Preparation and Properties of Kapok Fiber Enhanced Oil Sorption Resins by Suspended Emulsion Polymerization

Jintao Wang,[1,2] Yian Zheng,[1,2] Aiqin Wang[1]

[1]Center of Eco-Material and Green Chemistry, Lanzhou Institute of Chemical Physics,
Chinese Academy of Sciences, Lanzhou 730000, People's Republic of China
[2]Graduate University of the Chinese Academy of Sciences, Beijing 100049, People's Republic of China

Correspondence to: A. Wang (E-mail: aqwang@licp.cas.cn)

ABSTRACT: In this work, a series of polybutylmethacrylate/kapok fiber (PBMA/KF) composites were synthesized by suspended emulsion polymerization and well characterized by means of Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). The effects of reaction parameters, such as amount of initiator, crosslinker, emulsifier, KF content on oil absorbency, were investigated in detail. The optimum polymerization conditions were obtained as initiator of 0.4 wt %, crosslinker of 1.0 wt %, emulsifier of 2.0 wt %, and KF content of 8.0 wt %. Compared with PBMA, the as-prepared PBMA/KF (8 wt % KF) showed better oil sorption capacity, with the increase percentage of 58.7% in toluene and 66.7% in chloroform. Swelling behavior indicated that the sorption equilibrium was established basically within 15 min, and the incorporation of KF can promote the oil sorption rate. Furthermore, the composite exhibited better reusability and oil retention capabilities, and can be recognized as a kind of low-cost oil sorption materials for oil pollution treatment. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

INTRODUCTION

It is well known that oil pollution from petroleum industry, marine oil transportation, and industrial effluents has posed a great threat to human economic actions and ecological environment.[1,2] Various materials, including natural absorbents and synthesized polymeric materials, have been developed to deal with oil pollution hazards and recover the spilled oils.[3–9] High-efficient oil absorptive materials are required to possess desirable characteristics, such as high oil sorption capacity, fast oil sorption rate, favorable sorption selectivity of oil over water, readily recycling possibility, and lower density compared with water.[10–12] Alkylacrylate and olefins have been universally used to synthesize oil absorbing polymers due to their good affinity for nonpolar solvents.[13–17] However, up to now, most of oil absorption resins are still synthesized by suspension polymerization, and the resulting resins have low oil absorption capacity and slow oil absorption rate. To reach oil absorption equilibrium, several hours or even longer time is needed, which is far below the needs for the treatment of oil pollution. Therefore, it is important and urgent to develop new techniques to improve the properties of an oil sorption resin.

Suspended emulsion polymerization is an effective polymerization method derived from suspension and emulsion polymerization, by which a series of porous polymers can be obtained.[18,19] Because suspended emulsion polymerization process was proposed formally, this technology has gained increasing attentions of many scholars. In suspended emulsion polymerization, organic solvent is generally used to diffuse into the network of polymers to form porous structure, which is beneficial to the enhancement of absorption rate. The particles formation mechanism, particle features and kinetics of polyvinyl chloride and polymethylmethacrylate resins prepared by suspended emulsion polymerization had been studied.[20,21] Porous acrylonitrile/itaconic acid copolymer was also successfully synthesized for the first time.[22] The high oil absorption resin with short-chain acrylic ester as monomer was synthesized and its oil absorbing behavior was superior to that of the resins reported in literatures.[23] In addition, several functional adsorbent materials were prepared using this method and the resulting porous polymers exhibited excellent adsorption capacities for Hg^{2+}, Ag^+, and Cu^{2+}.[24,25]

Crosslinking is responsible for the formation of a three-dimensional network structure, which can restrict the expansion of

polymeric chains and enhance the gel elasticity of oil absorbing resin. So, the change of crosslinking structure can affect the swelling behavior of oil absorbing resin. Long polymer chain, inorganic material, and other filling materials have been introduced into oil absorption resin to enhance the oil sorption capacity.[26–28] For instance, polybutadiene as a physical-filling crosslinker was introduced into oil absorption resin to form a type of relaxing three-dimensional network with improved swelling capability.[26] Adding paraffin into oil absorption resin was also beneficial to enhance the oil absorbency of oil absorption resin because paraffin can increase the distance of crosslink points and weaken the bondage of chemical crosslinking on the extension of polymeric chains.[27] Kapok fiber (KF), a fiber derived from the fruits of silk-cotton tree, is mainly composed of cellulose, lignin and polysaccharide. Due to its distinct hollow structure and hydrophobic characteristics, KF exhibits high oil absorbing capacity.[29–31] In addition, it is reported that KF suffered from the alkali treatment can be used as composite reinforcement material. Mechanical interlocking and new chemical bond formed by the grafting of polymer chains on the hydroxyl groups of KF are responsible for the composite effect.[32]

