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Evaluation of different PP grafted sorbent for oil spill cleanup

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ABSTRACT

In this work, a new kind of polypropylene-based sorbent was prepared by ultraviolet radiation-induced graft polymerization of octadecyl acrylate onto polypropylene fiber and polypropylene nonwoven, respectively. Effects of grafting time, monomer concentration, and iniferter concentration on the grafting degree were systematically investigated. FTIR was used to characterize the chemical changes of the polypropylene surface, and SEM results confirmed the morphological changes after graft polymerization. Two forms of modification polypropylene (PP) sorbents were evaluated through oil-over-water experiment in this study in terms of oil sorption capacity, oil-retention property, and reusability performance. The grafted PP sorbent could be straightforward to operate, easy to apply and maintain, affordable to implement, and therefore has the potential to become widely employed for the removal of oil from the spill site.

Keywords: Polypropylene sorbent; Grafting degree; Octadecyl acrylate; Sorption; Reusability

1. Introduction

Nowadays, with frequent and massive oil spill accidents, ocean ecology and the natural environment are becoming seriously damaged. The spilled oil contributes an undesirable taste and odor to water environment, and even very low concentration of oil can be toxic to micro-organisms, which are responsible for biodegradation in conventional sewage processes. In addition to the loss of energy source and chemical materials, the spilled oil also results in a direct threat to the survival of humans and animals [1–5].

Treatment of oil spills remains a challenge to environmental scientists and technologists. When oil comes in contact with water, it forms oil-in-water emulsion or floating film that needs to be removed before it is discharged into the environment [6]. Therefore, it is important to establish and develop new techniques for oil removal from the spilled sites. Among all the existing techniques used for combating oil spills, one of the most economical and efficient methods is oil sorption by sorbents. The use of sorbent materials for oil removal is attractive principally due to its ability to transform organic contaminant in the liquid phase into solid or semi solid phase [7–9]. Once this change is achieved, the recovery of the spilled oil by removal from the sorbent structure is not difficult.

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The efficient and economical sorbent materials are those which, besides being inexpensive and readily available, demonstrate fast oil sorption rate, high oil sorption capacity, low water pickup, high oil retention capacity during transfer, high recovery of the sorbed oil with simple methods, and good reusability [10–12]. At present, most of the commercially available oil sorbents are synthetic products such as high-sorption acrylate resin, which were prepared by radical suspension copolymerization of acrylate monomer, made them good in flexibility, sorption properties, and retention performance [13–15]. However, the long sorption equilibrium (6–8 h) have become obstacles for potential application in oil spill-treatment, in addition, the sorbed oil can be difficult to recovery due to their certain granular morphology and usually be burnt [16–18].

By contrast, polypropylene-based sorbents were chosen as an efficient sorbent because of their rapid saturation-sorption rate, simple recovery method, and high selectivity for the organic chemicals over the water. Polypropylene nonwoven sorbents are consolidated fibrous materials, which are different from the conventional textile fabrics. Nonwovens are based on webs of individual PP fibers. These fibrous webs, which contain small pores, facilitate the transport of liquids into the sorbents and retain the liquids after sorption. Zahid et al. [19] researched the important factor of polypropylene fiber adsorption performance on the water surface. Use of polypropylene fiber in the removal of oil from the surface of seawater was investigated. Wei and Fotheringham [20] obtained the adsorption capacity and retention performance of different type polypropylene nonwoven. The investigation revealed that the sorbent porosity and oil property are the most important factors in the oil-sorption performance of polypropylene nonwoven. In addition, it is convenient to graft monomers onto polypropylene matrixes which provide large spaces for the improvement of their sorption properties. However, due to inadequate hydrophobicity and flexibility, the polypropylene sorbents exhibit poor sorption capacity and retention capacity. They may also experience collapse of their interior structure after several sorption-regeneration cycles [5]. The limitations of the acrylate resin and polypropylene sorbents have led to the recent interest in developing alternative materials.

Nowadays, much attention has been paid to the technique of graft polymerization on polymeric matrixes to prepare polymeric sorbents; the technique usually can enhance sorption efficiency of the sorbents [21–23]. In this research, the direct radiation grafting technique was used to graft octadecyl acrylate (OA) monomer onto PP fibers and nonwovens matrixes [24]. The optimum grafting conditions at which the grafting process proceeds extensively and homogeneously were investigated. The new polypropylene graft modification sorbent created opportunity to utilize specific properties of both PP sorbent and acrylate resins to overcome the shortcomings of each used alone, and exhibited a fast sorption rate, high sorption capacity, good retention characteristic, and excellent reusability. To the best of our knowledge, the relationship between the structural properties and the adsorption performance of the different PP grafted sorbent was not clear so far. In this respect, this study systematically evaluated two forms of grafted PP sorbent for its oil-sorption properties, oil-retention performances, and reusability characteristics. The oil types investigated were diesel and diluted crude oil, which will remain onto the water surface after oil-spill accidents.

2. Materials and methods

2.1. Materials

The polypropylene fibers and polypropylene nonwovens were obtained from Haidexin Chemical Factory, Jiangsu, China. OA and isopropanol were from Tianjin Chemical Reagent Institute. Photosensitizer benzophenone and sodium diethyl dithiocarbamate were obtained from Tianyi Chemical Factory, Tianjin, China. All of the chemicals were of analytical grade and used as received.

