cm^{-3})	Oil Sorption Capacity (%)	Oil Sorption Capacity (g g^{-1})
0.0506	1550.75	15.51
0.0759	1082.83	10.83
0.1012	875	8.75

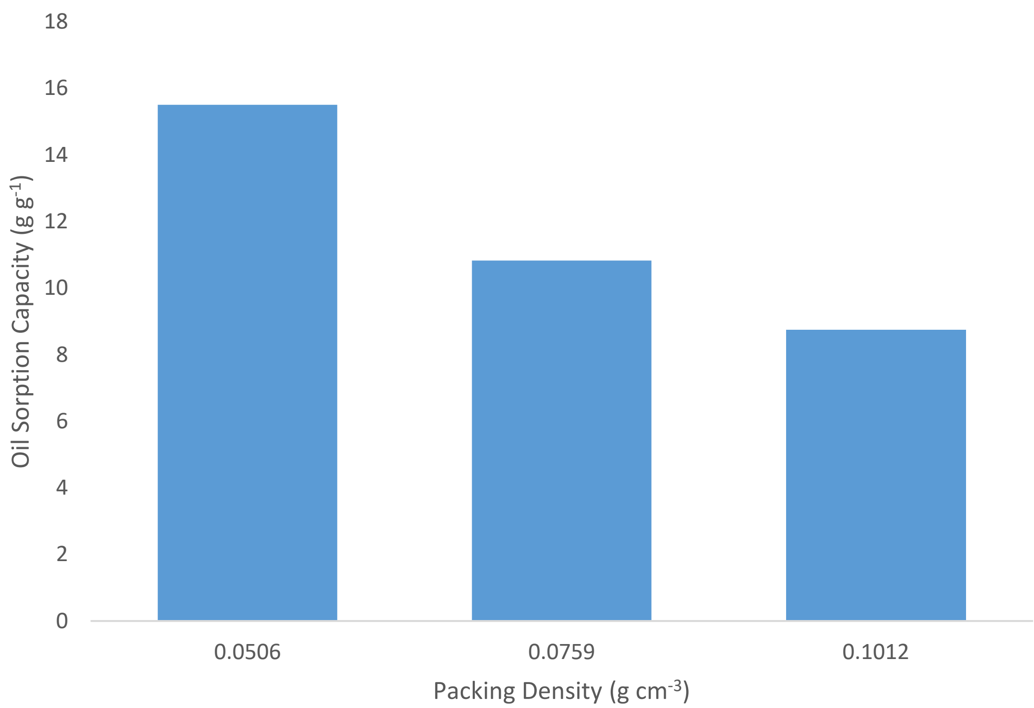

Figure 1: Oil sorption capacity vs packing density

From Figure 1 shown that the packing density of 0.0506 gm^{-3}, the oil sorption capacity is a 15.51 g g^{-1} or 1550.75 %. The sorption capacity decreases as the packing density decrease to 0.0759 gm^{-3}, with the oil capacity is at 10.83 g g-1. When the packing density at 0.1012 gcm^{-3}, the oil sorption capacity decreases to 8.75 gg^{-1} or 875 %. However, the diesel sorption values obtained from previous studies in range 27.55 gg^{-1} to 30.5 gg^{-1} [14][15]. With increasing packing densities, the vacancy percentage within the kapok microstructure decreases linearly, resulting in less capacity for oil absorbing. Lower packing density does not imply more ability to absorb oil, but it does indicate the highest efficiency rate for absorbing 1 L diesel [16]

The condition of raw kapok before and after immersion in diesel are as in Figure 2:

