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Phthalate esters removal using modified fiber prepared by two-step electron beam irradiation of methacrylate monomer onto polypropylene substrate

Shaoning Li[a,b], Junfu Wei[a,c,*], Lei Wang[c], Hang Yang[c], Ao Wang[c], Yuexia Nie[c]

[a]*State Key Laboratory of Hollow Fiber Membrane Materials and Processes, Tianjin Polytechnic University, Tianjin 300387, China*
Email: jfwei1963@yahoo.cn
[b]*School of Material Science and Engineering, Tianjin Polytechnic University, Tianjin 300387, China*
[c]*School of Environmental and Chemical Engineering, Tianjin Polytechnic University, Tianjin 300387, China*
Tel. +86 022 8395 5898; Fax: +86 022 8395 5451; email: jfwei1963@yahoo.cn

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ABSTRACT

A novel modified polypropylene-methacrylate fiber (PP-g-(AA+BMA)) was prepared by grafting acrylic acid (AA) and butyl methacrylate (BMA) onto the polypropylene (PP) fiber substrate using a two-step electron beam irradiation method. It was used as a sorbent to recover high concentrations of phthalate esters (PAEs) from water. AA was first grafted onto PP substrate by electron beam, and then the pretreated PP was soaked into BMA solution and the loaded monomer was grafted on the PP substrate by electron beam induced. Fourier transform infrared spectra and contact angle (CA) were employed to characterize the chemical changes of PP substrate. In addition, the effect of the first and the second reactions on the degree of grafting was studied. The results indicated that the PP-g-(AA+BMA) could successfully be used to remove PAEs from water, leading to superior improvement of environmental preservation.

Keywords: Phthalate esters; Sorption capacity; Graft polymerization; Polypropylene fiber; Butyl methacrylate

1. Introduction

Over the past several decades, phthalate esters have been widely used with the continuing progress of the industrial development [1–3]. The stability, fluidity, low volatility, and higher molecular weight make phthalate esters highly suitable as plasticizers. Extensive application of PAEs increases the possibility of their release into the water environment. PAEs are rather stable compounds in the natural environment, and their hydrolysis half-life is estimated to be about 20 years. In addition, some of the PAEs were classified as the top priority pollutants for the risk assessment, and the release of PAEs into the water has caused serious toxicological effects on aquatic life and hence on humans [3–4]. Nowadays, removal of phthalate esters contaminants from drinking water continues to be a central problem in environment remediation.

Sorption is one of the most effective techniques for the remediation of organic spillages [5–6]. Although many sorbents have been developed to solve this

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*Corresponding author.

problem, few are successfully applied in a sustainable manner due to the economic and technical limitations. Nowadays, much attention is being paid to innovation and development of the modified fiber sorbents [7–9]. Though many kinds of methacrylate polymer have been synthesized to sorb organic matter, there still have been few reports on using methacrylate sorption-fiber that is directly prepared by these kinds of polymers. In fact, these kinds of polymers cannot be made into fiber because the methacrylate monomer can neither dissolve into organic solvent nor be melted [10–12].

This work aims at evaluating the possibility of removing PAEs from water by a novel fiber sorbent PP-g-(AA+BMA). The preparation of sorption-fiber is based on butyl methacrylate monomer and low-density polypropylene substrate by two-step electron beam irradiation. In general, the electron beam irradiation can form radicals on the end-groups of polymer chains leading to the formation of long branches through sequential grafting between radicals and monomers [13]. The PP fiber was chosen as a matrix for the grafting reaction because its capillary structure could facilitate the transport of PAEs molecules, and, therefore, exhibit a fast sorption rate [8,9,14]. The achieved PP-g-(AA+BMA) could exhibit an excellent sorption performance, fast saturation-sorption rate, and adequate reusability, which provides a very competitive sorbent for the removal of PAEs from water.

2. Experimental

2.1. Materials

The polypropylene fiber was kindly offered by Nanjing Haidexin Technolgy, Co., Ltd. (Jiangsu, China), whose properties are presented in Table 1. Acrylic acid (AA, purity ⩾99%) and butyl methacrylate (BMA, purity ⩾99%) were purchased from Zibo Haipeng Fine Chemical Co., Ltd. (Shandong, China). Dimethyl phthalate (DMP, purity⩾99⩾%) and diethyl phthalate (DEP, purity ⩾99%) were obtained from the Sheng Da Chemical Reagent Co., Ltd. (Tianjin, China). Their properties are presented in Table 2 and the molecular structure is shown in Fig. 1. Photosensitizer benzophenone (BP, purity ⩾98%) was obtained from Tianyi Chemical Reagent Institute Co., Ltd. (Tianjin, China). All other chemicals including isopropanol and ethanol were of analytical grade and purchased from Tianjin Kermel Chemical Reagent Co., Ltd. (Tianjin, China). All reagents and solvents were used as received.

