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Investigation of acetylated kapok fibers on the sorption of oil in water

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Abstract
Kapok fibers have been acetylated for oil spill cleanup in the aqueous environment. The structures of raw and acetylated kapok fiber were characterized using Fourier transform infrared (FT-IR) spectroscopy and scanning electron microscopy (SEM). Without severe damage to the lumen structures, the kapok fibers were successfully acetylated and the resulting fibers exhibited a better oil sorption capacity than raw fibers for diesel and soybean oil. Compared with high viscosity soybean oil, low viscosity diesel shows a better affinity to the surface of acetylated fibers. Sorption kinetics is fitted well by the pseudo second-order model, and the equilibrium data can be described by the Freundlich isotherm model. The results implied that acetylated kapok fiber can be used as the substitute for non-biodegradable oil sorption materials.

Key words: kapok fiber; acetylated; oil sorption capacity; kinetics; isotherms
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Introduction
The environmental pollution caused by various kinds of oil has becoming a serious problem of affecting aquatic life survival and human economic activity (Aguilera et al., 2010; Ha et al., 2008). The common measures used to remove oil in water involve dispersants, skimmers, oil booms, and sorbents, etc. (Banerjee et al., 2006). Thereinto, the most of dispersants can cause a secondary pollution and do harm to fish, fowl, and various mammals, while skimmers and oil booms are ineffective for trace oil removal from oil-water mixture. Up to now, sorption technology has attracted increasing academic and industrial interests as one of the most efficient techniques, not only for the possibility of complete cleanup to oil, but also for the convenient post-treatment of oil-loaded sorbent with a solid or semisolid phase.

Various types of materials such as organic natural materials, inorganic mineral materials and synthetic organic polymers have been tested and investigated for oil removal (Sayed and Zayed, 2006; Srinivasan and Viraraghavan, 2008; Inagaki et al., 2000; Medeiros et al., 2009; Liu et al., 2011; Wu and Zhou, 2009). Among them, the treatment of water containing oil by organic natural materials is economical due to the low costs of natural renewable materials. The representative natural materials include kapok fiber (Huang and Lim, 2006), cotton fiber (Deschamps et al., 2003), wool fiber (Rajakovic et al., 2007), milkweed (Choi and Cloud, 1992), sawdust (Annunciado et al., 2005), etc., and these materials have been exploited for simple, effective and inexpensive treatment of spilled oil. Generally, the mechanism of oil sorption by fibers is mainly governed by fiber surface adsorption and capillary action of the voids (Choi and Moreau, 1993). In addition, fibers can be easily collected after the oil sorption for another reuse. Hence, along with the frequent appearance of oil pollution, this type of abundant and biodegradable natural fibers shows a good prospect of application in the treatment of oil pollution.

The oil sorption capability of natural fibers depends mainly on the surface void ratio and the surface composition, and in this case, esterification modification is usually used to increase the hydrophobicity of fibers to enhance the oil sorption capacity (Dankovich and Hsieh, 2007; Sun et al., 2003). By far, the modification of agricultural by-products has been reported for the purpose of oil cleanup operations in the aqueous environment. Banerjee et al. (2006) found that compared with raw sawdust, the oleic acid modified sawdust had a higher oil sorption capacity for crude oil and weathered oil. Furthermore, acetylated rice husks showed high oil absorbency and rapid oil uptake

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rate for crude oil spilled on aqueous media (Thompson et al., 2010), raw bagasse modified with fatty acid exhibited good affinity to oil (Said et al., 2009), and oleic acid-modified banana trunk fibers also demonstrated the best sorption capacity for engine oil and light oil fractions in various organic acids-modified banana trunk fibers (Sathasivam and Haris, 2010). Kapok fibers derived from silk-cotton tree is an effective natural sorbent, which show high oil sorption capacity due to the huge hollow lumen and hydrophobic-oleophilic surface. Recently, the research about the application of kapok fiber in the treatment of oil-containing wastewaters has attracted much attention (Lim and Huang, 2007a, 2007b; Abdullah et al., 2010; Rahmah and Abdullah, 2011; Wang et al., 2012). Although these studies on kapok fiber for oil absorption have been carried out, there is no information about the use of modified kapok fiber for the oil absorption in an aqueous environment.