Diesel, crude oil, and toluene were obtained from the Company of Nan Jiang. The oil used in this experiment is diluted with toluene (10%). The oil density and viscosity were measured using a digital density meter and dynamic viscosity meter (shear rate 100 s^{-1}) at 20˚C, respectively. The characteristics of the investigated oil properties are shown in Table 1.

Table 1
Oil properties

Oil type	Density (g/cm^3)	Viscosity (cP)	Content of water (wt.%)	Weight-losing (wt.%)	
				After 24 h	After 48 h
Diesel	0.887	1.32	<0.05	0. 21	0. 34
Crude oil	0.895	2.59	none	0. 06	0.11

2.2. Preparation of OA grafted polypropylene sorbent (PP-g-OA)

The new polypropylene sorbent was prepared by UV radiation-induced graft polymerization of OA monomer onto polypropylene fiber or polypropylene nonwoven matrices. UV reactor radiation (Analytical Instruments, Germany) with a wavelength of 312 nm was applied to initiate the graft polymerization at ambient temperature. The PP sorbent matrix was first immersed into the ultra-pure water for 24 h to remove the impurities. After drying in the oven, it was placed in a Pyrex polyethylene bag with the thickness of 2 mm which was used as a matrix for the grafting reaction. In the first step, PP sorbent matrixes (0.5 ± 0.01 g) cut into pieces of 15 cm by 10 cm and then immersed in different concentrations of OA solutions ranging from 0 to 20% (wt.%) in isopropanol/water mixture solution. The mixture was sealed in polyethylene bags purged with nitrogen gas for 10 min, which was subsequently exposed to an UV reactor with wavelength of 312 nm after a pre-determined period. In the second step, the grafted polymers were then removed and extracted thoroughly by acetone to remove the homopolymer and the unreacted monomer. The grafted fibers were dried at 60℃ until constant weight, and then weighed to get W_g.

Percentage of grafting degree based on% mass was calculated as:

$$G\ (\%) = [(W_g - W_0)/W_0] \times 100\% \tag{1}$$

where W_0 and W_g are the weight of the original and the grafted PP fiber or nonwoven after extraction, respectively [25].

2.3. Material characterization

Functional groups on the original and grafted PP fibers or nonwovens were analyzed using fourier transform infrared spectra (VECTOR22, BRUKER Co.). The surface morphology was analyzed using scanning electron microscope (QUANTA 200, FEI). The specific surface area and porosity were determined by BET method (TriStar 3000, Micromeritics) and BS EN 29073, the measurements were repeated three times and the mean value was calculated.

2.4. Sorption experiments

In the sorption experiments, 30 g of experiment oil was added into a 200 mL beaker containing 100 mL of deionized water. After that, 0.5 g PP fiber or nonwoven sample was gently placed onto the oil layer, and then removed using a nylon net collector after a predetermined time (s). At last, the sorbed samples were then removed and weighed. All tests were carried out at 20 ± 2℃. The remaining liquid in the beaker was poured into a measuring cylinder to settle over night in order to examine whether the sorbents had absorbed water together with the experiment oil.

Sorption time ranged from 1.0 to 10.0 s, the above measurements were repeated five times and the mean value was calculated. The American Society of Testing and Materials method was used for oil sorption capability tests [5]. The sorption capacity was calculated as the ratio of sorption to dry grafted PP fiber or nonwoven:

$$\text{Sorption capacity} = (S_1 - S_0)/S_0 \tag{2}$$

where S_0 is the dried PP sorbent (g) and S_1 is the total mass of the sorbed samples (g).

2.5. Retention capacity and reusability test of PP-g-OA sorbents

In order to investigate the oil retention capacity of grafted PP fiber and grafted PP nonwoven, the sorbent after weighed were hung on a digital balance, whereupon the sorbed oil began to drip from the sorbents [26]. The sorbent samples were recorded after 1–60 min. The dynamic retention capacity was also evaluated as follows:

$$\text{Dynamic retention capacity} = W\text{t} = i/W\text{t} = 1 \times 100\% \tag{3}$$

where $W\text{t} = 1$ is the weight of oil bath (g) at 1 min dripping, $W\text{t} = i$ is the weight of oil bath (g) at t min dripping, $i = 2, 3, 4, \ldots$

The reusability efficiencies of the grafted PP sorbents were determined after repeated sorption-regeneration cycles. Ten cycles of the sorption process were performed for each sample. Between each cycle, the sorbent was squeezed and weighed again. The weight of the squeezed material was used as the initial weight of the sorbent in each subsequent sorption cycle.

3. Results and discussion

3.1. Characterization and graft polymerization of PP-g-OA

The grafted polypropylene-based sorbent PP-g-OA was grafted with OA initiated by ultraviolet (UV) radiation. Fig. 1 shows the differential FTIR spectrum, which was obtained by subtracting the spectrum of the original and the grafted PP fiber and nonwoven after extraction by acetone. Compared with the original PP, the appearance of the new adsorption bands at 1,733 cm^{-1}, which was not present in the spectrum of the original PP matrix, can be attributed to the presence of the stretching vibrations of carbonyl groups, confirming the existence of ester groups [24]. The remaining spectrum of PP-g-OA was all in good agreement with the spectrum of original polypropylene. Therefore, the characteristic peaks showed that OA was successfully grafted onto the PP matrixes after graft modification.