Table 1
Physical properties of PP fiber

Sample	PP fiber
Fiber diameter (μm)	19∼21
Initial modulus (cN/dTex)	26.51
Density (g/cm^3)	0.91

Table 2
Properties of DMP and DEP

Formula	DMP $C_{10}H_{10}O_4$	DEP $C_{12}H_{14}O_4$
Molecular weight (g/mol)	194.19	222.24
Density (g/cm^3)	1.191∼1.195	1.118∼1.122
Acidity (%)	⩽0.01	⩽0.01
Content of water (w%)	⩽0.01	⩽0.01

2.2. Preparation of PP-g-(BA+BMA)

Electron beam treatment of the samples was carried out using a resonance-transforming electron accelerator RTE-1V (produced by the Efremov Institute of Electrophysical Apparatus, St. Petersburg, Russia) at the energy 700 keV, current 0.5 mA, and irradiation dose values 10, 20, 30, 40, and 50 kGy. The schematic of electron beam reactor is presented in Fig. 2.

The procedure for the preparation of PP-g-(AA+BMA) is as follows:

1. *Pretreatment*: The PP substrate 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.
2. *The first grafting step*: Isopropanol/water solution (if there is no special explanation, the

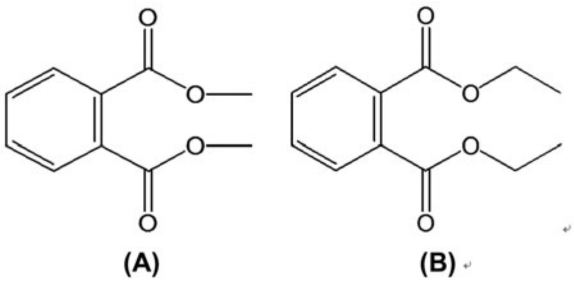
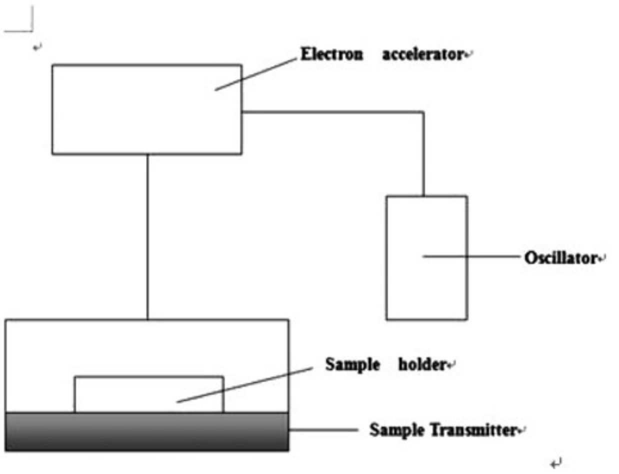

(A) (B)

Fig. 1. Molecular structures of (A) dimethyl phthalate and (B) diethyl phthalate.



Fig, 2. Schematic of the electron beam reactor used for induced graft polymerization.

proportion of isopropanol/water is 1:4) as solvent was added first, followed by initiator (BP, 0.625‰ based on the weight of monomer), predetermined concentration of AA monomer, and the oxygen which was purged by bubbling nitrogen for 10 min. After the first grafting, the electron beam was irradiated for a given time to carry out the graft polymerization of PP-g-AA. The unreacted monomer and homopolymer were removed from the sample by extraction with ethanol at room temperature for 8 h.

(3) *The second grafting step*: The PP-g-AA samples were then put into the monomer of BMA grafting solution for the grafting reaction again, while leaving a sample as a control. After grafting, the achieved PP-g-(BA+BMA) samples were extracted with ethanol again.

For comparison, both one-step method and two-step one were employed to conduct the graft polymerization of AA/BMA onto PP substrate; the sample graft polymerization of BMA directly was also prepared. The grafting conditions are shown in Table 3.