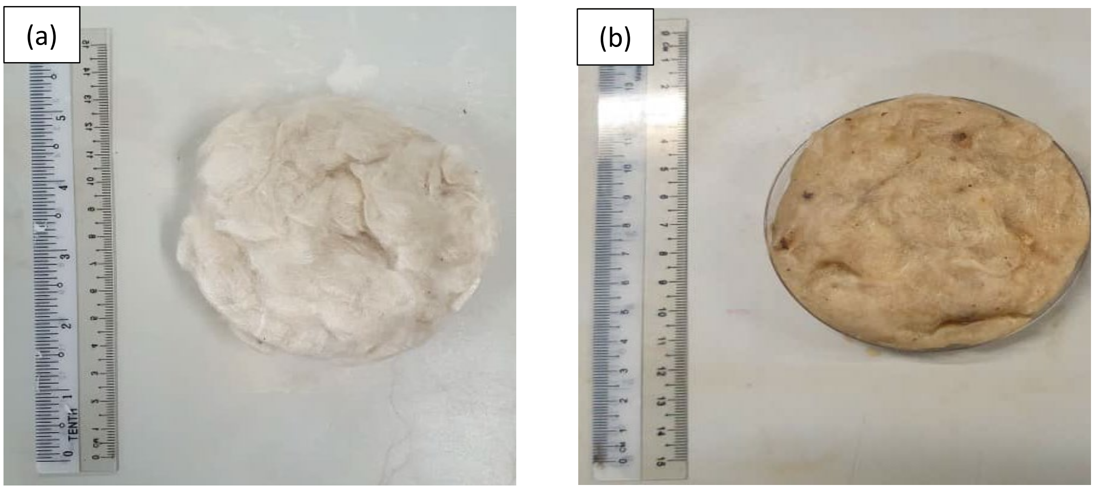

Figure 2: Condition of kapok (a) before immersion (b) after immersion

3.2 Surface Analysis of Raw Kapok Fibre Using SEM

Figure 3 show the raw kapok fibre have a silky appearance and smooth surface. This is because the wax that is covering the surface of kapok. Due to the wax covering the surface of kapok fibre, it is known that kapok fibre has the characteristic of hydrophobic. Inside of kapok fibre is hollow tubular structure [17] with diameter of 27.1 ± 2.5 µm was found in the obtained kapok fibre and when compared to study by Yang et al. (2017), it is slightly larger than the average diameter of kapok fiber obtained from Indonesia at 19.31 µm [18]. Figures from SEM shows that the kapok obtained were similar to studies made from various researchers. Raw kapok has a tubular structure filled with air. When kapok is treated with alkali, the air inside the tubular structure disappeared [19] that shows why the raw kapok have a perfect tubular structure.

Figure 3 shows the morphology of raw kapok fibre that have silky appearance and smooth surface.

