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Preparation and properties of poly(butyl methacrylate/lauryl methacrylate) and its blend fiber

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Abstract The functionalized copolymers, based on butyl methacrylate (BMA), and lauryl methacrylate (LMA) with crosslinking agent HEMA (hydroxyethyl methacrylate) or DVB (divinyl benzene), have been innovatively synthesized by suspension polymerization for oil absorption. Further, the copolymers and polypropylene (PP) blend fiber were attained via melt spinning. Swelling behaviors were evaluated by equilibrium swelling experiment, oil absorbency test, gel fraction measurement, and optical observations in toluene. The thermal properties and morphologies of the blend fibers were analyzed by thermogravimetry (TGA) and a field-emission scanning electron microscope, respectively. The results show that the copolymers and their blend fibers have an impressive absorbency. PBMA/LMA/HEMA can be up to 35.18 g/g, showing the highest absorption in trichloroethylene. Optical images of swollen polymers in toluene depicted a colloidal translucence with gel structure. Thermogravimetric measurement demonstrates that the copolymer and PP are incompatible and PBMA/LMA/DVB component possesses more thermal stability. The micrographs of the blend fibers exhibit coarse surface and porous cross-section, which leads to the fibers being much more readily wetted by oil and provides a huge space for oil storage.

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Introduction

In recent years, there frequently occur spill accidents in exploiting oil and transportation of dangerous organic liquids cargo. Once liquid-release accident occurs on water, it is critical to control the spill as quickly as possible in order to minimize danger and potential damage to human and environment. Floating barriers is the most commonly used technique to control the spread of oil, whereas absorbent materials are most often used to remove final traces of oil. The traditional choice is sawdust, which, however, suffers from large coincident water absorption along with the oil [1]. As a novel class of absorbents, alkyl acrylate copolymers [2, 3], which have hydrophobicity, lipophilic, and gel-type structure consisting of a 3-D elastic network, make them excellent candidates for oil absorption. Jang and Kim [4] have reported maximum capacity of alkyl acrylate copolymers about 15 g/g. Recently, Ceylan and his co-workers [5] attained butyl rubber (BR); the sorption capacity of BR for crude oil and petroleum products is 15–23 g/g. The highest oil absorptivities of cinnamoyloxy ethyl methacrylate and dodecy acrylate copolymer, and cinnamoyloxy ethyl methacrylate and octadecy acrylate copolymer are 22.9 and 34.8 g/g, respectively, reported by Atta et al. [6]. Here we demonstrate such functionalized copolymers, based on BMA and LMA with crosslinking agent HEMA or DVB. Oil absorbency of PBMA/LMA/HEMA copolymer can be up to 35.18 g/g, showing the highest absorptions toward trichloroethylene. Moreover, the crosslinked alkyl acrylate copolymers synthesized by long- and short-alkyl chain methacrylate are actually preferred in treating multi-ingredient oily wastewater.

Oil absorbency of materials depends on surface area, pores, and lipophilicity [7]. It is known that fiber has desirable advantages, such as large-specific surface area, outstanding mechanical anisotropies, easy recycle, and promoting potential in environmental applications. A substitute for oil absorbents should be an interesting alternative to oil-absorptive fibers. Polypropylene (PP) fiber-based oil sorbent products have been found to be mostly used to clean up oil spill [8]. In general, PP can absorb almost 7–9 times its weight from different oils [9].

To develop the fibrous oil absorbent, the blend fibers based on PP and the copolymers have been prepared by melting spinning in this study. To test the absorption of the copolymers and as-spun fibers, toluene, kerosene, trichloroethylene, and chloroform, as representative volatiles and harmful matters, are explored in the study. Basic physical properties were evaluated by density and melt flow index measurement. Thermogravimetric measurement, swelling observation, and surface and cross-section morphologies of the blend fiber are also investigated in the paper. This study focuses on copolymer and its blend and reports the effect of either physical crosslinking systems such as hydrogen bonds, entanglement, or chemical crosslinking on microstructure, processability, solubility, oil absorption, and incompartibility.