Table 3
The recipe for preparation of PP-g-(AA+BMA)

	Irradiation dose (kGy)	Irradiation time (min)	BMA concentration (v/v)
Typical recipe	30	45	20%
Parameters	10∼50	0∼60	1.25∼20%

The grafting degree (DG) was calculated by the following equation:

$$\mathrm{DG}\ (\%) = [(W_1 - W_0)/W_0] \times 100\% \qquad (1)$$

where, W_0 and W_g are the weights of dried PP sample before and after graft polymerization, respectively. All the results are the average of five parallel experiments.

2.3. Characterization of PP-g-(AA+BMA)

Infrared spectra were obtained with Fourier transform infrared spectra (VECTOR22, Bruker Co), and the measurements were carried out in a range of 3,000–1,000 cm^{-1}. The contact angel measurements were performed on Cam 100 optical angle meter (YH-168A instruments Ltd., Japan). The tensile test was performed by using universal electronic tensile instrument (LLY-06, China) at 25℃, 65% RH. The PP monofilament with a length of 10 mm between clamps was stretched at a rate of 20 mm = min until the sample broke.

2.4. Sorption test

In the study of removal of trace PAEs from water by PP-g-(AA+BMA), accurate amount of 0.5 g DMP or DEP was poured into a 500 ml volumetric flask, then diluted with distilled water and ethanol to achieve the standard solution. UV absorption at 224.0 nm was used for quantitative estimations. The sorption properties of PP-g-(AA+BMA) were evaluated by the sorption of trace PAEs (the concentration is 1 ppm) from the aqueous system at different grafting degrees, contact time, and pH values of the aqueous system. When the sorption capacity of the grafted fiber was tested, the degree of grafting varied from 0 to 17.24%. The sorption time ranged from 0 to 90 s, and pH values of organic-over-water solution were conducted within the range of 1.00–10.00. For temperature influence on sorption capacity, the analyses were performed from 298 K to 318 K in steps of 5 K.

3. Results and discussion

A new modification of fiber sorbent was prepared by graft polymerization of AA/BMA monomers onto the surface of PP substrate. The method consists of an electron beam irradiation in the presence of acrylic acid for an *in situ* graft polymerization of butyl methacrylate (BMA) by the two-step method.

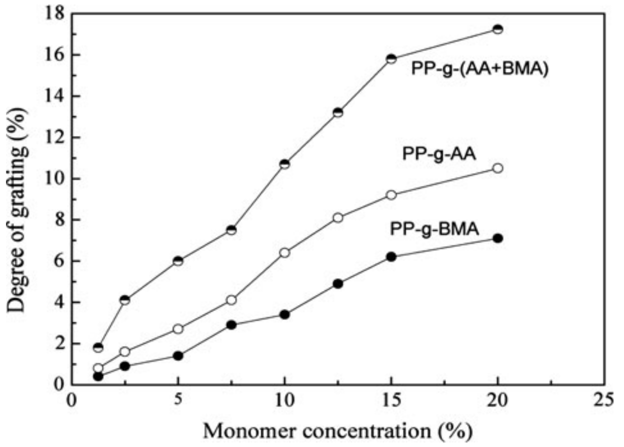


Fig. 3. Effect of monomer concentration on the grafting degree of PP-g-(AA+BMA) (AA was grafted in first step, BMA was grafted in second step, the remaining grafting conditions: irradiation dose values 30 kGy at 60℃ for 45 min).

3.1. Influencing factors in preparation of PP-g-(AA+BMA)

Fig. 3 shows the effect of AA concentration on the degree of BMA grafting. With the increase of monomer concentration, the surface free radical in PP substrate had more chances to contact with monomers and increased the possibilities of the reaction between monomer and PP, which achieved a higher grafting degree [15–17]. The grafting degree of BMA increased with the increase of the AA grafting degree. The grafting degree of BMA onto the PP-g-AA increased significantly compared to that onto the PP substrate directly by one-step method. The trend may result from the BMA monomer being easily grafted onto PP-g-AA matrix, and the amount of BMA grafting degree increased with the increasing of the AA monomer grafted onto the surface of PP substrate. When the grafting degree of AA was 10.50%, the grafting degree of PP-g-(AA+BMA) reached the maximum of 17.24% with BMA monomer concentration of 20%. Therefore, if there is no special explanation, the optimal AA grafting degree of 10.50% was selected for the following experiments.