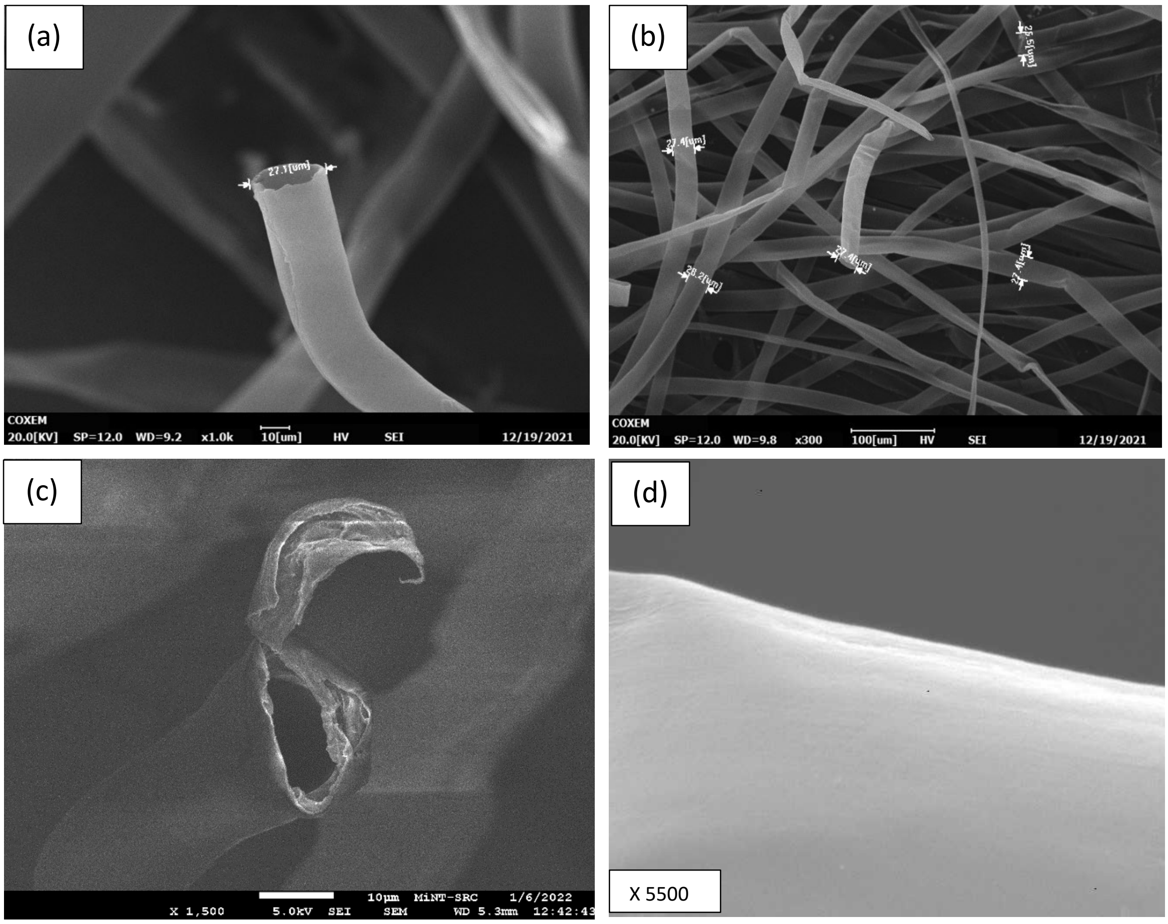

Figure 3: Kapok fibre under SEM (a) diameter of kapok ends (b) diameter of kapok strands (c) end of kapok (d) surface of kapok

Figure 4 show the EDX/EDS analyzation of a sample. The results of EDX/EDS acquired shows that kapok contains carbon (C), oxygen (O), aluminum (Al), gold (Au), potassium (K), copper (Cu), calcium (Ca) and indium (In). Three of the highest elements are C, registered weight at 59.56 %, O at 26.57 %, and Al at 10.52 %, while a study by Quek et al. (2019) found that kapok contains 45.20 % of C, 48.10 % of O and no Al using X-ray Diffraction spectrum [20]. The elements found in the study is corresponding to the molecular formula of cellulose ($C6H10O5$). Au is presence in the data at 2.29 %, might come from the coating process before running it under SEM.

Figure 4 shows the element presence in kapok and Table 2 explain the presence of elements precisely.