In this study, a series of PBMA (polybutylmethacrylate)/KF composites were synthesized using suspended emulsion polymerization, wherein the incorporation of KF can form oil penetrating channel in oil sorption resin and conduce to enlarge the space of a three-dimensional network. It is speculated that the oil absorbency would be improved by introducing KF into the resin polymerized with single monomer. The effects of synthetic variables (amount of initiator, amount of crosslinker, amount of emulsifier, and content of KF) on the oil absorbency were determined. The swelling behavior and kinetics for toluene and chloroform were evaluated.

EXPERIMENTAL

Materials

Butylmethacrylate (BMA; chemically pure, from Shanghai Chemical Reagent Factory, China) and ethylene glycol dimethacrylate (EGDMA; chemically pure, from Tokyo Chemical Industry, Japan) was purified by washing with 5% aqueous sodium hydroxide (analytical grade, from Tianjing Li-An Chemical Reagent, China), and then dried by anhydrous $CaCl_2$ (analytical grade, from Tianjin Chemical Reagent, China). $NaClO_2$ (chemically pure), ammonium persulfate (APS) (analytical grade), acetic acid (analytical grade), toluene (analytical grade), and sodium dodecyl sulfate (SDS) (chemically pure) were received from Beijin Hua-Wei Chemical Reagent, China, Tianjin Chemical Reagent Factory, China, Tianjin Li-An Chemical Reagent, China, Tianjin Xin-Yue Chemical, and Shanghai Bai-He Factory, China, respectively. Cyclohexane and chloroform (analytical grade) were provided by Shanghai Chemical Reagent Factory, China. KF was purchased from Shanghai Pan-Da, China.

KF Pretreatment Process

The KF (8 g) was added into 400 mL of $NaClO_2$ solution (1 wt %) adjusting pH to 4.5 with acetic acid and treated for 1 h at 80 °C. Afterward, the sample was washed continually with distilled water to remove any waxy residue. The resulting fiber was dried at 60 °C for 6 h under vacuum condition, ground, screened through a 20-mesh sieve and put in a desiccator for further use.[33]

Preparation of PBMA/KF Oil Absorbing Composite

The treated KF (0.64 g) was placed into cyclohexane (12.8 mL) in a four-necked flask equipped with a mechanical stirrer, a reflux condenser, a thermometer, and a nitrogen line. After being purged with nitrogen for 20 min to remove the oxygen from the system, a mixture of BMA (8 g) and EGDMA (0.08 g) was introduced. Afterward, the reaction flask was slowly heated to 80 °C in an oil bath, while a solution (3.2 mL) containing SDS (0.16 g) and APS (0.032 g) was added. The reaction system was kept for 3 h. A nitrogen atmosphere was maintained throughout the reaction process. After polymerization was completed, the obtained resin was poured and washed several times with ethanol and then dried in an oven at 60 °C to constant weight.

Preparation procedure of PBMA is similar to that described above except for the addition of KF.

Measurements of Oil Absorbency

The test procedure of oil absorbency was similar to that reported by Zhou.[11] The dried sample (0.15 g) was put into a stainless steel mesh weighed beforehand and immersed in toluene and chloroform at room temperature. The sample and the mesh were picked up from the oil together after given time periods, drained for 10 s, and wiped with filter paper to remove excess oil from the bottom of the mesh. The oil absorbency of the sample was determined by weighing the samples before and after the sorption and calculated by the following formula:

$$Q = (m_2 - m_1)/m_1$$

where Q is oil absorbency calculated as grams of oil per gram of sample, m_1 and m_2 are the weights of sample before and after oil sorption, respectively. All oil absorbency was measured three times, and an average value was used.

The swelling kinetics of samples was studied by repeating the previous measurements at different time intervals.