Therefore, the aim of this work is to improve the oil sorption capacity of raw fiber in oil/water mixture by the acetylation, and demonstrate the role of the fiber interface for the oil retention capability of fiber assembly. Low viscosity diesel and high viscosity soybean oil were chosen as the model oils. The pseudo second-order model, and Langmuir and Freundlich models were used to fit the experimental data. In addition, Fourier transform infrared (FT-IR) and scanning electron microscope (SEM) were performed to investigate the reaction.

1 Materials and methods

1.1 Materials

Kapok was purchased from Shanghai Pan-Da Co. Ltd., China. Pyridine (analytical pure) was received from Tianjin Chemical Reagent Factory, China. N-bromosuccinimide (NBS, chemically pure) and dimethylformamide (DMF, analytical grade) were provided by Shanghai Chemical Reagent Factory, China. Acetic anhydride (analytical pure) was supplied by Tianjin Li-An Chemical Reagent Co. Ltd., China. Diesel and soybean oil came from local commercial market, Lanzhou, China.

1.2 Preparation of acetylated kapok fiber

Raw kapok fibers were ground into a short shape in a high-speed grinder. The resulting fibers were used in all the later reaction. Acetylation in pyridine-acetic anhydride system: kapok fibers (0.7 g) were placed in a round flask equipped with a mechanical stirrer, a reflux condenser and a thermometer, then a mixture of pyridine and acetic anhydride (\(V_{\text{acetic anhydride}} : V_{\text{pyridine}} = 0.6\)) was added. The oil bath was slowly heated to 110°C and the reaction was kept for 1 hr, then the product was filtered and washed with acetone and hot water to remove unreacted reagents, and then dried in an oven at 60°C to a constant weight. Acetylation in DMF-acetic anhydride catalyzed by NBS:kapok fibers (0.7 g) was placed in the flask with a mixture of DMF and acetic anhydride (\(V_{\text{acetic anhydride}} : V_{\text{DMF}} = 0.6\)) containing 0.08 g NBS. The reaction was carried out at 70°C for 1 hr, and the resulting product was washed and dried according to the above procedure. In this study, raw kapok fibers and two acetylated kapok fibers are denoted as RKF, PAKF (pyridine-catalyzed) and NAKF (NBS-catalyzed).

1.3 Measurements of oil sorption capacity

The diesel or soybean oil was mixed with 50 mL of water in a 100 mL conical flask for 10 min at 120 r/min over an orbital shaker. After agitation, the oil will float to the surface of the water and form the oil layer. Then, 0.06 g sorbent was added to the oil/water mixture. The concentrations of diesel and soybean oil used varied from 0.01 to 0.12 g/mL of water, which is in the range of the maximum oil sorption capacity of raw and acetylated fiber. The sorbent was left in the mixture and shaken for 30 min at 25°C. The sample was then removed from the flask using mesh screen and drained for 1 min. The oil sorption capacity (\(Q_{\text{oil/g-sorbent}}\)) of the sorbents was calculated according to Eq. (1):

\[
Q = \frac{M_{a} - M_{i} - M_{w}}{M_{i}}
\]

where, \(M_{a}\) (g) is the weight of the wet sorbents after draining, \(M_{i}\) (g) is the initial weight of sorbents and \(M_{w}\) (g) is the weight of water absorbed in the sorbents. Water content was determined by the method of extraction separation using n-hexane as the solvent. All tests were done in triplicate and an average value was used.

1.4 Characterizations

FT-IR spectra were recorded on a Nicolet NEXUS FT-IR spectrometer in the 4000–400 cm\(^{-1}\) region using KBr pellets. The micrographs of samples were examined using SEM (JSM-5600LV, JEOL). Before SEM observation, all samples were fixed on aluminum stubs and coated with gold.