Experimental

Materials

Polypropylene (PP Y3700, Sinopec Corp.); Butyl methacrylate (BMA, $M_w = 142.20$, $d = 0.896$ g/cm^3, distilled before use) was purchased Tianjin Fuchen Chemical Reagents; Lauryl methacrylate (LMA) was purchased from Merck or Acros Organics (Geel, Belgium, $M_w = 254.41$, $d = 0.871$, and used as-received); Hydroxyethyl methacrylate (HEMA) was purchased from Tianjin Chemical Research Institute and used as-received; Benzoyl peroxide (BPO, recrystallized) was purchased from Shanghai Chemical Reagent Co., Ltd., a branch of China National Pharmaceutical Group Corp.; Poly (vinyl alcohol) (PVA-1799, degree of polymerization 1,700, degree of hydrolysis 99 %, purified by washing with deionized water before use) was purchased from Tianjin Chemical Reagent Co., Ltd.; Divinyl benzene (DVB) was from Shanghai Qunli Chemical Co., Ltd., and used as-received.

Synthesis of PBMA/LMA copolymers

3 g PVA as a dispersant and 0.9 g BPO as a free radical initiator were dissolved in 600 mL deionized water in a 1 L beaker for the suspension polymerization. A mixture of BMA, LMA and crosslinking agent (HEMA or DVB) in a special composition (including five different compositions as follows: 200 ml BMA only; 200 ml LMA only; 160 ml BMA, and 40 ml LMA; 160 ml BMA, 40 ml LMA, and 1 g HEMA; 160 ml BMA, 40 ml LMA, and 1 g DVB) was stirred to form solution, and the homogeneous solution was transferred into a 1 L three-neck flask. The experiment was carried out under a nitrogen atmosphere using standard suspension polymerization techniques. The mixed solution was stirred at 85 °C for 4 h reaction, and then another 1 h reacted at 95 °C. Finally, the product was washed and dried, and then respective copolymer resin was obtained.

Density testing and equilibrium swelling

The density of resin was measured according to simplified ASTM D792 method. A 25 mL pycnometer in a constant-temperature bath (an appropriate constant-temperature bath adjusted to maintain a temperature of 23 ± 0.1 °C) was weighed by precision balance.

Calculate the density of the plastic (d_{p}) as follows (Eq. (1)) [10]:

$$d_{\mathrm{p}} 23/23\,^{\circ}\mathrm{C} = a \times d/(b - c) \quad (1)$$

a, apparent mass of specimen; *b,* apparent mass of pycnometer filled with 96 % ethyl alcohol at 23.0 °C; *c,* apparent mass of pycnometer filled with 96 % ethyl alcohol and totally immersed specimen (eliminate bubble) at 23.0 °C; *d*, 0.7988 g/cm^3, the density of 96 % ethyl alcohol at 23.0 °C.

Oil absorbencies at equilibrium of the samples were determined by swelling tests. A weight of ~0.2 g each sample was placed in 50 ml toluene, kerosene, trichloroethylene, or chloroform for 24 h at room temperature, when the samples reached equilibrium swelling. The samples were weighed after removing excess oil using a filter paper.

Melt flow index measurement and spinning process of the copolymer and PP blends

PP and the copolymer weighted by 50/50 were mixed using SHJ-20 co-rotating twin-screw extruder (Nanjing Jieya Corporation, China) at 210 °C. MFI of extrusion product was investigated using a XNR-400A melt flow index instrument (Chengde Testing Machine Co., Ltd., China). According to ASTM D1238-04 Standard test method for melt flow rates of thermoplastics by extrusion plastometer, the MFI data on PP and copolymer blends were displayed in Table 1.

PP and the copolymers were dried in a vacuum oven at 60 °C for 48 h before melt spinning to remove moisture, completely. The blends with different compositions were prepared by using a SHJ-20 co-rotating twin-screw extruder. The composition of the blends was 50 wt % of PP and 50 wt % of the copolymer as PBMA/LMA, PBMA/LMA/HEMA, or PBMA/LMA/DVB, respectively. The melt spinning was carried out using a twin-screw extruder equipped with a spinneret diameter of 0.5 mm and 36 holes (L/D 30) followed by air-cooling. The barrel temperatures were set to 205–205–205–210–215–215 °C in the hopper-to-nozzle direction for the first zone, the second zone, the third zone, the fourth zone, the fifth zone, and the sixth zone, respectively. In addition, the melt temperature at the outlet and spinneret temperature were 215 and 200 °C. The screws rotating speed were maintained at 30 rpm and the melt pump rotation speed was about 80–90 rpm. These processing conditions resulted in a final diameter of 0.6 ± 0.05 mm, and eventually, the blend fiber was preserved for test and analysis.