In order to further determine the morphology of the surface characteristics of PP-g-OA, TEM observation was conducted (Fig. 2(a–d)). A considerable difference between original PP and PP-g-OA in TEM observation suggested that the distance and cross-sectional area between the filaments of PP fiber were higher than PP nonwoven. In addition, the smooth surface of PP became rough after graft polymerization, a heterogeneous grafting layer structure

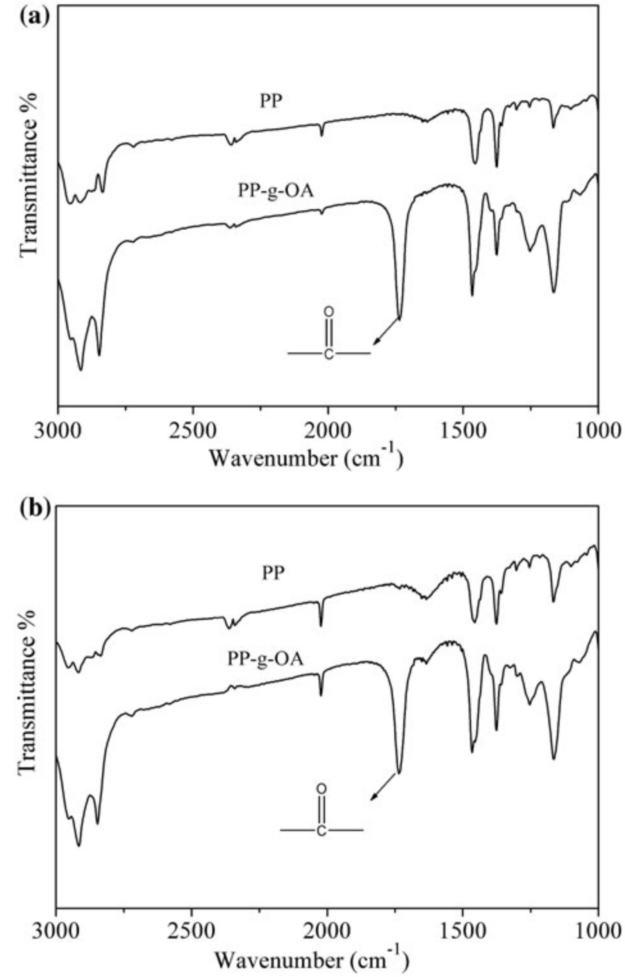

Fig. 1. FTIR spectra of original and grafted polypropylene-based sorbent, (a) PP fiber; (b) PP nonwoven.

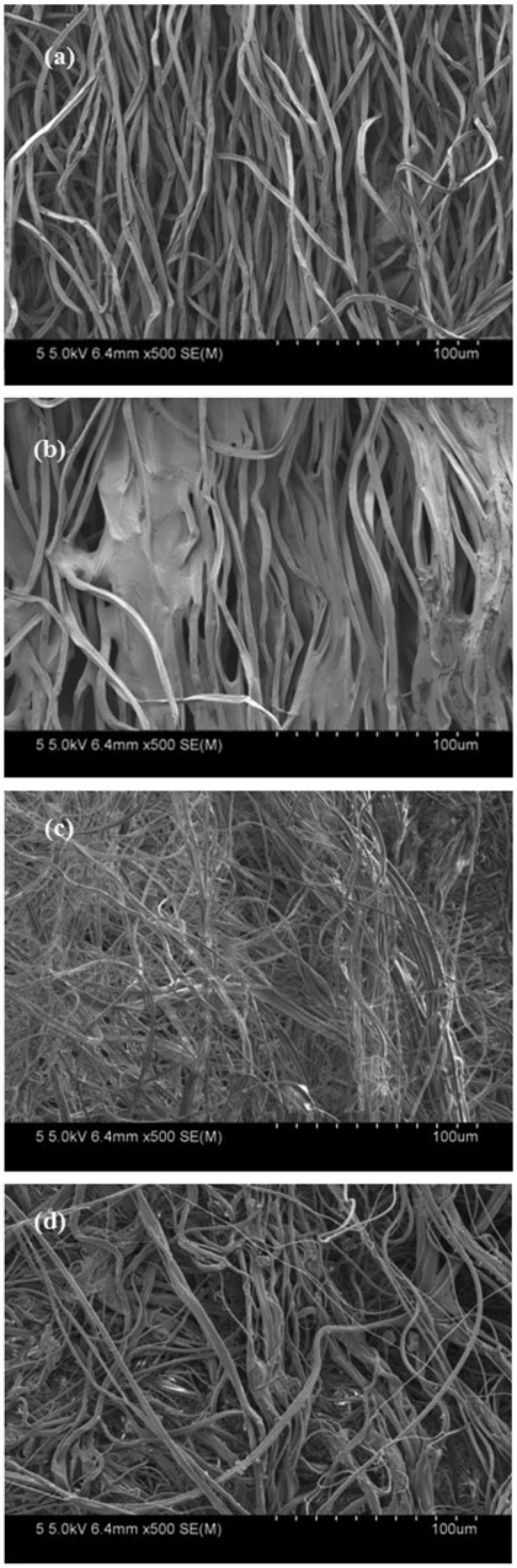

Fig. 2. SEM micrographs of original and grafted PP fiber (a), (b) and original and grafted PP nonwoven (c), (d).

was formed on the PP surface randomly which was consistent with the thickness and coarseness of PP fiber matrixes obviously.