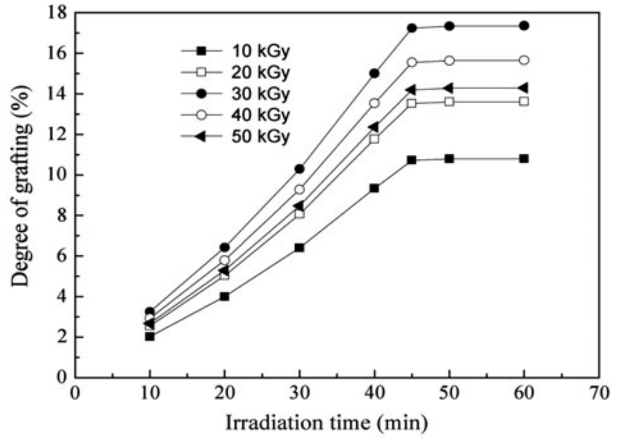


Fig. 4. Effect of irradiation dose and irradiation time on the grafting degree of PP-g-(AA+BMA) (AA was grafted in first step, BMA was grafted in second step, the remaining grafting conditions: BMA monomer 20% at 60℃).

Next, we studied the effects of irradiation dose and irradiation time on the grafting degree of polymerization of BMA monomer onto PP-g-AA substrate. As shown in Fig. 4, in this region of low absorbed dose (10∼30 kGy), the grafting degree increased with the increase in the absorbed dose. This is a consequence of the increasing number of active sites within the polymer matrix [13,15,18]. When the absorbed dose was 30 kGy, the grafting degree of PP-g-(AA+BMA) reached the maximum of 17.24%. However, when the absorbed dose was over the critical value, the grafting degree decreased continuously from 17.24 to 14.20% with the increase of the absorbed dose from 30 to 50 kGy. One of the reasons for this trend was the homopolymerization of methacrylate, which hindered the monomer diffusion to the radical sites and subsequently resulted in the decrease of the grafting degree with the sequent increasing of the absorbed dose. In addition, a large amount of the absorbed dose could also damage the mechanical properties of PP fiber substrate. In this case, the grafting degree was saturated in 45 min. The grafting degree increased dramatically with the increase of irradiation time at first. Following the prolongation of irradiation time, there were more active sites activated on the PP-g-AA matrix surface and much more BMA monomers accessible to the surface radicals resulted in the increase of grafting degree [16–18]. However, with the irradiation time further prolonged, the consumption of grafting reagent made the BMA monomer ineffectively grafted on the PP-g-AA matrix, and leaded to an asymptotic saturation of the grafting degree.

In consequence, the higher grafting degree of the PP-g-(AA+BMA) may be obtained for AA first, followed by grafting BMA. Using this method, the grafting degree can be easily adjusted by controlling the reaction conditions. According to the results of this study, the optimal monomer concentration, irradiation dose, and irradiation time were 20%, 30 kGy, and 45 min for a moderate grafting degree. The grafting degree of PP-g-(AA+BMA) under the optimal conditions was evaluated to be 17.24%.

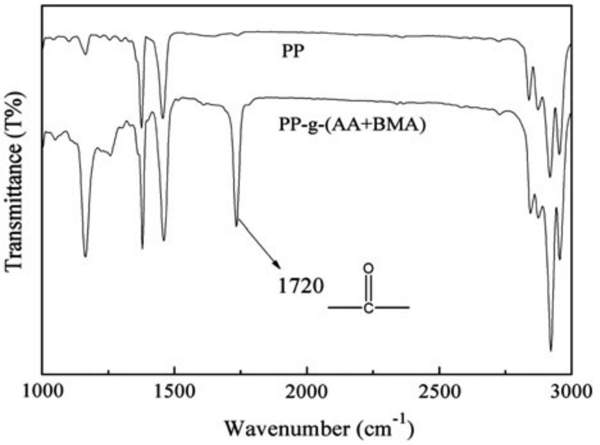


Fig. 5. Infrared spectra of original PP and PP-g-(AA+BMA) with grafting degree of 17.24%.