Oil absorbency

Crude oil contains numerous individual components with varying proportions. Toluene, trichloroethylene, and chloroform, having lower viscosities than bunker oil

Table 1 MFI parameters from the blend of PP/PBMA-*co*-HEMA

The compositions of PP and PBMA/LMA blends	Total load including piston, kg/approximate pressure, kPa	MFI, g 10 min^{-1}		
		240 °C	260 °C	280 °C
PP	2.16 kg/298.2 kPa	46.59	56.76	81.42
	3.8 kg/524.0 kPa	37.86	65.61	127.38
PP and PBMA/LMA (wt% 1:1)	2.16 kg/298.2 kPa	7.095	9.384	11.07
	3.8 kg/524.0 kPa	20.01	23.106	36.84
PP and PBMA/LMA/HEMA (wt% 1:1)	2.16 kg/298.2 kPa	0.009	2.04	11.07
	3.8 kg/524.0 kPa	0.192	3.6	36.84
PP and PBMA/LMA/DVB (wt% 1:1)	2.16 kg/298.2 kPa	-	0.15	-
	3.8 kg/524.0 kPa	0.012	0.75	0.282

in this experiment so that the swelling behavior of the samples could be easily evaluated, are prominent representatives of light oil fractions. They are commonly effective solvents for a variety of organic materials in pesticide industry and chemical plants. Because of its low viscosity and density, it can easily become part of oil-based organic pollutants [11]. Kerosene is obtained from the fractional distillation of crude oil between 150 and 275 °C, resulting in a mixture of carbon chains that typically contain between 6 and 16 carbon atoms per molecule [12]. As a petroleum product miscible with many industrial liquids, kerosene can be used as both a solvent, able to remove other petroleum products, such as a lubricant, jet fuel, and an additive in diesel fuel to prevent gelling or waxing in cold temperatures. So we used these organic liquids to test our newly synthesized polymeric oil absorbers.

Oil absorbency of the copolymers or as-prepared fibers was evaluated by ASTM (F726-81): a 0.1 g sample was put in a stainless steel mesh (4 × 4 × 2 cm) and immersed in oil. The sample and the mesh were together picked up from the oil, drained for 20 s, tapped with filter paper to remove excess oil from the bottom of the mesh, and then weighed. The oil absorbency (Q) was calculated by the following equation (Eq. (2)):

$$Q = \frac{\text{The weight of absorbed oil}}{\text{The weight of the sample before oil absorption}} \tag{2}$$

Gel fraction

Insoluble fractions of the copolymers and as-spun fibers may be discussed by gel fraction. A weighed quantity of the fiber was put in a conventional Soxhlet extractor and continuously extracted for 8 h, using butanone as solvent because of its low boiling point and its ability to dissolve methacrylate. After extracting, the samples were dried in vacuum at 60 °C until a constant weight. The gel fraction (G) was calculated according to the following equation (Eq. (3)) by [13]:

$$G(\%) = \frac{\text{The weight of dried sample after extraction with butanone}}{\text{The weight of dried sample before extraction}} \tag{3}$$

Thermogravimetric analysis

The thermal analysis of the fiber was carried out using a TGA instrument (NETZSCH STA 409) with a thermogravimetric module. The TGA scans were operated in the temperature range of 35–800 °C at a calefactive rate of 10 °C/min under nitrogen atmosphere. Weight loss at different temperatures was evaluated from the differential thermogravimetry (DTG) curve.

Field-emission scanning electron microscope

The morphologies of as-prepared fibers were monitored by a field-emission scanning electron microscopy (FESEM, Hitachi S-4800, and Japan). Samples were frozen in liquid N_2, followed by fracturing to expose their cross-sectional areas. The cross-section and surface of the fiber were coated with gold by an electro-deposition method to impart electrical conduction before recording the FESEM micrographs.