Reswelling Capability

The dried sample (0.15 g) in a stainless steel mesh was immersed in toluene or chloroform at room temperature to reach swelling equilibrium. Then the swollen samples were placed in an oven at 60 °C for 10 h to deswell thoroughly. The procedure was repeated and the saturated oil absorbency of the sample after six reswelling cycles was obtained.

Characterizations

Fourier transform infrared (FTIR) spectra were recorded on a Nicolet NEXUS FTIR spectrometer using KBr pellets. The micrographs of samples were examined using scanning electron microscopy (SEM; JSM-5600LV, JEOL). Before SEM observation, all samples were fixed on aluminum stubs and coated with gold.
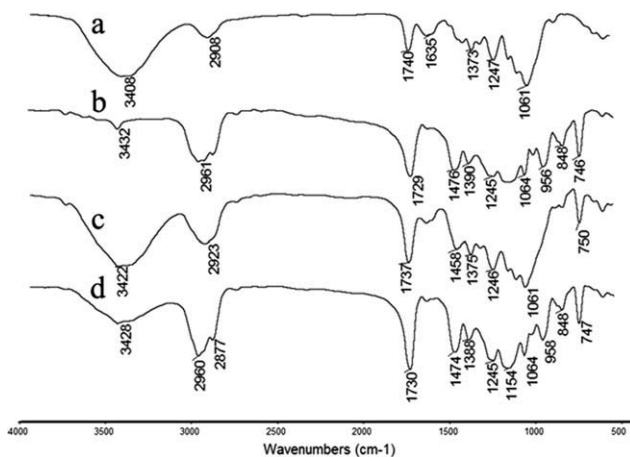
Figure 1. FTIR spectra of (a) KF, (b) PBMA resin, (c) physical mixture of treated KF and PBMA, and (d) PBMA/KF composite.

RESULTS AND DISCUSSION

FTIR Spectra

The FTIR spectra of KF, PBMA, the mixture of treated KF and BMA, and PBMA/KF composite are given in Figure 1. In Figure 1(a), the characteristic peaks of KF around 3408 cm^{-1} (O—H stretching vibration), 2908 cm^{-1} (C—H stretching vibration), 1740 cm^{-1} (C=O stretching vibration), and 1061 cm^{-1} (C—O stretching vibration) can be observed.[29,31] In Figure 1(b), the bands at 2961 cm^{-1} (C—H stretching vibration), 1729 cm^{-1} (C=O stretching vibration in ester groups), 1476 cm^{-1} and 1390 cm^{-1} (C—H bending vibration in CH$_2$ and CH$_3$), and 746 cm^{-1} (out-of-plane deformation vibration of —(CH$_2$)$_n$—, $n > 4$) are assigned to characteristic absorption bands of PBMA. Figure 1(c) indicates that the spectrum of the mixture holds the primary characteristics peaks of KF and PBMA. However, after the KF is incorporated into PBMA resin, the O—H stretching vibration around 3428 cm^{-1} in Figure 1(d) is not so remarkable compared with that of physical mixture of KF and PBMA, an indication that the hydroxyl groups of KF also participate in the reaction by grafting (Scheme 1). Besides, the band around 1064 cm^{-1} (C—O stretching vibration of cellulose) is not obvious in PBMA/KF composite and the intensity of absorption peak at 1154 cm^{-1} increases significantly, as a result of the superposition effect of distinguishable peaks and interfering action of peaks in the range of 1050–1160 cm^{-1}. The obtained information demonstrates that there involves the generation of new chemical bands, meaning that within the forming three-dimensional network, KF is not only a physical filler, but has bound to the network.

Morphology Analysis

The effect of KF on the surface morphologies of PBMA resin was observed with SEM, as shown in Figure 2. PBMA resin displays an undulant and incompact structure, with a large number of micropores on the surface, whereas PBMA/KF composite shows the same surface except for some embedded and dispersed KF hollow lumen in the polymer matrix. The porous surface and loose internal structure will lead to an increase of the surface area of the resin, which facilitates the diffusion of oil into the polymeric network of the resin and provides the final resin with high oil absorbency and rapid oil sorption rate. From the surface of PBMA/KF composite, the open KF hollow lumen still can be observed, implying the lumen was not filled with PBMA polymer and some channels with bigger diameter formed by interlaced lumen will be present in the net structure.