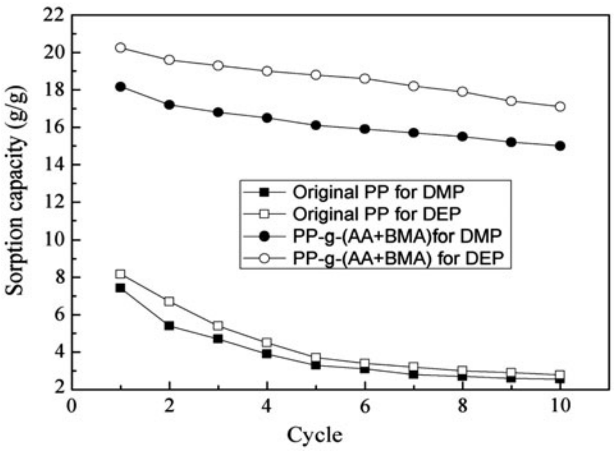


Fig. 6. The sorption capacity of PP-g-(AA+BMA) during 10th sorption cycles.

3.2. Characterization of PP-g-(AA+BMA)

The chemical change of PP substrate was monitored by FT-IR spectra. Fig. 5 shows the differential spectrum which is obtained from the original PP substrate and the PP-g-(AA+BMA). The spectrum was in good agreement with the original PP. There were strong bands at 1,458 and 1,379 cm^{-1} in PP-g-(AA +BMA) corresponding to the bending vibrations of CH_2 for both original PP and the grafted PP samples. As illustrated in Fig. 6, a major change on the PP substrate surface was the obvious appearance of ester carbonyl absorbances at a wavenumber of 1,720 cm^{-1}, which was attributed to the stretching vibrations of C=O [9].

3.3. Sorption test of PP-g-(AA+BMA)

As shown in Table 4, the water contact angle exhibited an increase from 75.30° to 130.17° with the increase of the grafting degree from 0 to 17.24%, which indicated that the hydrophobicity of the original PP substrate surface was greatly improved after graft polymerization. With the subsequent increase of the grafting degree, the sorption capacity and contact angle showed the same trend, indicating that the hydrophobic properties play an important role in the sorption behavior of PP-g-(AA+BMA), and the improved sorption capacity of PAEs can be attributed to the strong sorbate–sorbent hydrophobic interactions by BMA grafting layer. It was found that when the grafting degree was 17.24%, the maximal sorption capacity for DMP reached 18.17 and 20.25 g/g for DEP at room temperature, thus all sorption experiments in the present study were carried out at initial grafting degree of 17.24%.

Furthermore, the reusability is one of the important properties of the PP-based sorbent. When the original PP fiber was regenerated by squeezed method, the capillary structures of PP fiber were destructed, and the inter-fiber pores may not be achieved immediately when the sorbents were subsequently soaked into aqueous solution. In addition, the residual organic trapped in the inter-fiber pores was also contributed to the decrease of the sorption capacity. The sorption capacity of the original PP for the fifth cycle was only about 40% of that for the first one (Fig. 6).

Table 4
The sorption capacity and water contact angle of PP-g-(AA+BMA) with different grafting degrees

Degree of grafting (%)	Water contact angle (°)	Sorption capacity for DMP (g/g)	Sorption capacity for DEP (g/g)
0	75.30 ± 2.31	7.42 ± 0.14	8.17 ± 0.12
6.1	83.21 ± 1.48	11.21 ± 0.21	12.48 ± 0.15
10.7	99.40 ± 0.92	13.42 ± 0.17	14.43 ± 0.26
13.2	110.45 ± 2.17	15.17 ± 0.22	16.85 ± 0.17
15.8	122.48 ± 1.31	16.78 ± 0.09	18.43 ± 0.21
17.24	130.17 ± 0.72	18.17 ± 0.12	20.25 ± 0.23

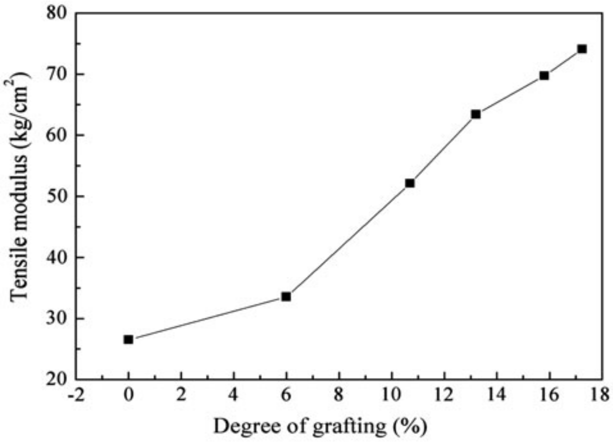


Fig. 7. The tensile modulus of PP-g-(AA+BMA) with different grafting degree.