Results and discussion

Oil absorbency and gel fraction

Oil absorbency versus time for three different copolymers is shown in Fig. 1. Absorption curves have a similar trend at different oils. More specifically, as the immersed time increases, oil absorbency begins to increase and then has a basically flat curve. The absorption curves approximate a monotonic, smooth curve, *S*-shaped and arise gradually till equilibrium. Note that results of the absorption are used to demonstrate the macromolecular structure in the study. Comparison of absorbency at equilibrium of three copolymers, the results displayed in Table 2 indicate stability of 3-D polymer networks: entanglement by long alkyl chain methacrylate in PBMA/LMA < entanglement and hydrogen bonds crosslinking in PBMA/LMA/HEMA < entanglement and chemical crosslinking in PBMA/LMA/DVB. This is because in case of PBMA/LMA/HEMA, a random and activating 3-D hydrogen bond network structure from PHEMA in absorption should be of benefit to permeation,

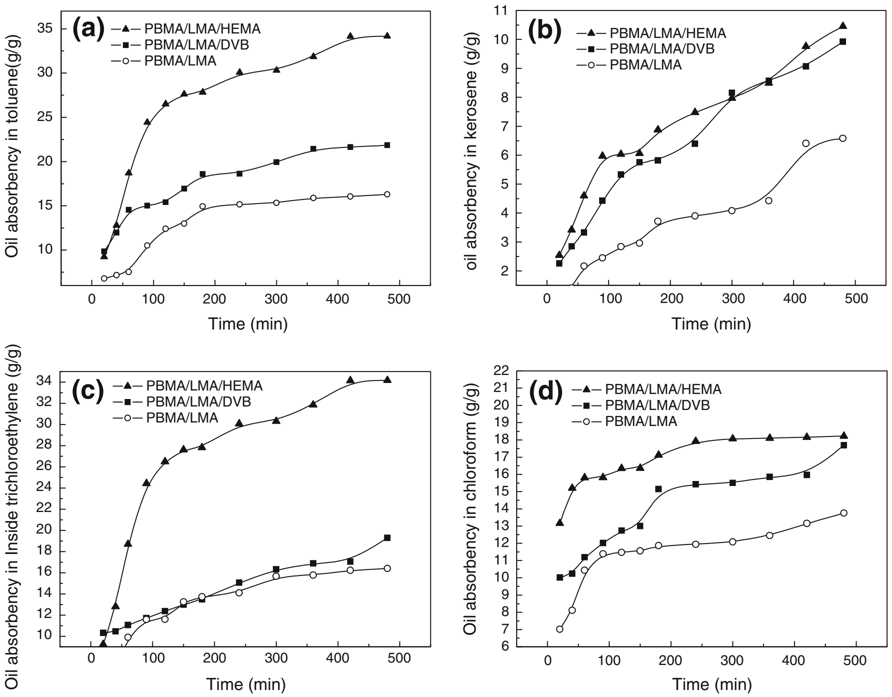

Fig. 1 Relationship between oil absorbency and time for three different copolymers in **a** toluene, **b** kerosene, **c** trichloroethylene, and **d** chloroform

Table 2 Physical property of poly (butyl methacrylate/lauryl methacrylate) resins

Sample	Product(s) resin and density (g/cm^3)	Conformation	Absorbency at equilibrium (g/g) and evaluation			
			Toluene	Kerosene	Trichloroethylene	Chloroform
PBMA	White granular, 1.041	Linear macromolecular chain structure	Soluble	No change	Soluble	Soluble
PLMA	Floc film, 0.897	Entanglement by long alkyl chain methacrylate	6.41, sparingly swollen	6.92, sparingly swollen	9.08, sparingly change	6.57, sparingly swollen
PBMA/LMA	Bulky solid, 1.024	Network by linear, entanglement	21.64, swollen	10.01, swollen	14.13, swollen	12.47, swollen
PBMA/LMA/HEMA	Unconsolidated solid, 0.947	Medium network by linear, entanglement and hydrogen bonds crosslinking	32.85, freely swollen	10.12, swollen	35.18, greatly swollen	17.53, swollen
PBMA/LMA/DVB	Rigid beads, 1.050	Stable network by linear, entanglement and chemical crosslinking	15.58, swollen, stable network structure	6.60, sparingly swollen, stable network structure	15.86, swollen, stable network structure	17.52, swollen, stable network structure

diffusion, and swelling. However, the limitation of chemical crosslinking points in PBMA/LMA/DVB during the swelling process is relatively negative for oil absorbency. For instance, Liu et al. [14] attained ethylene–propylene–diene (EPDM) and 4-*tert*-butylstyrene (tBS) copolymer with chemical crosslinking structure; the maximum toluene absorbency is 27.50 g/g as compared to the maximum value of 34.15 g/g to toluene obtained in our study.