Effect of the Amount of Initiator

As shown in Figure 3, the oil absorbency of the composite in either toluene or chloroform increases with the amount of initiator increasing from 0.15% to 0.4% or 0.65% but decreases rapidly with further increase in the amount of initiator. It is known that the amount of initiator has important influence on the size of molecular weight of polymeric chains and effective network dimension of synthetic resin.[9,13] When the amount of initiator is too low, the free radical generated is insufficient to initiate more active sites on monomer molecule which restricts the process of the chain transfer reaction and affects the growth of polymer chains. Besides, more monomer cannot be initiated and left in the three-dimensional network of the composite, and accordingly, the probability of an effective crosslinking was reduced among polymeric chains. As a result, the oil absorbency of the composite was improved with increase in initiator amount below the optimal value. Nevertheless, an excess of initiator is also unfavorable for oil sorption for that more initiator can accelerate the termination reaction of free radicals, decrease the chain length between crosslinking points of the network, and enhance the crosslinking density of the oil sorption composite.
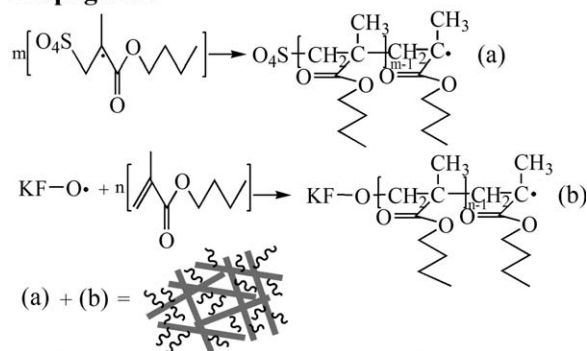
Effect of the Amount of Crosslinker

As shown in Figure 4, the amount of crosslinker displays obvious effects on the oil absorbency. It can be seen that the oil absorbency first increases slightly and then sharply decreases with increase in the amount of crosslinker from 1% to 4%. The swelling capacity of oil sorption resin is mainly affected by
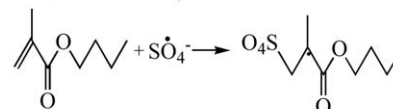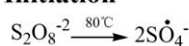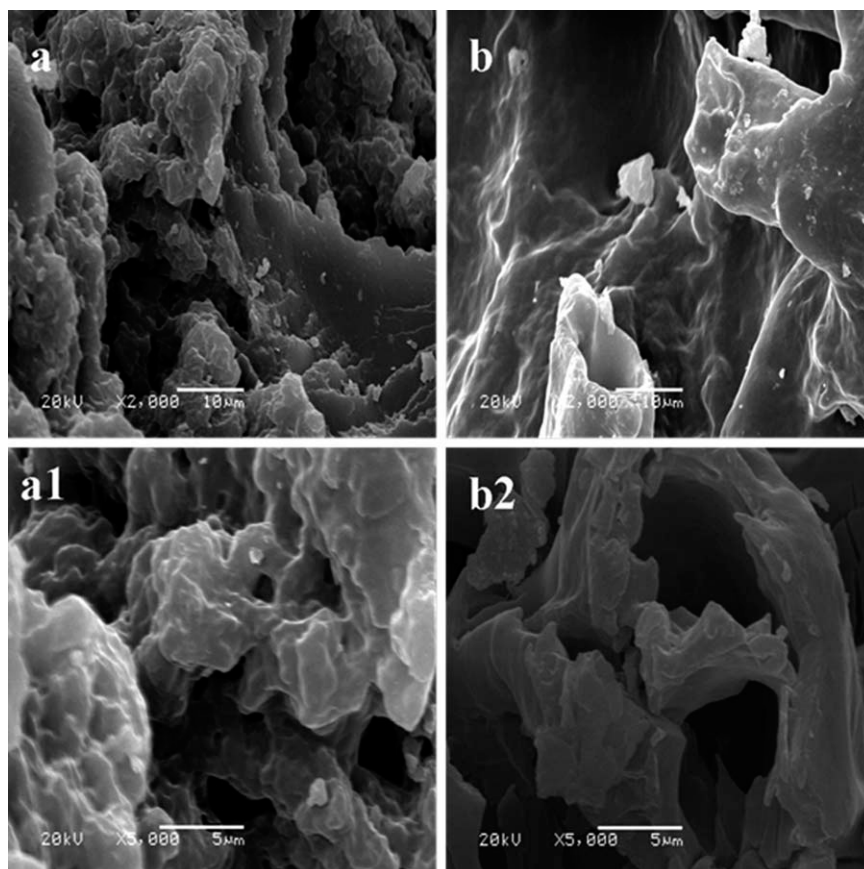