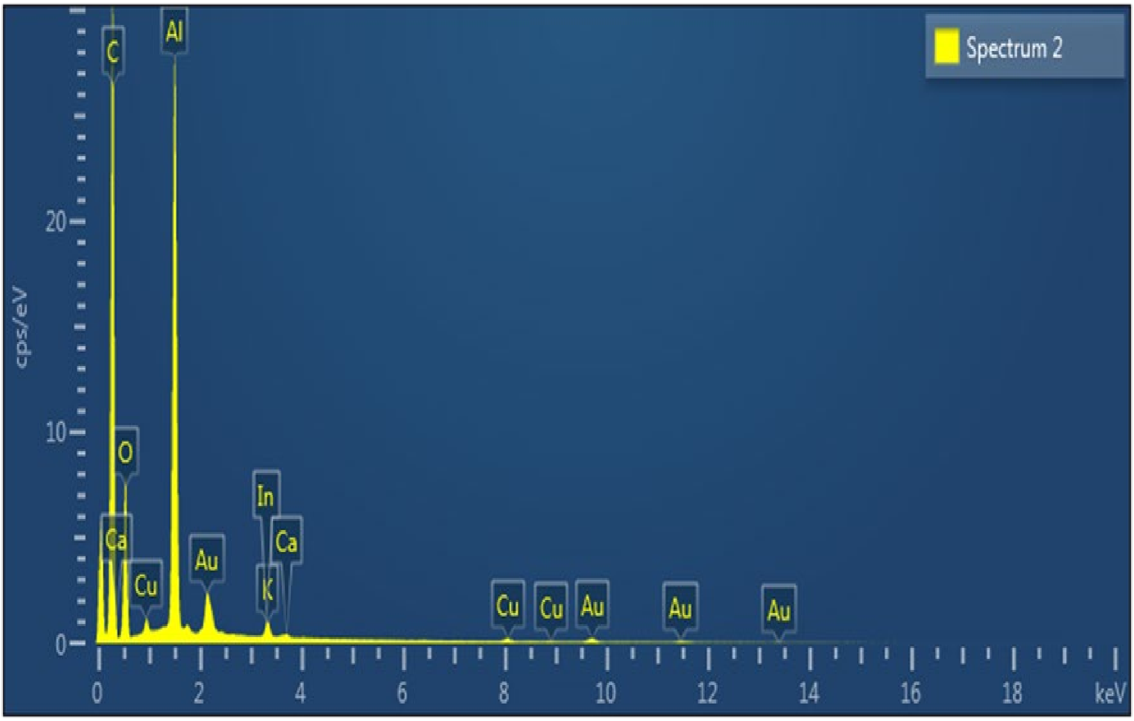

Figure 4: EDX/EDS analysis of raw kapok fibre

Table 2: Table of element presence in kapok fibre

		Spectrum 2		
Element	Line Type	Weight %	Weight % sigma	Atomic %
C	K series	59.56	0.24	70.42
O	K series	26.57	0.20	23.59
Al	K series	10.52	0.06	5.54
Au	M series	2.29	0.05	0.17
K	K series	0.40	0.01	0.14
Cu	K series	0.47	0.03	0.10
Ca	K series	0.09	0.01	0.03
In	L series	0.11	0.03	0.01
Total		100.00		

3.3 Surface Properties of Kapok Fibre Using Contact Angle

Figure 5 shown the water is dropped on flat raw kapok, it is observed that the angle between kapok surface and water droplet is 138.70 ° at minimum to Angles: (145.80°,146.30°) ntact angle of kapok fibre produced is higher than that of Zhang et al. (2016) [14], which is 114.70 °, and lower than that of Dong et al. (2015b) [21], which is 151.20 °. This shows that the kapok used in this study have lower wettability in comparison to the kapok used in Zhang's study. Other than raw kapok, kapok that has been immersed in diesel was also tested to find out its contact angle. The angle that has been obtained is 145.80° minimum and maximum at 146.30°. Figure 4.6 shows the contact angle between water and kapok immersed in diesel. The presence of diesel increases the contact angle between water and kapok, this is because the presence of oil which is hydrophobic to water [22]. Figure 5 is the angle of water on raw kapok and on raw kapok immersed in diesel:

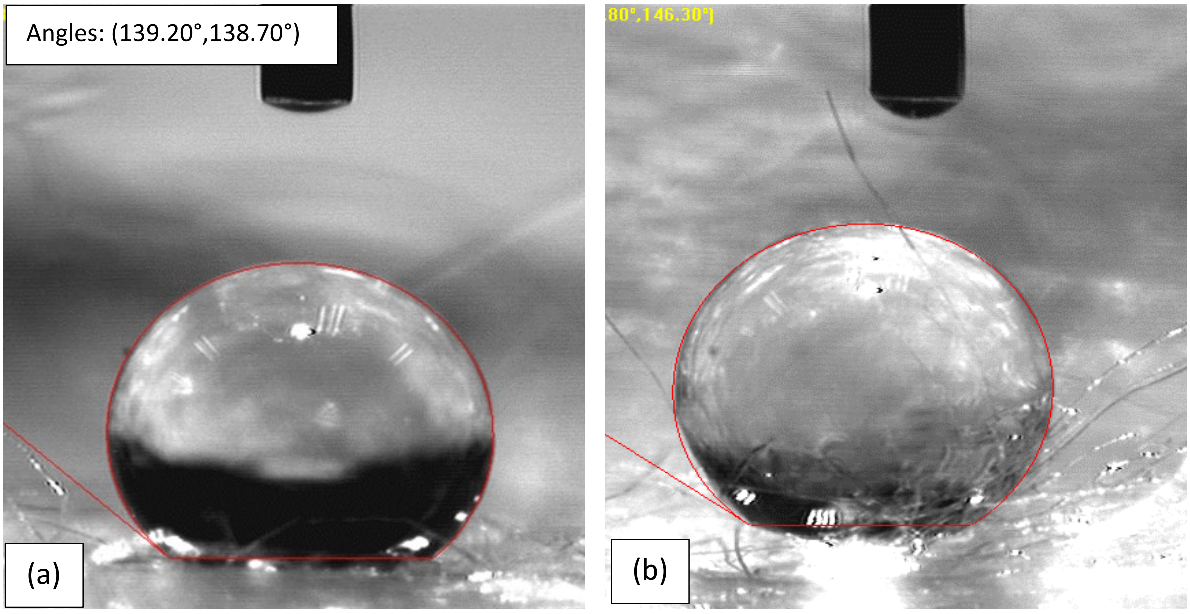

Figure 5: contact angle (a) on raw kapok (b) on raw kapok immersed in diesel

3.4 Structure Analysis Using FTIR

Figure 6 shown FTIR analysis on raw kapok fibre and raw kapok fibre immersed in diesel. For raw kapok fibre, it has various peaks emerged. The widest peak at 3342.97 cm^{-1} which according to Wang *et al.* (2012) [23] and Quek *et al.* (2019) [20] it happens because of the O-H stretching vibration. The following peak happens at 2917.53 cm^{-1}, where the stretching vibration on C-H. Stretching vibration on C=O, happens a 1732.11 cm^{-1} is similar to studies made by Chen *et al.* (2013) [24] and Draman *et al.* (2014) [19] where their values are 1737 cm^{-1}. At 1240.11 cm^{-1} a peak emerges, the value is almost similar to other researchers.