During absorption process in toluene, kerosene, trichloroethylene, and chloroform, organic liquid polarity plays a very important role. Most often chemists examine the dielectric constant of a solvent to determine whether it is polar or nonpolar, with higher values reflecting greater polarity [15]. In general, a polar solvent may be expected to dissolve readily polar solutes [16]. Dielectric constant of a solvent is a relative measure of its polarity. Dielectric constant value for toluene, kerosene, trichloroethylene, and chloroform is 2.38, 1.8, 3.42, and 4.8 [17], respectively, and they all were classified as nonpolar. However, asymmetry of organic liquid molecular geometry determined less polarity, that is how, for instance, commonly used low polarity solvents are toluene, kerosene, trichloroethylene, and chloroform [18]. PBMA/LMA/HEMA is measured of oil absorption to act as a hydrogen-bond donor, while trichloroethylene is considered as a hydrogen bond acceptor from a solute. Hydrogen bonds interactions appear in charge of absorption, up to 34.5 g/g, see Fig. 1c. Moreover, PBMA shown in Table 2 and PBMA/HEMA in our pervious studies [19] have hardly swollen in kerosene. It is reported that the number of carbon in alkyl groups of the copolymer is larger than the number of carbon in oil molecular, higher absorbency will be expected [4]. As we know, kerosene is a mixture of carbon chains that typically contain between 6 and 16 carbon atoms per molecule. The addition of LMA brings about an impressive kerosene absorption capacity. We can deduce that as-prepared copolymeric resins with long- or short-alkyl chain methacrylate are of potential importance in treating multi-ingredient oily wastewater. Given numerous individual components in oil spill, apparently, broad-spectrum absorbents are actually preferred in application of oily wastewater treatment plant.

Oil-absorptive fiber has greater superiority to textile or nonwoven industry [13, 20]. However, the fibration desire to produce improved products development is frustrated by its poor processability. To overcome these poor processing properties, we prepared the blend fibers via melting spinning. Comparison to the resins' absorbency, absorption capacity of the fiber has a significant decline. It is explained that polymer networks swelling retardation and macromolecular active stretching barrier reliance on crystallization and skeleton supporting of PP. At the same time, the curves depicted in Fig. 2 of oil absorbency versus time became smoothing and flat. It suggests arrangement and conformation of PP macromolecular skeleton structure are so stable that the fiber is not easy to deform let alone destroy while swelling. Nevertheless, the maximum absorbency of as-spun fiber is 10.2 g/g as compared to the value of 7–9 g/g obtained using pure PP fiber, widely used commercial oil sorbent [9].

Extraction tests showed that the as-prepared sorbents are insoluble in butanone. Gel fraction was commonly used as a parameter representing the degree of crosslinking of polymer materials. As shown in Fig. 3, for copolymer resins, apparently, chemical crosslinking by DVB is more stable, and insoluble. The