**Scheme 1.** Formation of PBMA chains on the KF surface initiated by APS.

Figure 2. SEM micrographs of (a and a1) PBMA resin and (b and b1) PBMA/KF composite.

rubber elasticity, the affinity to oil and crosslinking density.[34] An appropriate crosslinking among polymeric chains can generate a continuous three-dimensional network that facilitates the penetration of oil in the resin. On the contrary, an increase in the amount of the crosslinker results in the formation of more crosslink points and the reduction of average molecular of polymeric chains,[35] by which the crosslinking density increases and

the relaxation of three-dimensional network is more limited. As a result, the network space for holding oil was minimized and the oil absorbency of composites decreased. In addition, the composite with a too low crosslinking density cannot obtain a good oil sorption capability. This is ascribed to the fact that more soluble fraction exists in the composite with the low crosslinking, which provides the collapse of network of the
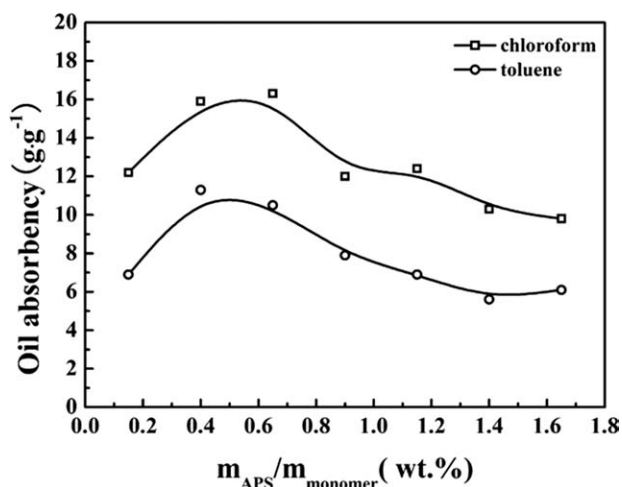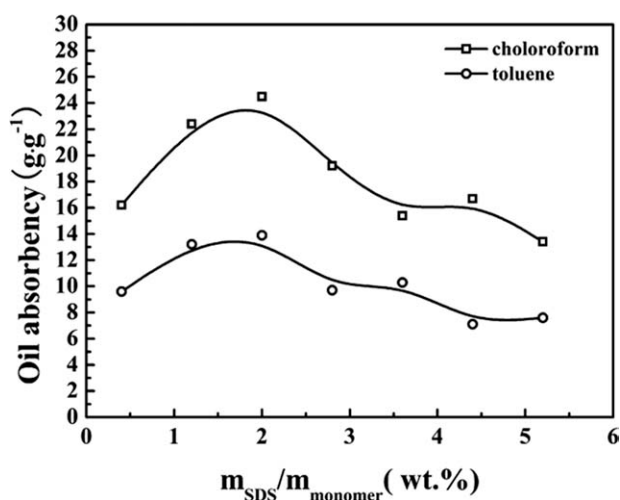

Figure 3. Effect of initiator content on oil absorbency: KF content, 4.0 wt %; crosslinker content, 1.6 wt %; emulsifier content, 1.2 wt %.
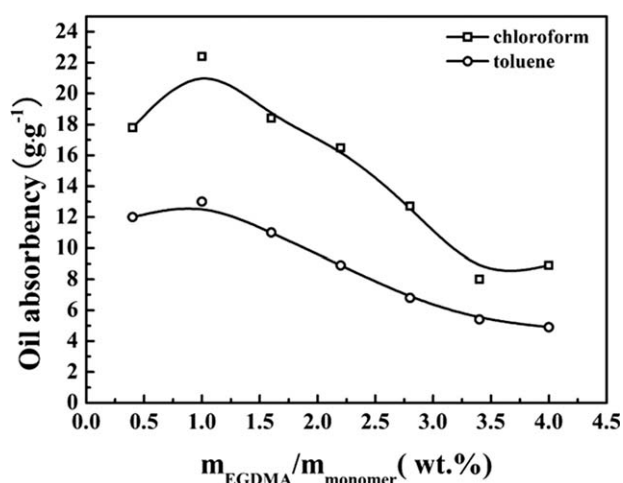
Figure 4. Effect of crosslinker content on oil absorbency: KF content, 4.0 wt %; initiator content, 0.4 wt %; emulsifier content, 1.2 wt %.