As shown in Fig 7, the tensile modulus of PP-g-(AA+BMA) increased from 26.51 to 74.11 kg/cm^2 with the grafting degree increased from 0 to 17.24%, which indicated that the physical properties were improved significantly than that of the original PP after graft polymerization. This trend may have resulted from the following two aspects: (a) litter electron beam energy was absorbed by PP substrate by electron beam irradiation and (b) BMA grafted onto the surface of PP helped the original PP substrate to maintain its elasticity and capillary, which can be of large help to the physical characteristic of the PP fiber. As a result, though the squeezed method would disrupt the initial nature of fiber network, the grafting layer could preserve the capillary structure of original PP and the physical properties did not change significantly in spill cleanup, thus prolonging the lifetime of grafted fiber [14,21]. The PP-g-(AA+BMA) after being regenerated 10 times was still able to maintain about 80% of its initial sorption capacity.

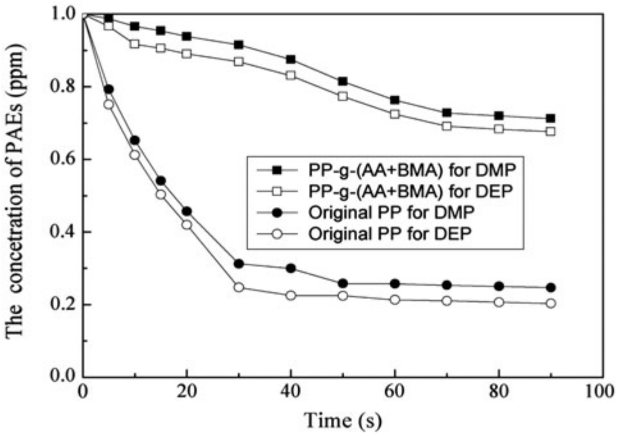


Fig. 8. The removal effect of original PP and PP-g-(AA+BMA) for trace DMP or DEP (the concentration is 1 ppm) from water (pH = 8.0 and 318 K).

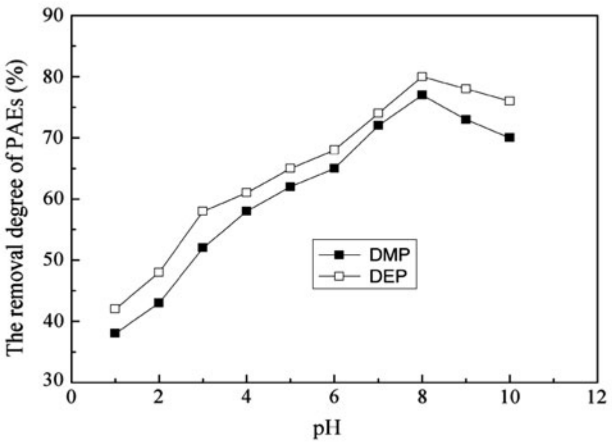


Fig. 9. The effect of the pH of aqueous solution on removal degree of PP-g-(AA+BMA) for trace PAEs (the concentration is 1 ppm) from water (318 K, sorption time = 30 s and degree of grafting = 17.24%, respectively).

However, for the real release into the ecosystem or wastewater effluent, the phthalate esters pollutants tend to accumulate in a complicated aqueous system. The sorption performance for the removal of trace DMP or DEP (the concentration is 1 ppm) from water with PP-g-(AA+BMA) was investigated. As shown in Fig. 8, the sorption equilibrium of PP-g-(AA+BMA) decreased to about 30 s which was only half about that of the original PP fiber. The PP-g-(AA+BMA) exhibited an excellent removal degree regardless of the sample used, which was about 2.8 times as high as that of original PP fiber indicating that the grafted

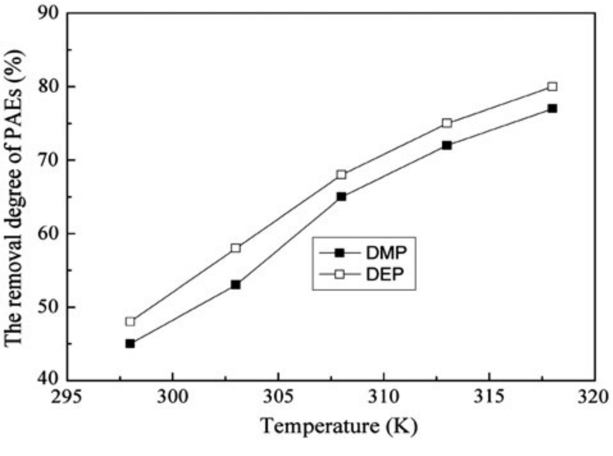


Fig. 10. The effect of the temperature of aqueous solution on removal degree of PP-g-(AA+BMA) for trace PAEs (the concentration is 1 ppm) from water (pH = 8, sorption time = 30 s and degree of grafting = 17.24%, respectively).