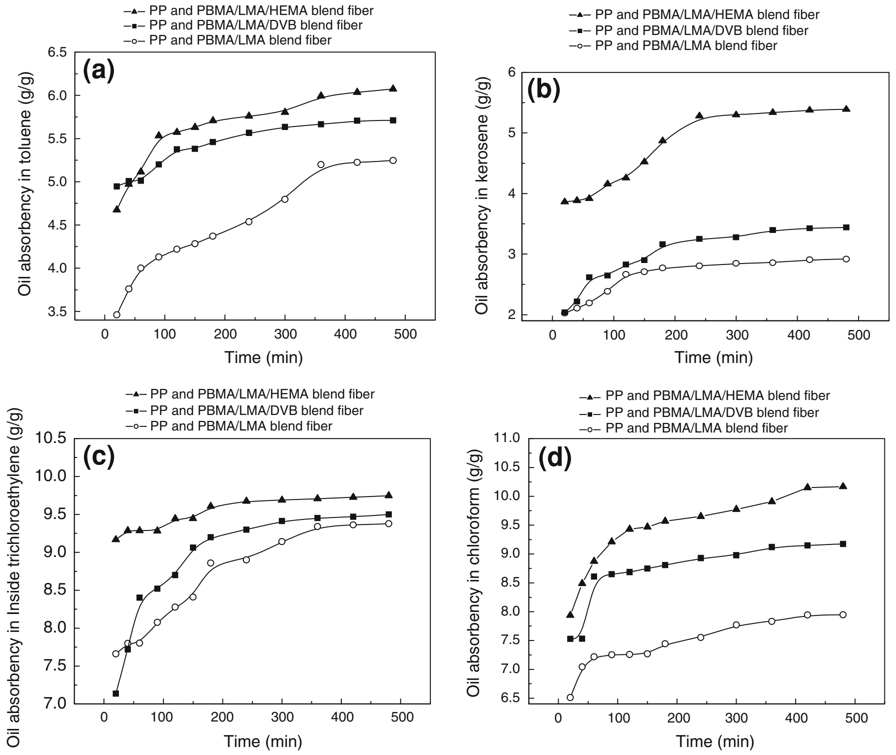

Fig. 2 Relationship between oil absorbency and time for as-prepared fibers in **a** toluene, **b** kerosene, **c** trichloroethylene, and **d** chloroform

solubility was consistent with the stability from absorbency at equilibrium in previous section. For the blend fibers, as PP was blended, gel fraction significantly increased. The results indicate that PP restrains the solubility of PBMA-*co*-HEMA in extract processing.

Thermal properties

Thermal behavior of polymer can be evaluated with weight loss by thermogravimetric measurement. As shown in Fig. 4, the curves demonstrate the onset of degradation for pure PP is delayed with the addition of copolymer. The polymer blend with crosslinking agent DVB exhibits more thermal stability, because fiber chemical crosslinked by covalent bonds possess high-thermal stability and high-solvent resistance, and it is normally inert [21]. In reverse, noncovalent crosslinking systems, such as hydrogen bonds and entanglements, can be expected to possess the tolerant stability. DTG curves for the blends indicate two peaks, one corresponding with the amorphous phase and the other with the PP phase, indicating the incompatibility of the blends [22]. The peak of PP shows a shift toward the left on the temperature axis, the finding may be ascribed to the different degradation patterns and mechanisms in the two cases as well as interface interaction.

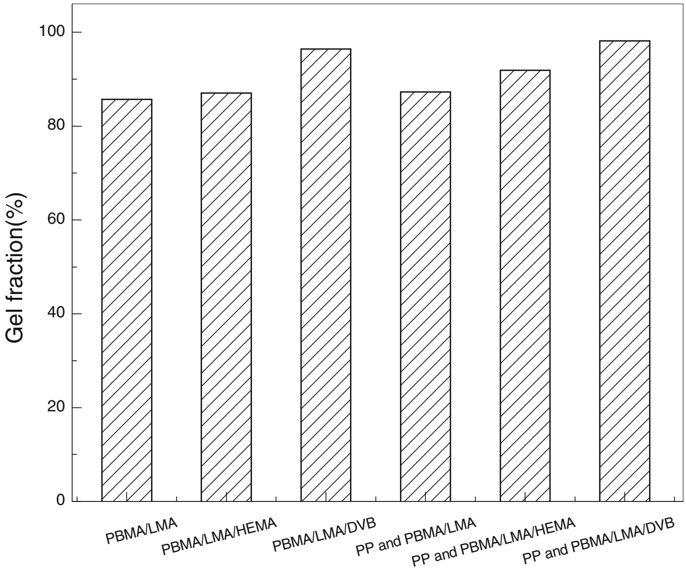

Fig. 3 Gel fraction of different samples

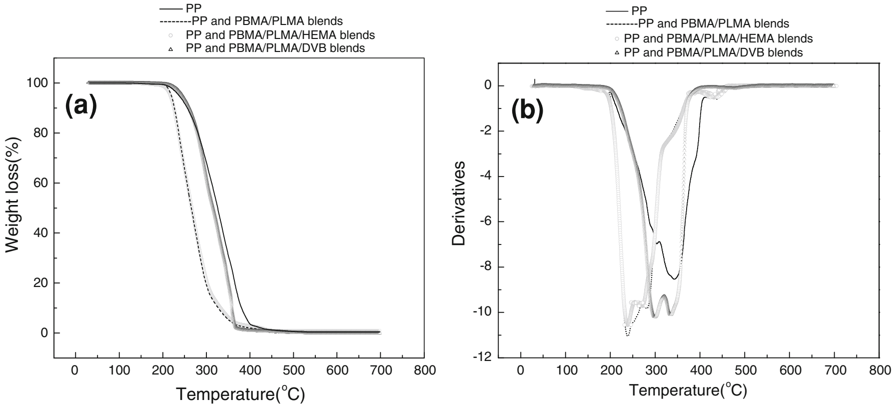

Fig. 4 TGA (**a**) and DTG (**b**) curves of as-spun fibers

Morphology analysis

Observations

Figure 5 vividly shows images of absorption process. The observations noted for the swelling test were carried out in the toluene. As the time increases, the sample volume largely swells with the oil molecular "sucked" up. Moreover, the sample becomes colloidal translucence with gel structures. Comparison to the resin, toluene