Figure 5. Effect of emulsifier content on oil absorbency: KF content, 4.0 wt %; initiator content, 0.4 wt %; crosslinker content, 1.0 wt %.

composite with greater probability during the swelling experiment. Accordingly, the oil absorbency of the composite was decreased.

Effect of the Amount of Emulsifier

In suspended emulsion polymerization, emulsifier is used to reduce interface tension, emulsify and disperse monomer. During this process, emulsifier will be present at not only the polymer particle/water interface but also the monomer/water interface. As shown in Figure 5, the oil absorbency of oil sorption composite increases with the amount of SDS increasing from 0.4% to 2% beyond which it decreases. This is because fewer SDS is insufficient to disperse monomer and stabilize primary particle, the smaller particles are inclined to form compact large particles, which is unfavorable to the oil sorption. Increasing the amount of SDS will obtain a more homogeneous distribution of particles which aggregate to form the smaller clusters and then come together into large agglomerates with abundant pore volume, resulting in the high oil absorbency. But the massive amount of SDS can enhance dramatically the polymerization rate of monomer owning to the increase of the initiator decomposition rate,[18,19] making the formed polymer cannot be effectively crosslinked among the polymeric chains in the limited time. Moreover, such hydrophilic emulsifier enriched in composites also reduces the affinity of oil to the oleophilic network. As a result, the dissolution of fractional network in the oil resulted in low oil absorbency.

Effect of the Content of KF

The effect of the content of KF on the oil absorbency is shown in Figure 6. When 8 wt % KF is incorporated into PBMA resin, the oil absorbency is improved remarkably, and subsequent increase up to 20 wt % would lead to a decrease in the oil absorbency. The sorption of the composite for oil is an expansion process of three-dimensional network, and the bondage of chemical crosslinking can restrict the extension of polymeric chains as much as possible.[26] The polymeric network derived from the polymerization of long-chain monomer is easy to collapse after the oil swelling. The introduction of KF into the

resin makes great contributions to the number of crosslink points that cannot be formed among branched chains. That is, the addition of KF would actually decrease the crosslinking density. Meanwhile, the distinct hollow lumen of KF can serve as an additional framework of supporting polymeric network, by which the oil storage space of the resin is enlarged. To some extent, this can prevent the collapse of polymeric network after oil sorption and more oil will be retained in the composite, so the oil absorbency was enhanced. However, excessive content of KF results in a sharp decrease of oil absorbency, and this may be attributed to the following facts: (i) more KF will act as the physical filler, which reduces the rubber elasticity and shrinkage property of the composite and (ii) excessive KF in the resin will reduce the percentage of hydrophobic group, also resulting in the decrease of the composite network for the affinity to oil.
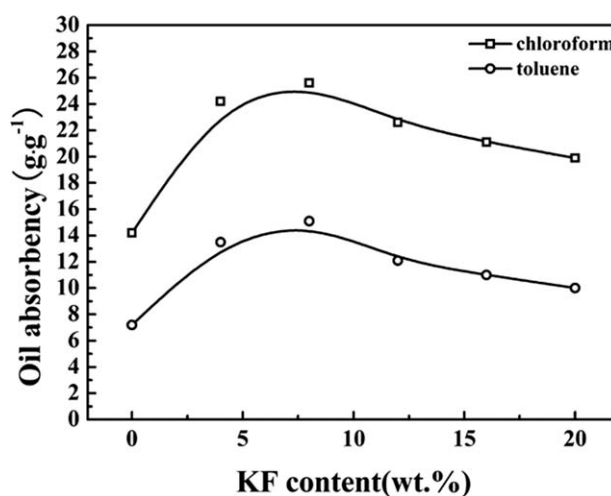
Swelling Kinetics of the Composite

Generally, the swelling kinetics of the oil sorption resin is significantly affected by various factors, such as the composition, the void content, the surface area and the type of interaction force in the crosslinked network. The incorporation of different levels of KF is certain to affect the swelling behavior of the resulting composites. The swelling kinetic curves of crosslinked PBMA and PBMA/KF in both chloroform and toluene are displayed in Figure 7. It can be seen that the oil absorbency increases with increasing immersion time and levels off after about 20 min. It is evident that an appropriate addition of KF is helpful to accelerate the sorption rate and enhance the sorption capacity. The reasons have been explained as above.

