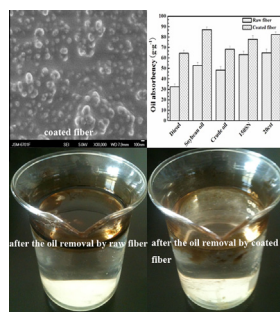


Investigation of oil sorption capability of PBMA/SiO₂ coated kapok fiberJintao Wang^{a,b}, Yian Zheng^{a,b}, Yuru Kang^a, Aiqin Wang^{a,*}^a Center of Eco-Material and Green Chemistry, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, PR China^b Graduate University of the Chinese Academy of Sciences, Beijing 100049, PR China

HIGHLIGHTS

- PBMA/SiO₂ coated kapok fiber was prepared and used for oil sorption.
- The formation of rough surface of coated kapok fiber was confirmed by SEM.
- Coated fiber exhibits higher oil sorption capacity than raw fiber in oil/water mixture.
- Coated fiber exhibits good reusability and absorbed oil can be easily recovered.

GRAPHICAL ABSTRACT



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ABSTRACT

A new kind of oil sorbent based on kapok fiber was prepared by the coating of the mixture of polybutyl-methacrylate (PBMA) and hydrophobic silica (SiO₂) on fiber surface. The formation of new surface was confirmed by Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). The sorption capacities of PBMA/SiO₂ coated fiber in pure oil and oil/water mixture, oil uptake rate and reusability were investigated. As-prepared fiber exhibits higher oil sorption capacity than raw fiber in oil/water mixture, with the findings that the growth rate of oil sorption capacity for diesel, soybean oil, crude oil, 150SN, and 20cst reaches approximately 99.7%, 65.0%, 41.1%, 23.1% and 26.8%, respectively. Coated fiber also has excellent reusability, fast oil uptake rate and high buoyancy in the removal of oil on artificial seawater surface. This study may provide a facile method for the preparation of oil sorbent with excellent properties for the removal of oils on water surface.

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1. Introduction

In recent years, oil spill contamination has become one of major problem of environmental pollution [1]. Recovering oil by sorbents is a very promising method and has gotten great attention due to their high clean-up efficiency. The floating oil can be transformed from a liquid to a semi-solid or to the solid phase, which can then be removed from the spilled area by mechanical way. Frequently-used oil sorbents can be divided into two categories: synthetic fibers and natural fibers. Many synthetic fibers including polypropylene (PP) [2,3], butyl methacrylate–lauryl methacrylate copolymeric fiber [4], polystyrene fiber [5,6], etc. have been used

to absorb oil owing to their excellent buoyancy, good hydrophobic property, and relatively low cost. However, the shortcomings such as low oil sorption capacity and poor biodegradability limit their wider application. Due to excellent buoyancy, low water uptake, and good hydrophobicity–oleophilicity property, natural fibers have also attracted much attention as the oil sorbents, such as kapok [7], wool [8], cotton [9], silkworm cocoon [10], rice husks [11] and oil palm leaves [12]. Most of natural fibers like kapok fiber have higher oil sorption capacity than synthetic fibers [13]. More importantly, the oil sorption capacity of natural fibers can be further enhanced by simple hydrophobic modification [14,15].

A hydrophobic–oleophilic surface is necessary for a good oil sorbent. Oil sorbent surface with low surface energy can be easily wetted by oil. To improve the oil sorption capability of sorbent, hydrophobic–oleophilic function groups can be introduced into

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the surface of materials, which will enable oil to adhere to the surface and interstice of oil sorbents better. Lately, superhydrophobic and superoleophilic materials with high surface area, high porosity, and nano-structured surface have been prepared and used for oil sorption [5,6,16]. Thereinto, modifying smooth surface into rough surface with the micro- or nanostructures is very efficient for enhancing the oil sorption capacity and hydrophobic property of a material. For instance, carbon nanotubes and nanofibers were deposited on the surface of expanded vermiculite, the obtained vermiculite showed excellent hydrophobic characteristics and improved oil sorption capacity [17]. Undoubtedly, generating a surface structure with nanometer range roughness and low surface energy is a simple, feasible and economic way to get oil sorbents with advantageous properties.

The aim of this work was to develop a low-cost oil sorbent that can be practically applied in the control and recovery of oil spilled on water surface. Kapok fiber is a type of natural fiber that possesses excellent oil sorption capability because of unique hollow lumen and hydrophobic plant wax on fiber surface [18–21]. In this study, a new type of oil sorbent based on kapok fiber was prepared by the fabrication of nanometer sized roughness on fiber surface using polybutylmethacrylate (PBMA) with low surface energy and hydrophobic modified silica as coating material. The as-prepared fiber was used to selectively absorb various oils, and interestingly the coated fiber exhibited high oil sorption capacities for studied oils especially for high viscosity oils in oil/water mixture, which is higher than that of previously reported superhydrophobic kapok fiber by our research group [16]. The findings of this study also provide a simple method for the fabrication of other natural materials as oil sorbent for cleanup of spilled oil on water surface.