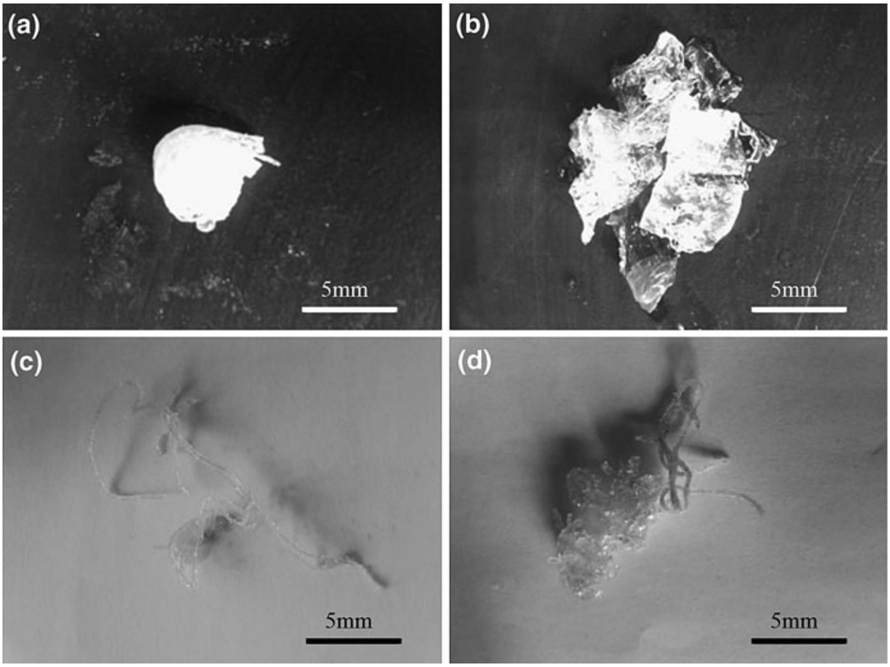

Fig. 5 **a** Optical image of the copolymer resin. **b** A piece of resin swollen by absorption. **c** The as-prepared blend fibers. **d** Swollen fibers

volume expansion of the blend fibers is barely satisfactory due to PP molecule's restriction. During the absorption, a twisting phenomenon is recording in absorption for the fiber.

FESEM analysis

The surfaces and cross-sections of the as-prepared fibers were morphologically observed by FESEM. As depicted in Fig. 6, the micrographs of the blend fibers show coarse surface and the presence of point-like convexities and cavities, which lead to fibers being much more readily wetted by oil. The features of PP and PBMA/LMA/HEMA fiber appear more pits and have an optimal performance in oil absorption. Comparison of Fig. 7c, f with a, d and b, e shows that a relative dense surface as well as many grains and cavities. This demonstrates addition of chemical crosslink agent DVB brings out macromolecular structure stability preventing prominent invasion of oil.

Figure 7 exhibits the porous structure on the cross-section of the blend fiber. The polymers are immiscible, and therefore, there is a kind of interfacial micro cavities (Fig. 7a–c) and rugged topography (Fig. 7d–f). It is expected that the porous and rugged structure provides a huge space for oil storage, which is superior to the prepared absorber [23, 24]. Moreover, the lumen-like cavities exists in the PP and PBMA/LMA/HEMA fiber as shown in Fig. 7b, e, which shows even more potential in terms of oil absorbency than two other blend fibers.