The degree of OA grafting on polypropylene fiber or nonwoven at different monomer concentration as a function of irradiation time is given in Fig. 3. It could be seen that, the degree of grafting increases with increasing irradiation time. As UV irradiation time increased, there were more active sites excited on the PP matrix and much more monomers accessible to the surface radicals, resulted in the increase of grafting degree. However, with the irradiation time prolonged, the consumption of initiator and monomer led to an asymptotic saturation of the grafting degree [27,28]. Thus, the irradiation time was set as 30 min in the following experiments. The grafting degree of PP fiber increased nearly proportional with an increase in the monomer concentration up to 10% and then declined. The grafting degree of PP nonwoven with different monomer concentration also showed the same trend, when the monomer concentration was 15%, the grafting degree of PP nonwoven reached the maximum. This trend is believed to be related to higher concentrations of monomer available to react with the active sites on irradiated PP matrix, so that a higher grafting degree was achieved with the increase in monomer concentration. The grafting degree inclined to level off at higher concentration can be due to the homopolymerization of OA monomer. In fact, the grafting is controlled by a cumulative effect of the monomer diffusion within the polymer matrixces, termination of the growing polymer chains, and the deactivation of the primary radicals [29]. If the surface of PP matrix was covered with the OA homopolymer, the diffusion of monomer into the inner area of PP matrix was hindered, resulting in the decrease of grafting degree with the sequent increase of monomer concentration. In addition, the grafting degree of PP fiber was relatively higher than that PP nonwoven under a same monomer concentration.

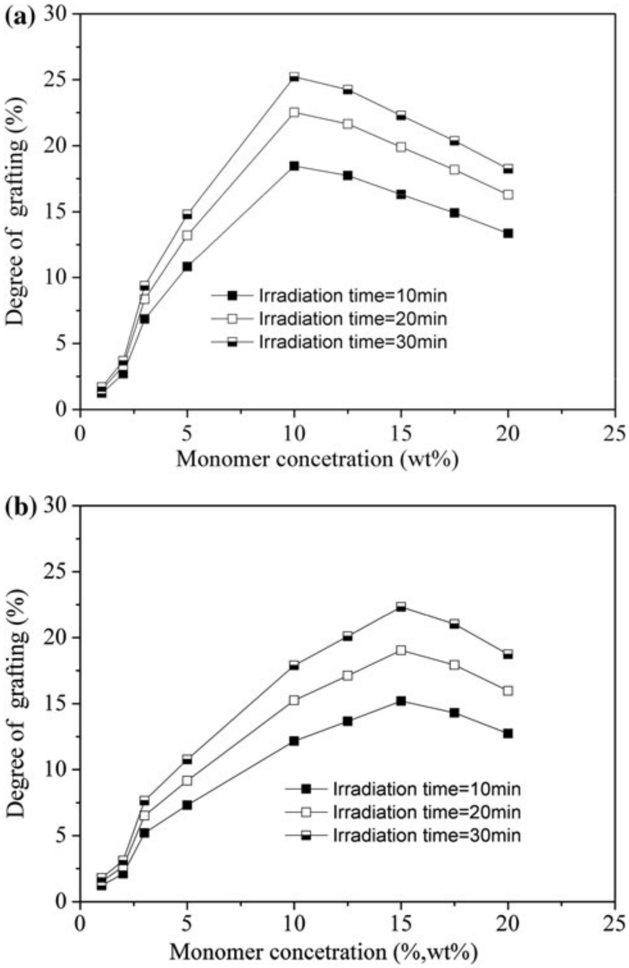

Fig. 3. The relationship between the monomer concentration and grafting degree, following different irradiation time. (a) PP fiber; (b) PP nonwoven.

3.2. Oil sorption performance of PP-g-OA

As shown in Fig. 4, it is clear that the grafting degree has a profound influence on the sorption capacity of grafted PP sorbent. Sorption capacity of the PP-based grafted sorbent for crude oil was higher than diesel commonly, this could be due to the higher viscosity of crude oil as compared to diesel. With the grafting degree increased from 0 to 14.79%, the sorption capacity of grafted fiber increased from 6.47; 12.12 to 18.78; 35.17 g/g for diesel and crude oil, respectively. However, the fiber diameter increased excessively with the grafting degree further increasing from 14.79 to 25.22%, which leaded the specific surface area decrease continuously (Table 2) and attached to a minimum sorption area, the sorption capacity of grafted fiber decreased considerably. Similarly, the sorption capacity of grafted nonwoven also showed the same trend, the sorption capacity of grafted nonwoven increased from 5.69; 10.46 to 16.51; 30.36 g/g for diesel and crude oil with the grafting degree increasing from 0 to 13.26%, and then decreased with the sequent increase of grafting degree of nonwoven.

Commonly, the sorption capacity of PP grafted fiber was relatively higher than that PP grafted nonwoven under a similar grafting degree. For evaluation the effects of grafting degree on the oil sorption capacity of PP-based sorbent, the surface morphology should also be considered. It indicated that the PP-based sorbent with a higher porosity and surface area tends to have higher sorption capacity, for example,