2 Results and discussion

2.1 FT-IR spectra

FT-IR spectra of RKF, PAKF and NAKF are shown in Fig. 1. The bands at 1246 cm\(^{-1}\) and 1376 cm\(^{-1}\) are assigned to C–O and C–H bending vibration, respectively. The sharp and strong bands at 1598 cm\(^{-1}\) are assigned to carbon skeletal stretching of aromatic ring. The absorption peak at 1738 cm\(^{-1}\) originates from C=O stretching vibration of ketones, carboxylic groups and esters in lignin and acetyl ester groups in xylan (Lim and Huang, 2007a; Anirudhan and Sreekumar, 2011), the strong peak at 2910 cm\(^{-1}\) is due to asymmetric and symmetric stretching vibration in CH\(_2\) and CH\(_3\), the broad band around 3403 cm\(^{-1}\) is
assigned to O–H stretching vibration. By comparison, the apparent increase of absorption bands on PAKF and NAKF can be observed around 1742 cm\(^{-1}\) (stretching vibration of the carbonyl group C=O in ester), 1375 cm\(^{-1}\) (C–H bending vibration in \(-O(C=O)-CH\_3\) ), and 1244 cm\(^{-1}\) (C–O stretching vibration in ester). In addition, the intensity of absorption peak at 2910 cm\(^{-1}\) also increases, which gives evidence for the acetylation of RKF. However, there is no obvious decrease in the intensity of absorption peak assigned to O–H stretching vibration at 3403 cm\(^{-1}\) for PAKF and NAKF. This is due to the fact that the hydroxyl groups of RKF can not be reacted completely by controlling the reaction conditions to avoid the loss of intrinsic oil sorption capability of the fibers.

### 2.2 Morphology analyses

The SEM microphotographs of RKF, PAKF and NAKF are shown in Fig. 2. It is obvious that RKF has a hollow structure and smooth surface with nearly closed orifice, while PAKF and NAKF show a tiny groove on the fiber surface and an open lumen orifice also can be observed. This indicates that the smooth fiber surface that is not in favor of the adhesion of oil is obviously improved, and the rough surface being beneficial to the adhesion of oil is generated. In addition, the hollow lumen of PAKF and NAKF is still intact, which means that the acetylation does not cause severe collapse of the fibers lumen. These changes in the structure of fiber are favorable for the retention of oil in kapok assembly.

#### 2.3 Effect of contact time on sorption and kinetics studies

The effect of contact time on the oil sorption of RKF, PAKF and NAKF is shown in Fig. 3. It can be seen that the oil sorption capacity slightly increases with the contact time within the first 10 min followed by a relatively slow process for all the sorbents. This can be attributed to the fact that the oil on the outside surface of fibers can slowly penetrate into the hollow lumen. The results also show that the sorbents have a fast sorption property on the oils studied so that only a slight difference is observed between the initial and final sorption. This observation is consistent with the findings by Thompson et al. (2010). To understand the sorption process, the experimental data were further analyzed using pseudo second-order model, as represented in linear forms as Eq. (2) (Ho and McKay, 1999):

\[
t/Q_e = 1/(k \times Q_e^2) + t/Q_e
\]

where, \(Q_e\) (g/g) and \(Q_t\) (g/g) are the amount of oil adsorbed at equilibrium and time \(t\) (min), respectively, and \(k\) is the sorption rate constant.

In Fig. 4, a linear relationship is observed for all the samples, and the values of \(Q_e\), \(k\) and correlation coefficient \((R^2)\) are evaluated from the plot of the linear and presented in Table 1. The theoretical values of the pseudo second-order kinetic model, for diesel \((Q_{d,\text{cal}})\) and soybean oil \((Q_{s,\text{cal}})\), are in line with the experimental values, for diesel \((Q_{d,\text{exp}})\) and soybean oil \((Q_{s,\text{exp}})\), respectively, and all the values of the correlation coefficient are above 0.999. This indicates that the pseudo second-order kinetic model is suitable for depicting the sorption of diesel and soybean oil on the sorbents. Furthermore, acetylation of RKF catalyzed by both pyridine and NBS can enhance the equilibrium sorption capacity of RKF towards diesel and soybean oils. For the oil sorption of kapok assembly, intermolecular force and surface wax of fibers play an important role.