In this section, the swelling kinetics can be determined by the following experimental equation[36]:

$$dQ/dt = K(Q_{max} - Q) \qquad (1)$$

where Q_{max} is the maximum oil absorbency, Q is the characteristic oil absorbency, t is the characteristic swelling time that defined at $Q = 0.632Q_{max}$, and K is the swelling kinetic constant. Equation (1) can be changed into equation:

Figure 6. Effect of KF content on oil absorbency: initiator content, 0.4 wt %; crosslinker content, 1.0 wt %; emulsifier content, 2.0 wt %.
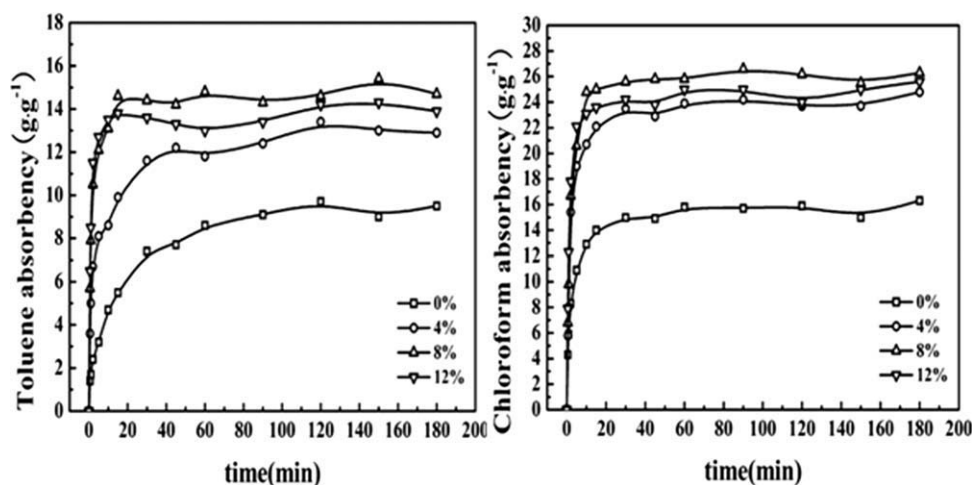
Figure 7. Swelling kinetics of PBMA resin and PBMA/KF composites.

$$\ln[Q_{max}/(Q_{max} - Q)] = Kt \qquad (2)$$

Thus, the data obtained from the oil absorbency at time t for each sample can be fitted through the linear regression of eq. (2) to obtain kinetic constant (K), characteristic oil absorbency, and characteristic time t. Because the kinetic constant K and characteristic time t are a measure of oil permeation rate, a higher K value and lower t value will reflect a quicker swelling rate. According to K and t values displayed in Table I, it can be concluded that the swelling rate of the resin is in the following order: PBMA/KF (0 wt %) < PBMA/KF (4 wt %) < PBMA/KF (8 wt %) < PBMA/KF (12 wt %). This phenomenon can be attributed to the fact that the incorporation of KF into PBMA resin can reduce the agglomeration of partial primary particles toward more compact form, a loose and porous microstructure with large-sized cavities is attained and the surface area of the composites also is improved, which makes the oil diffuse more easily because the presence of cavities would provide more spacious diffusion channels for oil into the polymer structure and leads to the increase of oil sorption rate. After all, the oil absorbency and the sorption rate rely on not only the number of the porosity but also the size of the cavities and the bulkiness.[11]

In addition, the penetration rate of oil into the vegetation materials, such as milkweed, kapok, and cotton, is higher than that of chemical-crosslinked resin. Thus, once the composites contact with oil, oil molecules can quickly penetrate into the low-crosslinked area with certain amount of KF filled and then interact with polymeric network, which is favorable to the swelling rate. Here, it should be mentioned that KF itself has efficient oil sorption capacity even in the form of shorter length, so the reduction of lipophilic group in a polymeric network with a certain amount of KF cannot reduce the oil sorption of the composites within the allowable range. By contrast, the effect of KF on improving oil sorption behavior is the most dominant. As a result, the introduction of KF cannot only enhance the oil absorbency but also improve the swelling rate.