2. Experimental

2.1. Materials

Kapok fiber was purchased from Shanghai Pan–Da Co., Ltd. China. Butyl methacrylate (chemically pure) was received from Shanghai Chemical Reagent Factory, China. Benzoyl peroxide (BPO, analytical grade) was provided by Beijing Jin-Long Chemical Reagent Co., Ltd. China. Tetraethylorthosilicate (TEOS, chemically pure) was supplied by Tianjin Chemical Reagent Factory, China. Dodecyltrimethoxysilane (DTMS, chemically pure) was provided by Sinopharm Chemical Reagent Co., Ltd. China. $\text{NH}_3 \cdot \text{H}_2\text{O}$ (analytical grade) was obtained Baiyin Chemical Reagent Factory, China. Ethanol (analytical grade) was supplied by Tianjin Li-An Chemical Reagent Co., Ltd. China. Gasoline, diesel and soybean oil came from market, Lanzhou, China. Crude oil, 150SN and 20cst (lube base oil) was supplied by PetroChina Lanzhou Lubricating Oil R&D Institute, China. The physical properties of six kinds of oils are displayed in Table 1.

2.2. Preparation process

Preparation of hydrophobic silica particles. TEOS (12 g) was added to 200 mL of ethanol, stirred at room temperature for 1 h. Then, 10 mL of $\text{NH}_3 \cdot \text{H}_2\text{O}$ was added into the system and continue

to stir for 4 h. Afterwards, the mixture was aged at room temperature for 12 h, DTMS (4 mL) was added into the solution, stirred and hydrolyzed for 1 h. Finally, the modified silica nanoparticles were collected by centrifugation, and then dried under vacuum at 60 °C for use.

Preparation of coating solution. The mixture of butyl methacrylate (6 g) and BPO (0.06 g) was put into a four-necked flask, and the reaction system was slowly heated to 80 °C in an oil bath and kept for 4 h. After that, 200 mL of tetrahydrofuran was added into the flask and stirred for 30 min at 60 °C to form polymer solution. Finally, the as-prepared silica nanoparticles (6 g) were ultrasonically dispersed in polymer solution at ambient temperature.

Treatment of raw fiber. Raw fiber was immersed into the above solution for 30 min, and the resulting fiber was filtered, dried to constant weight at 80 °C.

2.3. Measurements of oil sorption capacity

0.1 g of dried sample was placed in a stainless-steel mesh weighed beforehand and immersed in oil at room temperature. The mesh was taken out from the oil together after 15 min, drained for 1 min, and wiped with filter paper to remove excess oil from the bottom of the mesh. The oil sorption capacity of the sample was determined by weighing the samples before and after the absorption, and calculated by the following formula:

$$Q = (W_t - W_i - W_w) / W_i$$

where Q is the oil sorption capacity of the sorbents calculated as grams of oil per gram of sample, W_t is the weight of the wet sorbents after draining (g), W_i is the initial weight of sorbents (g) and W_w is the weight of water absorbed in the sorbents (g). In pure oil medium without any water, W_w is equal to zero.

In oil/water system: The oil (10 g) was mixed with 60 mL of artificial seawater (3.5 wt% NaCl) in a 100 mL conical flask for 10 min at 150 rpm over an orbital shaker. The shaking can make the oil float to the surface of the artificial seawater forming the oil layer. Then, 0.1 g of oil sorbent was added to the oil/water mixture. The sorbent was left in the oil/water mixture and shaken for 1 h at 30 °C. After that, the sample was removed from the flask using mesh screen, drained for 1 min and weighed. Water content was determined by the method of extraction separation using n -hexane as the solvent [16].

2.4. Reusability

Oil absorbing fiber from the water surface was removed by the help of a mesh screen, then it was placed on a sand core funnel and drained under vacuum for 10 min before weighing. The sorption/desorption cycle was repeated for six cycles to evaluate the reusability of the fiber.

2.5. Characterizations

Fourier transform infrared (FTIR) spectra were recorded on a Nicolet NEXUS FTIR spectrometer 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.

3. Results and discussion

3.1. Formation of fiber surface with nanometric roughness and oil sorption mechanism

The fabrication mechanism of PBMA/SiO₂ kapok fiber with rough surface and its oil sorption mechanism is displayed in Fig. 1. The

Table 1
Characteristics of investigated oils at room temperature.

Oils	Viscosity (mm^2/s)	Density (g/mL)
Gasoline	0.61	0.72
Diesel	4.60	0.85
Soybean oil	8.36	0.92
Crude oil	7.39	0.85
150SN	33.12	0.84
20cst	122.50	0.84

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