![Fig. 1](image1.png)  
**Fig. 1** FT-IR spectra of RKF (raw kapok fiber), PAKF (pyridine-catalyzed kapok fiber), and NAKF (NBS-catalyzed kapok fiber).

![Fig. 2](image2.png)  
**Fig. 2** SEM micrographs of RKF, PAKF, and NAKF.
Acetylation of RKF makes the more oil easier to be retained in kapok assembly owing to stronger intermolecular interactions between oil and acetyl groups on the fibers, which also facilitates the diffusion of oil through the successive fiber walls to the hollow lumen. Besides, the increase in surface roughness of RKF will also lead to a better locking-oil capability. Compared with the soybean oil, the improvement of PAKF and NAKF on the sorption of diesel is more significant. It is well known that the viscosity of soybean oil is higher than that of diesel, which means that the intermolecular force of soybean oil is much stronger. When soybean oil is absorbed into three types of sorbents studied, the liquid bridge with similar stability degree will be formed in any kind of fibers assembly. Thus, it can be concluded from the results that acetylated kapok fibers is more suitable for the sorption of low viscosity oil.

### 2.4 Effect of oil to water ratio

Figure 5 shows the sorption of oil as a function of oil/water ratio by RKF, PAKF and NAKF. As can be seen, there is an obvious difference between RKF and acetylated fibers. As the increase in oil concentration, the sorption capacity of all the sorbents increases until they reach equilibrium. During the sorption process, the floating oil was absorbed quickly into fibers and almost no dispersion of oil was observed once the sorbent was added in diesel/water or soybean oil/water mixture. The oil sorption capacity of the sorbents are in the following order: RKF (diesel 30.5 g/g, soybean oil 47.4 g/g) < NAKF (diesel 34 g/g, soybean oil 48.5 g/g) < PAKF (diesel 36.7 g/g, soybean oil 52.2 g/g).

<table>
<thead>
<tr>
<th>Sample</th>
<th>$Q_{d,exp}$ (g/g)</th>
<th>$Q_{s,exp}$ (g/g)</th>
<th>$Q_{d,cal}$ (g/g)</th>
<th>$k_d$ (min$^{-1}$)</th>
<th>$R^2$</th>
<th>$Q_{s,cal}$ (g/g)</th>
<th>$k_s$ (min$^{-1}$)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>RKF</td>
<td>29.3</td>
<td>48.2</td>
<td>28.4</td>
<td>0.1285</td>
<td>0.9996</td>
<td>47.2</td>
<td>0.0672</td>
<td>0.9996</td>
</tr>
<tr>
<td>PAKF</td>
<td>35.9</td>
<td>53.9</td>
<td>35.0</td>
<td>0.1437</td>
<td>0.9996</td>
<td>51.8</td>
<td>0.0627</td>
<td>0.9998</td>
</tr>
<tr>
<td>NAKF</td>
<td>34.1</td>
<td>49.8</td>
<td>33.6</td>
<td>0.1246</td>
<td>0.9996</td>
<td>48.8</td>
<td>0.0588</td>
<td>0.9998</td>
</tr>
</tbody>
</table>

Table 1: Sorption parameters of pseudo second-order rate models.
Table 2 Oil sorption capacities of RKF, PAKF and NAKF from this study and other sorbents