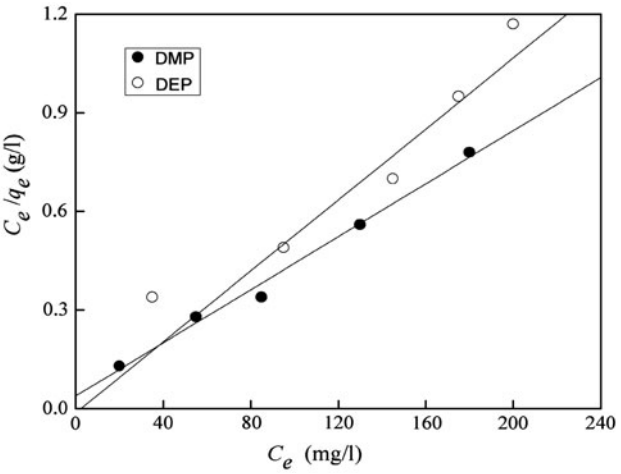


Fig. 11. Curve fitting of q_e^{-1}–C_e^{-1} from Langmuir plot for the removal of trace PAEs by PP-g-(AA+BMA) with a grafting degree of 17.24%.

fiber could also be successfully applied in removal of trace PAEs from aqueous system (Fig. 9).

There are several factors that influence the sorption performance of PP-g-(AA+BMA) for trace PAEs. Among these factors, the pH of the sample aqueous solution is a determining factor. To determine the optimal condition at which PAEs molecule are effectively sorbed on the PP-g-(AA+BMA), the sorption experiments were carried out at different pH values ranging from 1.0 to 10.0. As shown in Fig. 11, the removal degree for PP-g-(AA+BMA) was continuously increased by increasing pH value from acidic to alkali medium; the maximal removal degree was obtained when pH of complicated aqueous system was 8.00 and it decreased by either raising or lowering the pH. This trend would be due to the BMA grafting chains hydrolysis in acidic medium; the variation of pH can affect the surface charge of the PP-g-(AA+BMA) and may be attributed to the completion behavior between hydrogen and active group (–CO–CH_2–) on grafted fiber surface [19–21].

The effect of aqueous solution temperature on the sorption performance of PP-g-(AA+BMA) for trace PAEs was studied over the range of temperature from 298 to 318 K in steps of 5 K, while other parameters were kept constant (sorption time of 30 s, degree of grafting = 17.24% and pH = 8.0). As shown in Fig. 10, the result obviously indicates that the removal degree for trace PAEs was considerably increased with the temperature going up, which implies that the sorption process is an endothermic process [22].

As shown in Figs. 11 and 12, all experimental data could fit into the Freundlich sorption model. The

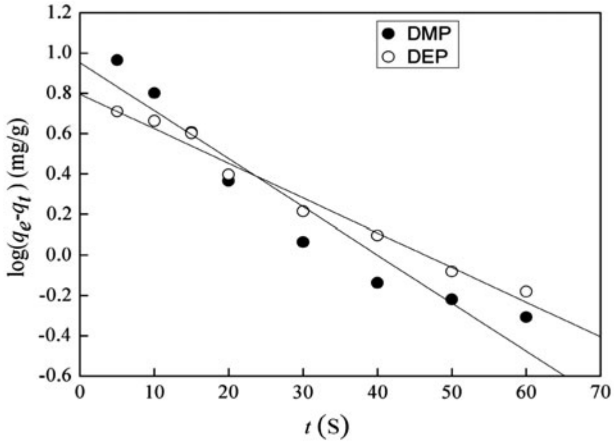


Fig. 13. Relation curves of pseudo-first-order plot obtained by linear fit, the PP-g-(AA+BMA) with a grafting degree of 17.24%.

