Kapok-cotton Carbon Sponges for Oil Recovery

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Abstract. Carbonized natural fibers show great promise as sorbents because of their low fabrication costs, high surface area, high sorption capacity, and improved oil selectivity. Pyrolysis was performed on cotton and kapok fiber blends to produce carbon fiber sorbents. The carbon sponges showed improved mechanical properties with the addition of cotton. Pure carbonized kapok fibers were quite brittle, leading to challenges in recovery after use. The static water contact angle of carbonized kapok fibers, carbonized kapok-cotton blend (50K50C), and carbonized cotton fibers were determined to be 137.0°, 135.0°, and 135.9° respectively. This was an observed improvement from 127.9° for raw kapok and 0° for raw cotton. Sorption experiments revealed that the 50K50C fibers have sorption capacities about 25-27 times its original weight at 27.77 g/g, 25.72 g/g, and 26.01 g/g for motor oil, palm oil, and diesel, respectively.

1. Introduction
The use of sorbents in oil spill clean-up has been found to be the cheapest and most effective compared with other remediation methods like chemical treatments and in situ burning. Oil sorbents must exhibit high oil uptake, low water retention, and high recyclability to be considered effective. They are classified based on the type of materials they were sourced from. They could be inorganic, synthetic, and natural [1]. Inorganic sorbents such as zeolite [2,3], and perlite [4], are the cheapest sorbents but are also the least efficient in terms of oil recovery. Synthetic sorbents such as polyurethane [5,6], waste plastics [7], and polylefins [8], have high oil sorption capacities but are not recyclable [9]. In addition, these sorbents have high production costs. Natural sorbents such as rice...
husk [10], kapok [11–15], cotton [16–18], banana peels [19], corn straw [20], and luffa [21], can be excellent sources of sorbents since they are renewable, recyclable, biodegradable, and non-toxic [22]. Cotton is a reliable natural sorbent source because it is composed mainly of cellulose at 88-96.5% [23]. However, raw cotton has poor buoyancy, low oleophilicity, and low hydrophobicity. Surface modifications may be necessary to improve the fibers’ properties to be an efficient oil sorbent [18,24]. Meanwhile, kapok is a natural plant fiber that has low density, good buoyancy, high hollowness, and excellent hydrophobicity [25–29]. These unique characteristics provide the kapok fibers (KF) with higher oil sorption capability compared with other natural oil sorbents. Cotton fibers are thicker and more compact, while kapok fibers have thinner and hollower fibers. This hollow fiber structure of kapok provides more surface area for sorption. One of the techniques to improve the fibers’ oil sorption capacity is by carbonization of the fibers through pyrolysis [25].

This paper investigates the effect of pyrolysis on raw kapok, raw cotton, and kapok-cotton blends on their morphology, chemical composition, and oil sorption capacity. Pyrolysis is one of the techniques employed in improving the hydrophobicity of natural fibers [30]. This method is known to be relatively cheaper as it does not require costly preparation techniques [18]. This is done by removing the non-carbon atoms in the material’s structure via high temperature (300-1000°C) in an inert nitrogen atmosphere. This paper study seeks to combine the pliability of the cotton fibers and the high surface area of kapok fibers in creating a carbon-based sorbent.

2. Formatting the title, authors and affiliations (Methodology)

2.1. Materials.

Kapok fibers were obtained from local fruit-bearing kapok trees. Cotton fibers were obtained from a local supermarket. Commercially available diesel, palm oil, and motor oil were used as model oils for sorption experiments.

2.2. Preparation and Characterization of Carbonized Natural Fibers.

Kapok-cotton fiber blends were prepared using a regular kitchen blender. Raw kapok, raw cotton, and kapok-cotton blends with 50-50 weight ratio were carbonized at 400 °C for 3 h in a tube furnace under nitrogen (N₂) environment. The samples prepared are listed in Table 1. Static water contact angle measurements of the carbonized fibers were performed using Dino-Lite AM2111-0.3MP USB Digital Microscope. Fourier Transform Infrared Spectroscopy (FTIR, Nicolet iS50 FT-IR) was done to determine the extent of the fibers’ carbonization. Surface morphology was observed using scanning electron microscopy (SEM, JEOL JIB-4000 Plus). The non-conductive samples were sputtered with platinum.

| Table 1. Composition of the CCK fibers. |
|---------------------------------------|
| Kapok composition | Cotton composition | Sample label            |
| 100%               | 0                   | carbonized kapok        |
| 50%                | 50%                 | 50K50C                  |
| 0                  | 100%                | carbonized cotton       |

2.3. Oil Sorption Capacity Measurements.
Maximum sorption capacity was performed by immersing 50 mg in 25 mL of oil for 30 min. The fibers were collected, drained to remove excess oil, and then weighed. Oil sorption capacity (q) was calculated using Eq. 1:

\[ q = \frac{m_f - m_i}{m_i} \]  

where \( m_i \) and \( m_f \) are the weights of the fibers before and after immersion in oil, respectively.