<table>
<thead>
<tr>
<th>Sorbent Type of oil</th>
<th>Oil sorption capacity (g/g)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw cotton fiber</td>
<td>Vegetable oil</td>
<td>30</td>
</tr>
<tr>
<td>Acetylated sugarcane bagasse</td>
<td>Machine oil</td>
<td>11–21</td>
</tr>
<tr>
<td>Vermiculite modified with carbon nanotubes and nanofibers</td>
<td>Diesel</td>
<td>3.5</td>
</tr>
<tr>
<td>Walnut shell</td>
<td>Standard mineral oil</td>
<td>0.30</td>
</tr>
<tr>
<td>RKF</td>
<td>Diesel</td>
<td>30.5</td>
</tr>
<tr>
<td>PAKF</td>
<td>Diesel</td>
<td>36.7</td>
</tr>
<tr>
<td>NAKF</td>
<td>Diesel</td>
<td>34</td>
</tr>
</tbody>
</table>

Fig. 5 Sorption of oil as a function of oil concentration by RKF, PAKF and NAKF.

In addition, the nonpolar nature of acetyl groups renders the surface of PAKF and NAKF hydrophobic. As a result, a great deal of oil is preferentially absorbed into the sorbents with slight amounts of water held in the fibers. In addition, as shown in Table 2, the acetylated fibers exhibit higher oil sorption capacity than other traditional oil sorbents, which illustrates that acetylated fiber used to the cleanup of oil on water surface is very effective, and the result is encouraging.

2.5 Sorption isotherms

It is very practical for equilibrium adsorption isotherm studies which can provide the adsorption capacity of an adsorbent toward adsorbate and examine the effectiveness of an adsorbent. The prediction of adsorption parameters and comparison of adsorption characteristics for various adsorbents will be obtained from the equilibrium adsorption isotherm models. In this part, the sorption data were analyzed by the two most widely used adsorption isotherm models, namely Langmuir and Freundlich. Langmuir isotherm assumes that only one mono-layer of adsorbate molecule can be adsorbed on a surface with no transmigration of adsorbate in the plane of surface. The linear form of Langmuir isotherm is given as Eq. (3) (Langmuir, 1918):

$$\frac{C_e}{Q_e} = \frac{1}{Q_m} + \frac{C_e}{Q_m}$$  \hspace{1cm} (3)

where, $C_e$ (g/mL) is the equilibrium concentration of the adsorbate, $Q_e$ (g/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, $b$ is the Langmuir coefficient related to the affinity between the adsorbent and adsorbate and $Q_m$ (g/g) is the maximum monolayer adsorption capacity.

The essential characteristics of the Langmuir isotherm is expressed in the light of a dimensionless constant separation factor ($R_L$), defined as Eq. (4) (Hall et al., 1966; Sathishkumar et al., 2008):

$$R_L = \frac{1}{1 + C_i}$$  \hspace{1cm} (4)

where, $C_i$ (g/mL) is range of adsorbent concentration. The value of $R_L$ is an indicator of the type of the isotherm to be either favorable ($0 < R_L < 1$), unfavourable ($R_L > 1$), linear ($R_L = 1$), or irreversible ($R_L = 0$).

Freundlich isotherm model assumes heterogeneous surface energies, and a heterogeneous adsorption surface that has unequal available sites with different energies of adsorption. This model is effective in describing non-ideal adsorption onto heterogeneous sites of adsorbent involving multilayer adsorption. The Freundlich isotherm equation was represented by Eq. (5) (Freundlich, 1906):

$$\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e$$  \hspace{1cm} (5)

where, $K_F$ is the Freundlich constant related to the adsorption capacity, the slope of $1/n$ ranging between 0 and 1 is a indicator of adsorption intensity and surface heterogeneity, which is more heterogeneous as its value gets closer to 0.
### Table 3 Parameters of the Langmuir and Freundlich models and correlation coefficients for sorption of diesel and soybean oil