Raw kapok immersed in diesel have a relatively similar result to raw kapok in the FTIR test except the peak is slightly more extensive as seen in the Figure 4.7. The broadest peak is at 3337.73 cm^{-1} for raw kapok immersed in diesel. The presence of a deep peak at 2922.24 cm^{-1} and 2853.78 cm^{-1} shows that there is an occupancy of diesel oil in the kapok.

Figure 6 shows the FTIR analysis on raw kapok and raw kapok immersed in diesel:

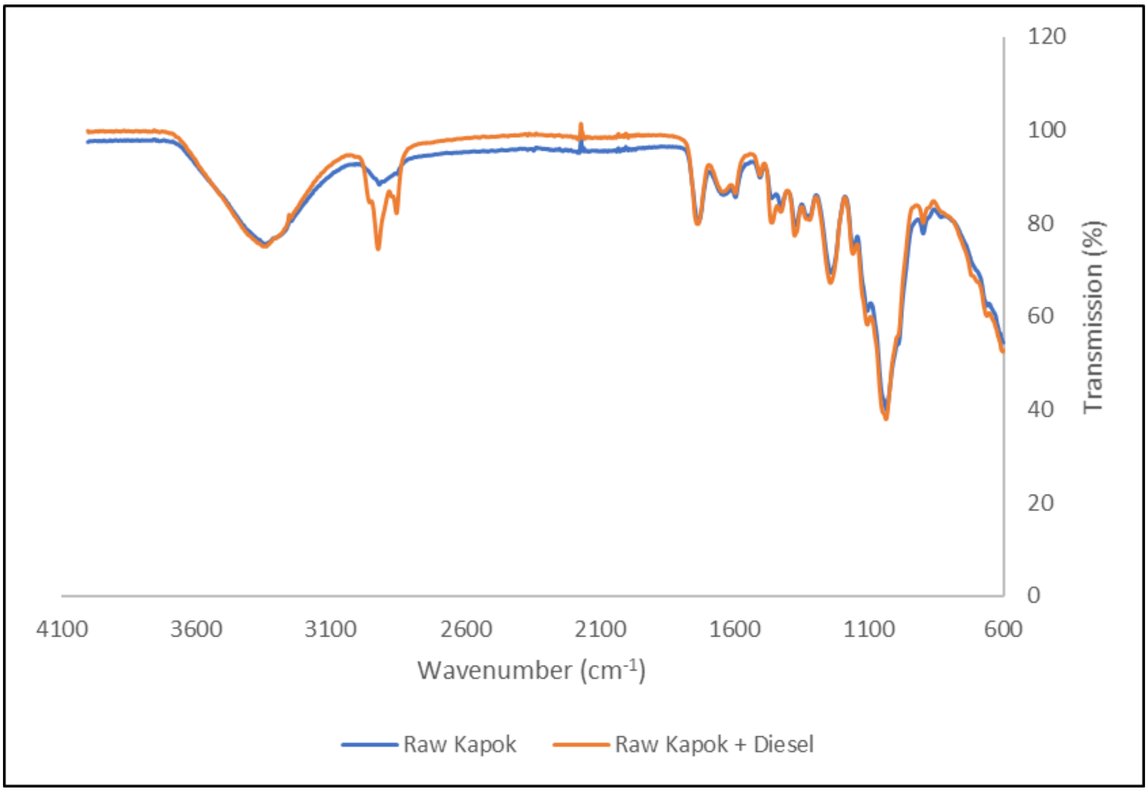

Figure 6: FTIR data on raw kapok and raw kapok immersed in diesel

3.5 Thermal Analysis Using TGA

Thermal analysis is conducted on raw kapok fibre before immersion. The data produce a curve called Difference Thermo Gravimetry (DTG) curve and thermogravimetry (TG) curve (Figure 7). Figure 7 shown the DTG curve, the changes of mass below 100 °C is believed to be the evaporation of water molecules from inside the kapok [25]. The first steep peak happens between 250 °C and 300 °C. The phenomenon happens here is the thermal degradation of hemicellulose [19]. The deepest peak continues from 300 °C to 370 °C where kapok started to degrade vigorously this is supported by a study by Jamat and Asik (2019) [26] where they found that kapok started to degrade at 350 °C. At 400 °C to 700 °C the graph experience less obvious peak because the only degradation of lignin happened [19].