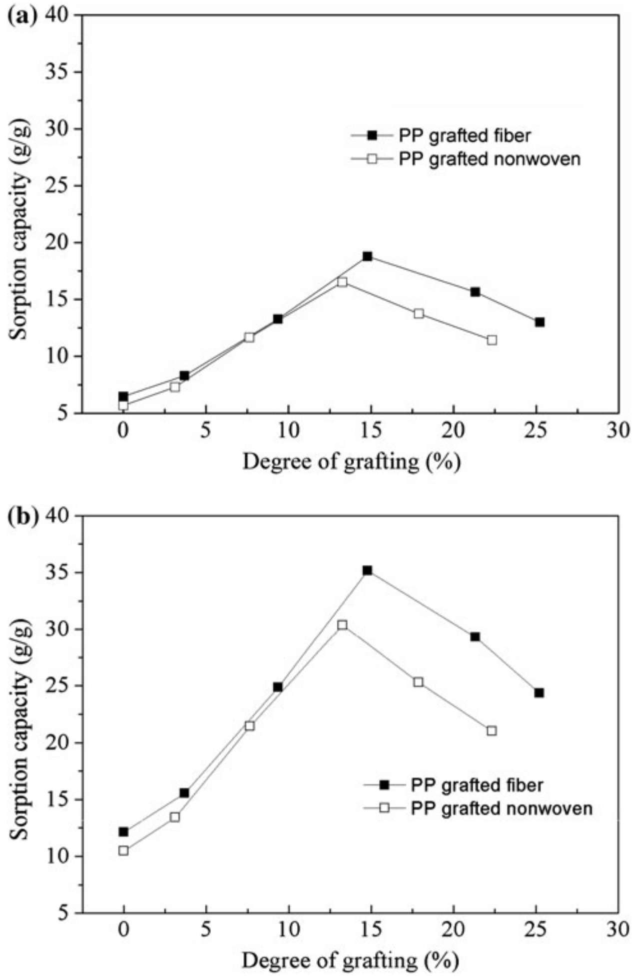

Fig. 4. The effect of the grafting degree on sorption capacity of PP-g-OA in demineralized water medium, (a) sorption for diesel; (b) sorption for crude oil.

Table 2
Surface properties of the PP grafted sorbent with different grafting degree

Sorbent type	Grafting degree (%)	Porosity (%)	Specific surface area (m^2/g)
Grafted PP fiber	0	70.81	0.58
	3.69	78.43	0.63
	9.36	85.12	0.79
	14.79	90.34	0.88
	21.32	87.39	0.78
	25.22	82.10	0.72
Grafted PP nonwoven	0	55.72	0.42
	3.12	62.64	0.45
	7.64	68.73	0.54
	13.26	61.25	0.59
	17.89	57.41	0.53
	22.34	52.32	0.50

the PP grafted fiber with a grafting degree of 14.79%, which has the highest porosity and surface area among all the sorbent tested, had the highest sorption capacity for experiment oil. As shown in Table 2, there appears to be a higher porosity and surface area in which grafted fiber compared to grafted nonwoven with a similar grafting degree. When the grafted fiber and grafted nonwoven were immersed into oil simultaneously, there were more pores and effective sorption site for oil to enter grafted fiber and contribute its complete sorption characteristic, which resulted in a higher sorption capacity. However, we believed that the surface morphology of the PP-based grafted sorbent was not the sole factor, obviously, PP nonwoven sorbents are consolidated fibrous materials and based on webs of individual fibers, and is the incorporation of a substance from one state into another. Therefore, the sorption process of grafted nonwoven usually occurs in these pores from a relatively small area of the external surface. On the contrary, oil sorption of PP fiber is through capillary bridges between fibers, the sorption capacity has been entirely attributed to the structure of sorbent [30]. The oil sorption on PP fiber was not only sorbed by the fiber matrix and grafted OA bulk but also formed oil bridges between surface roughness sites, the oil molecule adheres to the surface of PP fiber matrix and capillary facilitates the transport of oil into the fiber, which endowed grafted fiber achieved a higher sorption capacity than grafted nonwoven. Based on all the consideration above, the higher sorption capacity of PP-g-OA fiber could be attributed to the synergistic effect of all those factors.

Since PP-g-OA may encounter water before oil in a spill cleanup, it is important to know the selected characteristics of grafted PP fiber or nonwoven. In present study, OA was selected as the modifier due to its ester group, which had good hydrophobicity. Then hydrophobic modification of PP matrix was achieved by the surface immobilization of OA assisted by UV irradiation, and the hydrophobicity-oleophilic nature of –COO– groups renders the surface of grafted PP hydrophobic. As shown in Fig. 5, the water sorption-capacity of grafted fiber exhibited a decreased from 2.85 to 0.78 g/g with the grafting degree increasing from 0 to 25.22%; the grafted nonwoven exhibited a decreased from 1.43 to 0.41 g/g with the grafting degree increasing from 0 to 22.34%.

Furthermore, as shown in Figs. 6 and 7, when being applied in oil-over-water experiment, no

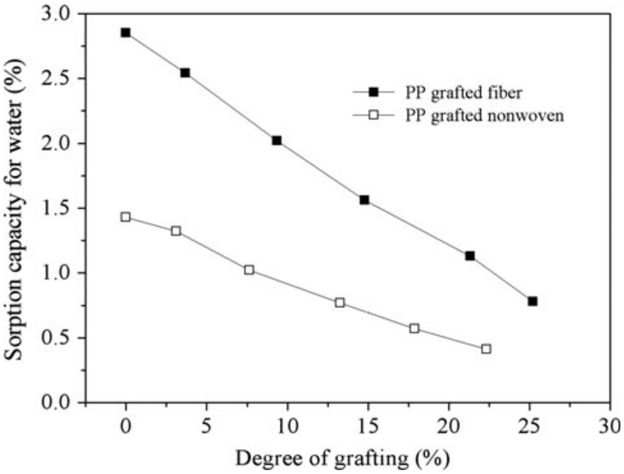

Fig. 5. The sorption capacity of grafted fiber or nonwoven for water with different grafting degree.