3. Formatting the title Results and Discussion

3.1. Characterization of Carbonized Cotton-Kapok Fibers.

FTIR spectroscopy was done to determine the extent of carbonization of the fibers after pyrolysis. Fig. 1 shows the FTIR spectra of the carbonized samples and raw kapok. After carbonization, changes can be seen at the peaks found at 3328 cm\(^{-1}\), which are attributed to \(-\text{OH} \) stretching, 2918 cm\(^{-1}\) for \(-\text{CH} \) stretching, and at 1250 and 1057 cm\(^{-1}\) for \(-\text{CO} \) stretching. This is due to the removal of non-carbon components (oxygen and hydrogen) in cellulose and hemicellulose of the cotton and kapok fibers [31,32]. The noticeable peak at 1598 cm\(^{-1}\), which is linked to C=C bonds, has not changed after the carbonization process. This indicates the existence of some lignin in the carbonized kapok fibers even after pyrolysis [31]. Lignin, which burns at a wide range (100-900°C) [33–35], remained at the carbonized samples since the pyrolysis took place at 400°C.

![FTIR spectra](image)

**Fig. 1.** FTIR spectra of (a) 50K50C, (b) carbonized kapok after carbonization at 400°C for 3h compared against that of (c) raw KF.

Figure 2 shows the optical images of the carbon sponges after carbonization. After carbonization, the kapok fibers feel more brittle and break down easily while the carbonized cotton fiber appears to hold its shape. Meanwhile, the carbonized kapok fibers appear less brittle than the carbonized kapok fibers but also less pliable than the carbonized cotton fibers. The combination of kapok fibers and cotton fibers was done to help the carbon sponges retain its shape during oil recovery.
Figure 3 shows the morphology of the carbonized kapok, carbonized cotton, and carbonized kapok-cotton blends viewed using SEM. The tubular structure of the kapok fibers was retained after pyrolysis as seen from the SEM images of the carbonized kapok and carbonized kapok-cotton blends in Figure 3. However, the heat due to the pyrolysis might have caused the fibers’ tubular structure to shrink. It was reported that the inner diameter of raw kapok fibers is in the range of 16-25 µm [36]. Based on the SEM images, the estimated inner diameters of the carbonized kapok fibers at Fig. 3b and Fig. 3d have shrunk to 7-10 µm. The diameter of the cotton fibers was reduced to about 5-7 µm from 15-20 µm as previously reported by literature [18]. SEM images also showed improved surface roughness due to the pyrolysis of the fibers. Meanwhile, the cotton fibers were observed to have higher surface roughness as well. These changes in roughness were due to the evolution of volatile compounds that were released during pyrolysis [37].

Figure 4 shows the average static water contact angle measurements on the raw kapok fibers and carbonized kapok fibers. The measured contact angle of cotton fibers and raw kapok is 0° and 129.7°, respectively. All carbonized samples have higher contact angles with water when compared to raw kapok. The increase in water contact angle for the carbonized fiber is due to the higher surface roughness after pyrolyzing and/or the lowering of the surface/interfacial energy when the organic carbon fibers were carbonized [38]. Furthermore, the existence of the polar moieties in the organic
kapok fibers might have been lost or reduced when they transformed into inorganic carbon during carbonization.

### 3.2. Oil Sorption Capacity

The oil sorption capacity of the carbonized samples was observed using three model oils of differing viscosities (motor oil > palm oil > diesel). It was observed that the sorption capacity of 50C50K fibers was lower for all three model oils but was able to retain its shape throughout the sorption experiments. The observed shrinking of the cotton and kapok fibers might have reduced the fibers’ surface area despite the observed roughening of the surface. In general, diesel has the lowest sorption capacity followed by palm oil and motor oil. Motor oil showed to have the highest sorption capacity. Motor oil is mainly comprised of C7 to C8 (toluene, ethyl benzene, xylene) [39], which means ease of access to reach the surface of the carbonized fibers. This is followed by palm oil (C16 to C18) that is composed mostly of aliphatic linear chains of palmitic, stearic, oleic and linoleic fatty acids [40]. The low sorption capacity of diesel (C10 to C15) is attributed to the presence of its long aromatic rings that can or sterically hinder its interaction with the binding sites on the surface of the composite fibers.

![Fig. 4. Static water contact angle measurements of (a) raw KF and (b) carbonized kapok, (c) 50K50C, and (d) carbonized cotton.](image)

![Fig. 5. Sorption capacities of carbonized kapok fiber after immersion in motor oil, palm oil, and diesel.](image)
4. Conclusion

This study investigated the effect of differing cotton and kapok ratios on the sorption capacity of carbonized kapok fibers on various oils. FTIR spectroscopy revealed the removal of the cellulosic components in the carbonized kapok fibers after carbonization while retaining some lignin in the structure. SEM imaging shows that the tubular structure of the kapok fibers in the carbonized kapok was maintained. High surface roughness was also observed, resulting in a higher water contact angle at a maximum of 137.0°. Oil sorption capacity measurements showed that 50C50K carbon sponges were able to collect about 25-27 times its original weight at 27.77 g/g, 25.72 g/g, and 26.01 g/g for motor oil, palm oil, and diesel, respectively.

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