<table>
<thead>
<tr>
<th></th>
<th>Diesel</th>
<th></th>
<th></th>
<th>Soya bean oil</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Langmuir</td>
<td></td>
<td></td>
<td>Freundlich</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$Q_m$ (g/g)</td>
<td>$b$ (mL/g)</td>
<td>$R_L$</td>
<td>$K_L$ (mL/g)</td>
<td>$R^2$</td>
<td>$1/n$</td>
</tr>
<tr>
<td>RKF</td>
<td>37.9</td>
<td>0.040</td>
<td>0.9769</td>
<td>0.1724-0.7142</td>
<td>106.17</td>
<td>0.9818</td>
</tr>
<tr>
<td>PAKF</td>
<td>55.5</td>
<td>0.020</td>
<td>0.9514</td>
<td>0.2941-0.8333</td>
<td>223.82</td>
<td>0.9878</td>
</tr>
<tr>
<td>NAKF</td>
<td>47.4</td>
<td>0.027</td>
<td>0.9612</td>
<td>0.2588-0.7474</td>
<td>148.49</td>
<td>0.9854</td>
</tr>
<tr>
<td></td>
<td>Langmuir</td>
<td></td>
<td></td>
<td>Freundlich</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$Q_m$ (g/g)</td>
<td>$b$ (mL/g)</td>
<td>$R_L$</td>
<td>$K_L$ (mL/g)</td>
<td>$R^2$</td>
<td>$1/n$</td>
</tr>
<tr>
<td>RKF</td>
<td>79.0</td>
<td>0.013</td>
<td>0.9594</td>
<td>0.3906-0.8805</td>
<td>261.64</td>
<td>0.7285</td>
</tr>
<tr>
<td>PAKF</td>
<td>107.3</td>
<td>0.008</td>
<td>0.9333</td>
<td>0.5102-0.9299</td>
<td>454.67</td>
<td>0.8770</td>
</tr>
<tr>
<td>NAKF</td>
<td>97.4</td>
<td>0.009</td>
<td>0.9455</td>
<td>0.4808-0.9174</td>
<td>310.10</td>
<td>0.7825</td>
</tr>
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By the linear regression of RKF, NAKF and PAKF on the diesel and soybean oil adsorption equilibrium data, the corresponding Langmuir and Freundlich model, isotherm constants, correlation coefficients ($R^2$) are listed in Fig. 6 and Table 3. The regression correlation coefficient $R^2$ values of the Freundlich model are much closer to 1 than those of the Langmuir model. Furthermore, the difference between calculated $Q_m$ values from the Langmuir equation and experimentally determined values is very large. This indicates that the adsorption data of diesel and soybean oil onto RKF, PAKF and NAKF can be better described by the Freundlich model. Meanwhile, no matter diesel and soybean oil, the values of $R_L$ and $1/n$ are in the range of 0–1, meaning that the sorption process is favourable. For the diesel, the values of $1/n$ for PAKF and NAKF are higher than that of RKF, which imply that the acetylation of hydroxyl groups in fibers matrix will be propitious to the uniform distribution of diesel in kapok assembly (Ajmal et al., 1998). However, for the soybean oil, the gap among the values of $1/n$ for the three sorbents is not so prominent. This is because the viscosity of soybean oil is high so that it can be better adhered to the surface of the sorbents. Therefore, the acetylation of the RKF surface has little influence on the distribution homogeneity of oil in the kapok assembly. Despite this, the as-prepared PAKF and NAKF exhibit higher oil sorption capacity than RKF, implying that acetylated fibers is an excellent biomaterial for the removal of oil from aqueous surroundings.

### 3 Conclusions

The present study shows that acetylated kapok fibers had a higher oil sorption capability than raw fibers in oil/water mixture. FT-IR and SEM characterization were
used to investigate the surface morphology and chemical compositions of acetylated fibers. The acetylation of kapok fibers can enhance the equilibrium sorption capacity of the fibers towards the model oils, especially for the diesel with low viscosity. The relevant kinetics data can be fitted by pseudo second-order model. The determination of the sorption isotherm values indicates that the experimental data can be well described by the Freundlich model than the Langmuir model, by which the sorption procedure was multilayer and favorable. Based on these results, it is not doubtable that the modified fibers could be used for the cleanup of oil spilled on aquatic environments.

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References


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