significant difference between the sorption capacity of PP-based grafted sorbent for all investigated oil samples in medium of without water, demineralized water, and that in seawater. The results indicate that the hydrophobicity of PP matrix surface was greatly improved after graft modification and exhibited a high selectivity. In addition, the saturation-sorption time is also an important parameter for sorbent evaluation. Figs. 6 and 7 show the graft PP-based sorbent still reserved the fast sorption rate of original PP and exhibited a quick saturation-sorption rate for the oils over the water. Because the advantage on the surface morphology and the capillary facilitates the transport of oil into the fiber, the grafted PP fiber possesses a relatively quicker saturation-sorption rate than PP grafted nonwoven under a same medium. In addition, the sorption process for crude oil reached sorption equilibrium was quicker than diesel, the trend could be attributed to the high viscosity of crude oil, which can inhibit the oil penetration into the sorbent interior, the oil molecular only adherence on the surface of the grafted PP-based sorbent, which reduced the saturation-sorption time. In this study, almost all experiments oils could be removed in 3–8 s by grafted PP-based sorbent and only an invisible oil leakage on water is left. Therefore, it can be concluded that the PP grafted sorbent should be suitable to remove oil from surface of water.

3.3. Oil retention performance of PP-g-OA

The oil retention capacity during field application, transfer, and handling operation is an important parameter for PP-based sorbent evaluation. The dynamic oil retention was measured by allowing the oil to drip from the sorbent for a specified duration. Wei and Fotheringham [20] also divided the retention curve into three distinct zones. In the first stage, the rate of oil dripped out from the sorbent was very high. And then the rate of release is substantially reduced. Finally, the oil in fiber tended to come into a steady-state and this period was much longer. As shown in Fig. 8, regardless of the viscosity, the amount of oil retained on original PP-based was about 28–36%, while those of PP-g-OA was more than 54–72%. The oil retention properties of PP grafted fiber showed the same improved trend with the subsequent increasing of grafting degree. The enhancement in hydrophobicity increased the affinity between PP-based sorbent and oil, which stabilized the interaction force between sorbent and oil, and the capillary

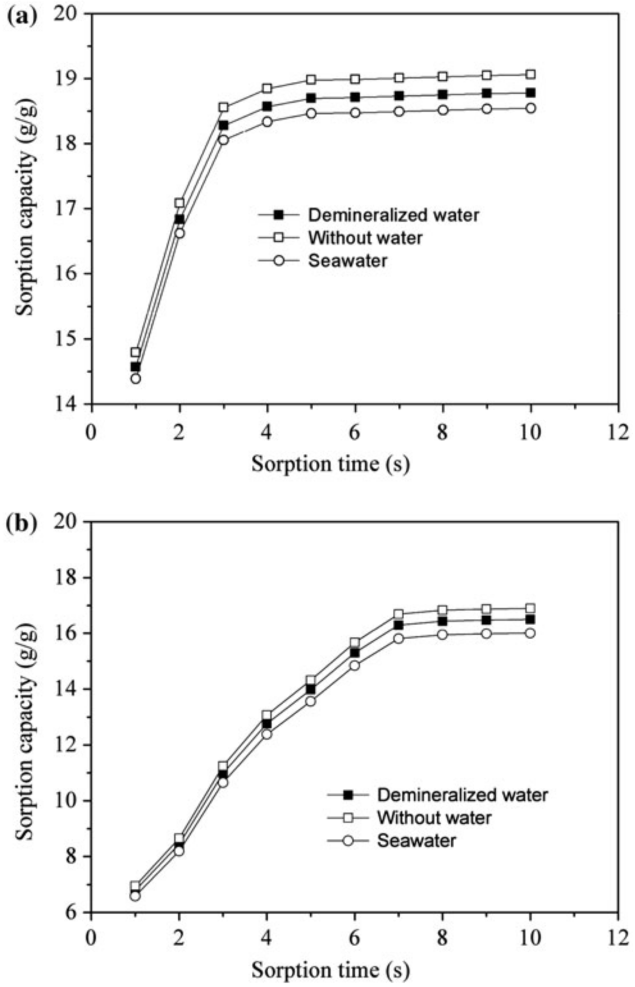

Fig. 6. Sorption capacity of PP-g-OA for diesel depending on sorption time in different medium, (a) grafted fiber with a grafting degree of 14.79%; (b) grafted nonwoven with a grafting degree of 13.26%.