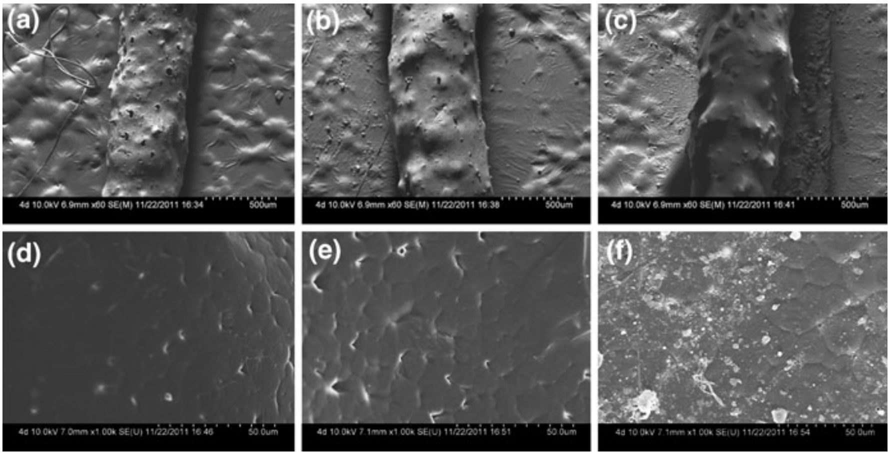

Fig. 6 Surface morphology of the blend fiber: **a**–**c** whole surface and **d**–**f** enlarged surface for PP and PBMA/LMA fiber, PP and PBMA/LMA/HEMA fiber, PP and PBMA/LMA/DVB fiber, respectively

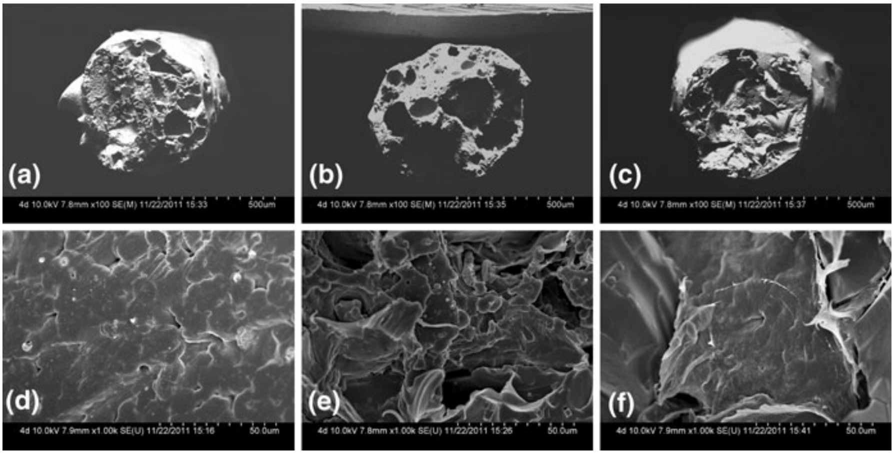

Fig. 7 Cross-section morphology of the blend fiber: **a**–**c** whole cross-section and **d**–**f** enlarged cross-section for PP and PBMA/LMA fiber, PP and PBMA/LMA/HEMA fiber, PP and PBMA/LMA/DVB fiber, respectively

Conclusion

As absorbents, the copolymers and their blend fibers have impressive absorbency for potential oil spill application: maximum capacity at equilibrium in different organic liquids of PBMA/LMA is up to 21.64 g/g in toluene, PBMA/LMA/HEMA to 35.18 g/g in trichloroethylene, PBMA/LMA/DVB to 17.52 g/g in chloroform, respectively. To the same organic liquid, PBMA/LMA/HEMA exhibits ahead absorptive capacity among the copolymers. The density of the copolymers is ca. 1 g/cm^3, in favor of floating absorption. Melt flow index measurement as a guide for the melting spinning indicates poor thermoplasticties of the copolymer. Optical images of swelling and absorption in toluene reflect the morphology variations, recording a colloidal translucence with gel structure. Absorbency at equilibrium and gel fraction indicate stability of polymer networks: entanglement by long alkyl chain methacrylate in PBMA/LMA < entanglement and hydrogen bonds crosslinking in PBMA/LMA/HEMA < entanglement and chemical crosslinking in PBMA/LMA/DVB. Thermogravimetric measurement demonstrates that the synthetic copolymer and PP are incompatible and PBMA/LMA/DVB with crosslinking agent possesses more thermal stability. The micrographs of the blend fibers show coarse surface and the presence of point-like convexities and cavities, which lead to fibers being much more readily wetted by oil. The porous and rugged structures of cross-section provide a huge space for oil storage.

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