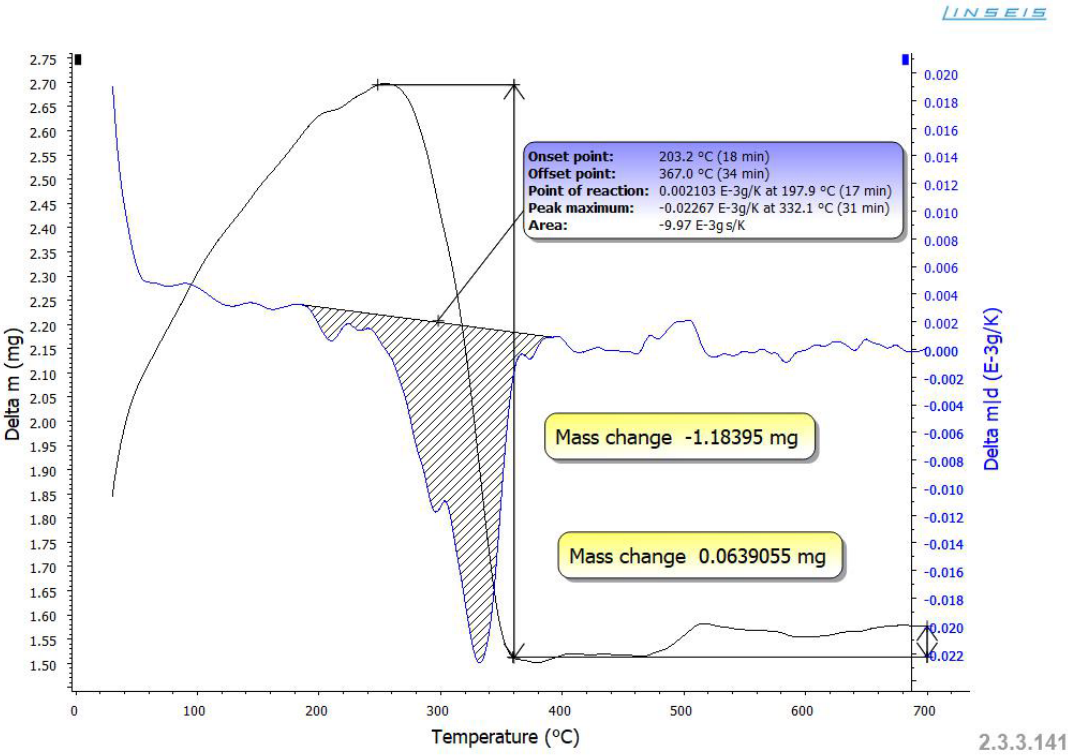

Figure 7: DTG and TGA curve of raw kapok

4. Conclusion

This study was conducted to find out the performance of kapok fibre in water/diesel absorption by comparing the packing density of kapok fibre by performing characterization using SEM, contact angle, TGA and FTIR. The oil sorption capacity that were observed in this study is lower than the oil sorption capacity conducted by other researchers. The characterization results of kapok fibre through SEM, contact angle, TGA and FTIR were corresponding to the results obtained by other researchers.

The oil sorption capacity with packing densities of 0.0506 g cm^{-3}, 0.0759 g cm^{-3} and 0.1012 g cm^{-3} are 15.51 g g^{-1}, 10.83 g g^{-1} and 8.75 g g^{-1} respectively. The oil sorption capacity of this study is lower than other researchers is because the high packing density which is weak when comparison are made.

The morphology of raw kapok fibre on the subject of diameter have an average of 27.1 ± 2.5 µm in which have a difference about ±8.00 %. The contact angle obtained from this study for raw kapok fibre and raw kapok fibre immersed in diesel are 138.95 ° and 146.05 ° sequentially. The broadest peak of FTIR result for raw kapok is 3342.97 cm^{-1} while the broadest peak for raw kapok immersed in diesel is 3337.73 cm^{-1}. Below 100 °C the decreasing mass is caused by the evaporation of water and at 250 °C to 300 °C, hemicellulose degraded. At 400 °C to 700 °C, lignin degraded slowly.

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