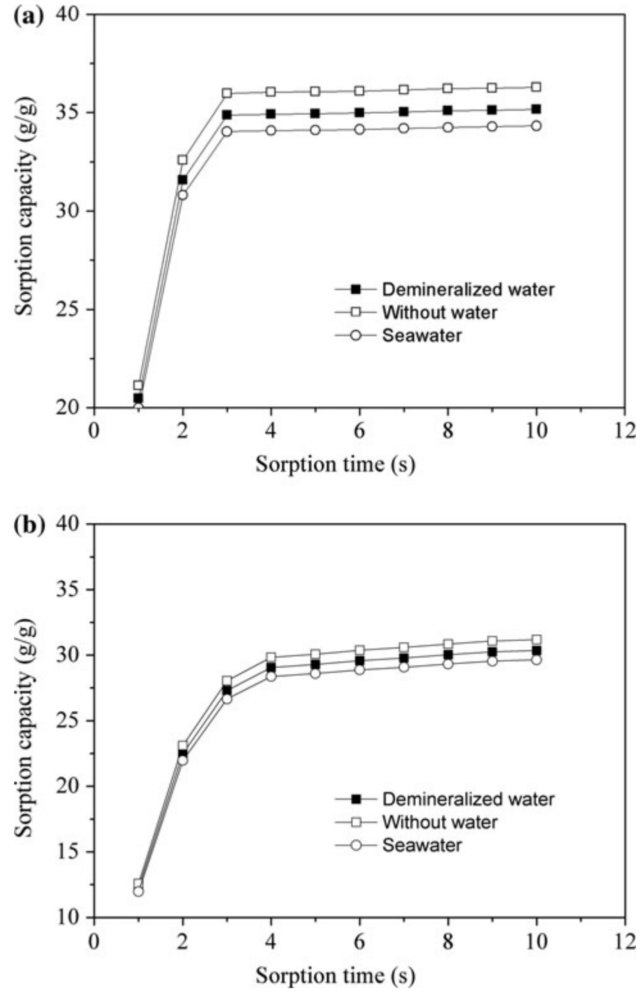

Fig. 7. Sorption capacity of PP-g-OA for crude oil depending on sorption time in different medium, (a) grafted fiber with a grafting degree of 14.79%; (b) grafted nonwoven with a grafting degree of 13.26%.

pressure of the grafted fiber was sufficient to hold the weight of the oil molecule retained in the sorbent matrix and acrylate grafted layer. As shown in Fig. 9, it is well established that the grafted nonwoven have higher dynamic retention capacity than PP grafted fiber. The sorbent construction played an important role in the retention behavior of a sorbent. The PP grafted fiber usually had a relatively higher porosity compared to PP grafted nonwoven, as a result, the oil tended to be released from the fiber sorbent easily, which often leaded to a poor retention capacity and the equilibrium time was only half about that of PP grafted nonwoven [20]. On the contrary, the oil recovery of PP grafted nonwoven was decreased to 60–70% after graft polymerization, and more than 80% of the sorbed oil was removed from the PP grafted fiber by a simple mechanical action, as given in Table 3.

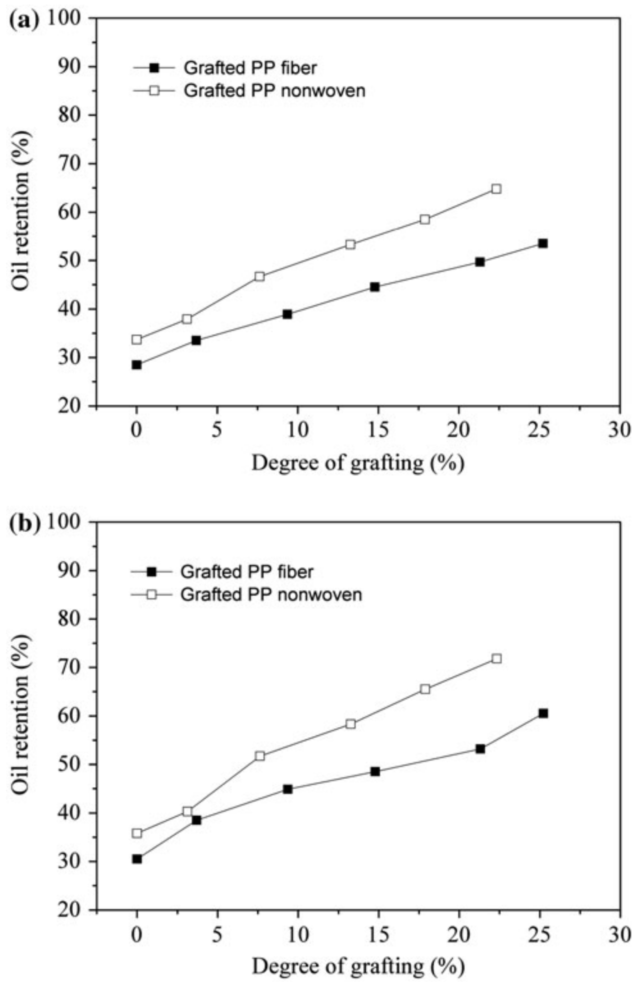

Fig. 8. The oil retention of grafted fiber or nonwoven with different grafting degree, the value was recorded at 60 min, (a) sorption for diesel; (b) sorption for crude oil.

3.4. Reusability performance of PP-g-OA

PP grafted sorbent reusability experiment evaluates the modification PP sorbent durability over prolonged usage, without significant reduction in sorption capacity. Lin et al. found that combined with tire powder, PP fiber could be used repeatedly for at least 100 times [5]. Fig. 10 showed that the reusability of original PP-based sorbent was very poor. For example, the oil sorption capacity of original PP fiber was maintained only about 39.4 and 46.5% for diesel and crude oil, respectively. Lack of elasticity and irreversible deformation of the PP-based sorbent was believed to be one of the main factors causing the reduction in oil sorption capacity. When the original PP-based sorbent was regenerated by squeeze, the structures of PP were destroyed and lost its puffiness, the inter-fiber pores may not be achieved immediately when the sorbents were subsequently soaked into aqueous

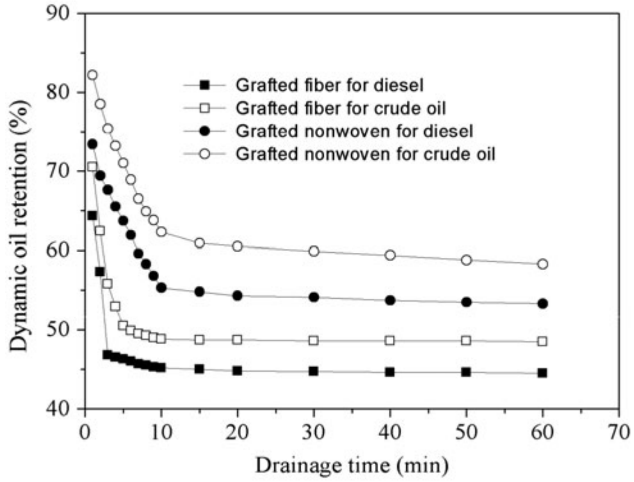

Fig. 9. The percentage of dynamic oil retention of grafted PP sorbent for diesel and crude oil, the grafted fiber with a grafting degree of 14.79%, the grafted nonwoven with a grafting degree of 13.26%.