Reusability Capability

The oil sorption capability of the composites in both toluene and chloroform to undergo six cycles of swelling and deswelling is shown in Figure 8. It can be seen that the oil absorbency of composites exhibits a downward trend on the whole, and the significant decrease in oil absorbency is observed after the first cycle of reswelling. This can be explained the fact that a small amount of the soluble fraction existed in the polymeric network is removed in the repeated swelling process, and the crosslinked structure in polymeric network also is destroyed partially in the course of heating and drying, which results in declining oil sorption capacity. Nevertheless, the resulting dry composite with 8 wt % KF still exhibits a better oil absorbency than the resin without KF. Under the same experimental conditions, the composite (8 wt % KF) only loses approximately 31% and 33.5% of initial oil absorbency for toluene and chloroform after reswelling six times, but the resin without KF can only retain

Table I. Absorption Characteristics of PBMA Resin and PBMA/KF Composites

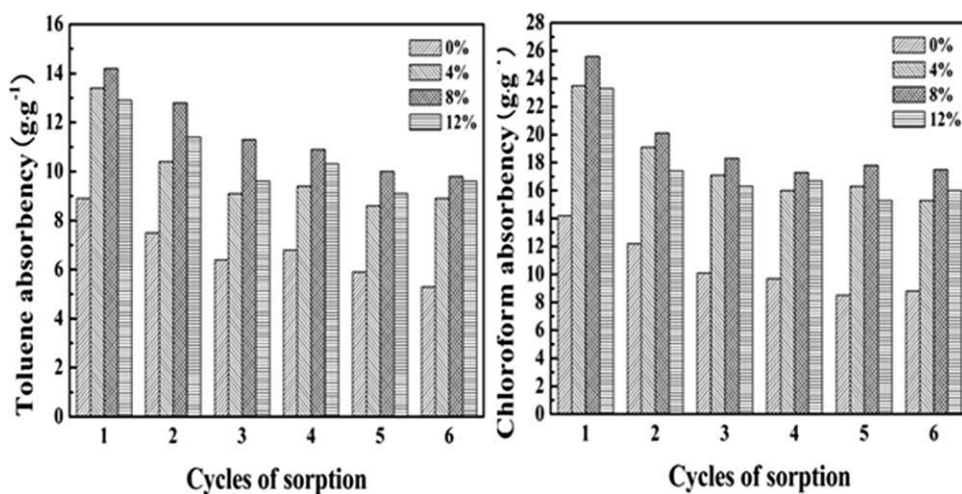|	Toluene				Chloroform			
KF content (%)	Q_{max}	Q	t (min)	K (min^{-1})	Q_{max}	Q	t (min)	K (min^{-1})
0	9.2	5.8	14.2	0.086	15.6	9.9	4.9	0.358
4	13.2	8.3	9.9	0.160	23.8	15.0	4.4	0.501
8	14.6	9.2	3.2	0.528	26.0	16.4	3.3	0.695
12	13.5	8.5	2.0	0.792	24.9	15.7	3.0	0.750

Figure 8. Reusability of PBMA resin and PBMA/KF composites.

58.5% and 45.3% of its initial oil absorbency for toluene and chloroform, respectively. These results support the conclusion that the incorporation of KF is beneficial to the improvement of reusability of the oil absorbing resin, implying that the composite is reusable and recyclable oil sorption materials.

CONCLUSIONS

PBMA/KF composite was synthesized by suspended emulsion polymerization. To improve the oil sorption capacity of the copolymer resin, KF as filler was introduced into PBMA resin to construct a kind of network with a low crosslinking and a loose structure. The optimum polymerization conditions were as follows: amount of initiator, 0.4 wt %; amount of crosslinker, 1.0 wt %; amount of emulsifier 2.0 wt %; content of KF, 8.0 wt %. The best oil absorbency of the composite was 14.6 g/g for toluene and 26.0 g/g for chloroform, whereas the PBMA resin hold only the oil absorbency of 9.2 g/g in toluene and 15.6 g/g in chloroform. Furthermore, the oil sorption composite showed better oil swelling rate and reusability capability. With fast oil absorbing capability and lower cost, the as-prepared composite exhibits certain application potentiality in some oil removal occasions.

ACKNOWLEDGMENTS

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