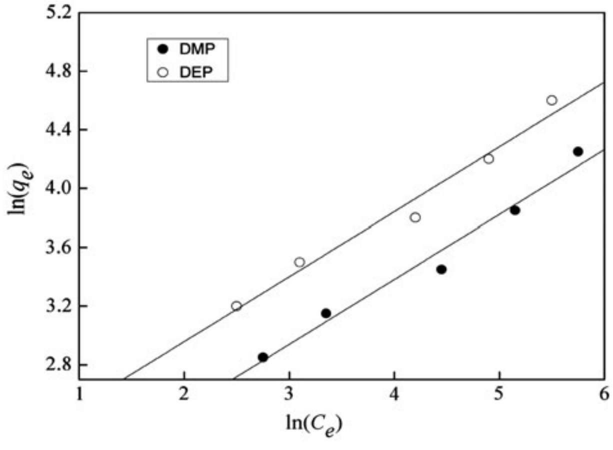


Fig. 12. Curve fitting of ln q_e—ln C_e from Freudlich plot for the removal of trace PAEs by PP-g-(AA+BMA) with a grafting degree of 17.24%.

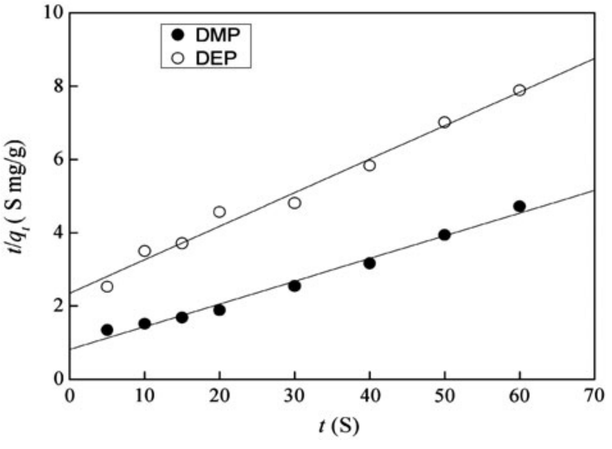


Fig. 14. Relation curves of pseudo-second-order plot obtained by linear fit, the PP-g-(AA+BMA) with a grafting degree of 17.24%.

Table 5
Langmuir and Freundlich sorption isotherm constants for removal of trace PAEs by PP-g-(AA+BMA) with a grafting degree of 17.24%

Degree of grafting (%)	First-order kinetics		Second-order kinetics	
	k_1 (s^{-1})	R^2	k_2 (g/mg s)	R^2
DMP	−0.01943	0.9138	0.9235	0.9687
DEP	−0.02415	0.9162	0.9167	0.9921

obtained data imply that sorption of PAEs molecules by the PP-g-(AA+BMA) is a monolayer type and PAEs molecules from the aqueous solution usually form a layer on the surface of the grafted fiber substrate. The fact implies that the sorption of PP-g-(AA+BMA) for trace PAEs from water is complex, involving more than one mechanism [23].

In order to investigate the sorption performance of PP-g-(AA+BMA) for trace PAEs, the pseudo-first-order and pseudo-second-order models were tested to fit the kinetic data. The kinetics data are plotted as the linear form of the models (Figs. 13 and 14), and the comparison between the experimental sorption capacity and the theoretical values estimated from the second-order equation is presented in Table 5. The second-order equation fitted well with the experimental data and the correlation coefficients of first-order kinetics model were also found to be slightly lower. These results showed that the second-order kinetics could better describe the data than the first-order kinetic model.

4. Conclusions

In this study, a novel polymeric sorbent of PP-g-(AA+BMA) was obtained after modifying PP fiber substrate using two-step electron beam irradiation method. The enhancements of sorption behavior were interpreted concerning certain improvement in the physiochemical properties of PP substrate. PP-g-(AA+BMA) exhibited a faster sorption rate, higher sorption capacity, and better physical mechanical properties. The obtained results showed that the removal of PAEs was highly dependent on the grafting degree of PP-g-(AA+BMA), sorption time, pH, and temperature of aqueous solution. In addition, the sorption behavior could be modeled using the Freundlich isotherm, and the kinetics studies showed that the sorption process obeyed pseudo-second-order kinetics. In conclusion, the PP-g-(AA+BMA) can be favorably deployed as a sorbent for the removal of PAEs spillage.

Acknowledgments

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