Table 3
Oil recovery of the PP grafted sorbent with different grafting degree

Sorbent type	Grafting degree (%)	Recovery of diesel (%)	Recovery of crude oil (%)
Grafted PP fiber	0	82.21	72.26
	3.69	86.95	79.42
	9.36	90.63	83.66
	14.79	93.62	84.29
	21.32	88.23	82.55
	25.22	80.16	80.46
Grafted PP nonwoven	0	67.36	57.42
	3.12	63.14	60.42
	7.64	65.78	62.54
	13.26	75.77	67.59
	17.89	72.38	64.53
	22.34	71.31	62.50

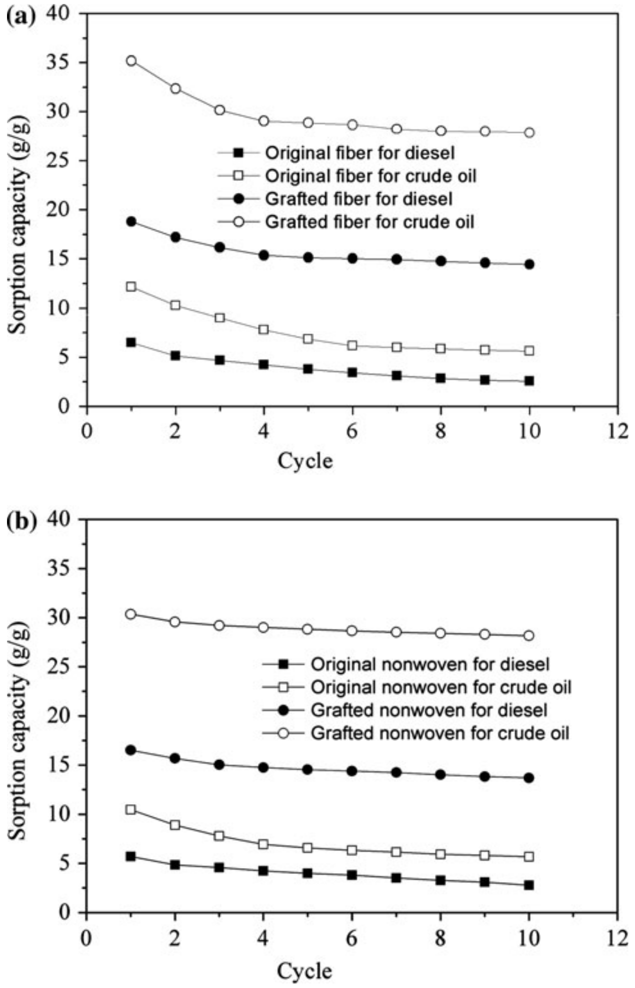

Fig. 10. Reusability of PP-g-OA during tenth sorption cycles, (a) grafted fiber with a grafting degree of 14.79%; (b) grafted nonwoven with a grafting degree of 13.26%.

solution. In addition, the residual organic trapped in the inter-fiber pores was also contributed to the decrease of absorption capacity. Compared to the original PP-based sorbent, it can be seen that the reusability of grafted fibers and grafted nonwovens was improved significantly after graft modification. Though the simple squeezing method would disrupt the initial PP-based sorbent network which reduced the possibility of reusability, the grafting layer could preserve the capillary structure, and as a result, the physical properties did not change significantly after squeezed. The grafted fiber after being regenerated 10 times was still able to maintain its initial sorption capacity about 76.8 and 79.2% for diesel and crude oil, respectively, thus prolonging the lifetime of grafted PP fiber. The reusability of grafted nonwoven also showed the same trend, because the matrix of PP nonwoven is more elasticity than PP fiber and its physical properties do not change when pressed, the reusability of PP grafted nonwoven was better than PP grafted fiber, from the third cycle until the tenth cycle, no significant changes in the sorption capacity were observed.

4. Conclusions

In this study, a new PP sorbent material was prepared through graft polymerization of OA onto polypropylene (PP) fiber or nonwoven initiated by UV radiation. The grafting degree could be controlled by adjusting UV irradiation time, concentration of monomer, and iniferter. Our results showed that the grafted PP sorbent could fully uses the advantages of both PP fibers and acrylate resins, such as fast sorption rate, high sorption capacity, excellent retention capacity, and good reusability. The sorption performance compared of grafted PP fiber and grafted PP nonwoven was especially highlighted throughout our study. In order to enlarge the choice of sorbents for oil removal, a specific task of this study was to analyze, compare, and extract the best features of two types of modification PP sorbents. The results of this study can be considered in making decision, which kind of PP-g-OA sorbent can be efficiently applied for oil removal in a real spillage treatment plant.

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