Surface grafting of styrene on polypropylene fibers by argon plasma and its adsorption-regeneration of BTX

J J Xu1, M L Guo2, Q G Chen1, Z Y Lian1,4, W J Wei1,4, Z W Luo3, G Xie1, H N Chen1 and K Dong1

1School of Environment Science and Engineering, Nanjing Tech University, Nanjing 211800, China
2School of Chemical Engineering, Nanjing Tech University, Nanjing 211800, China
3School of Safety Science and Engineering, Nanjing Tech University, Nanjing 211800, China

E-mail: lianzy@njtech.edu.cn; wjwei@njtech.edu.cn

Abstract. Active macromolecular free radicals were generated on polypropylene (PP) fibers surfaces by argon (Ar) plasma irradiation, then, PP surface modified fibers (PP-g-St fibers) were prepared by in-situ grafting reaction of styrene monomers (St). Effects of reaction parameters on grafting percentage were studied and adsorption capacities of PP-g-St fibers for benzene, toluene and xylene (BTX) were evaluated. Afterwards, regeneration adsorption efficiencies after maximum adsorption were explored. The results indicated that the optimum input power, irradiation time and grafting reaction time are 90 W, 3 min and 3 h respectively and the grafting percentage of St reached 5.7 %. The adsorption capacities of PP-g-St fibers towards toluene and xylene emulsions and solutions in water increased by 336.89 % and 344.57 % respectively, compared to pristine PP fibers. In addition, regeneration adsorption efficiencies of modified fibers remained > 90 % after six cycles of regeneration-adsorption experiments, which showed excellent regeneration ability.

1. Introduction
Benzene, toluene and xylene (BTX) are typical pollutants in waste waters, discharged either from the industries, or during transportation and storage [1-3]. They generally form emulsion with water. Due to their low degradability and highly poisonous nature, they pose a great threat to the environment and to human health if discharged without treatment [4, 5]. The main methods, till date, for treatment of BTX in wastewaters include biodegradation [6], thermal oxidation, catalytic oxidation, condensation and separation, membrane separation and adsorption [7]. Due to several advantages such as simple operation, no secondary pollution, fast processing and renewability of BTX, adsorption is considered to be one of the most efficient methods for treatment of organic pollutants in wastewaters [8-11]. The adsorption materials can be either inorganic (such as diatomite, attapulgite, volcanics), natural organic fibers (such as cotton, linen and wool) or synthetic organic nature (such as macroporous resin, polymeric fibers and polyurethane foams) [12].

Due to their characteristic large specific surface areas, good hydrophobic and lipophilic properties, good rigidity, and stable physicochemical properties, polypropylene (PP) fibers, as a kind of synthetic organic adsorption materials are widely used for the treatment of oily wastewaters [13, 14]. However, the affinity between PP fibers and BTX is low [15], due to difference in their solubility parameters.
which limits the adsorption of BTX, especially in case of BTX emulsion and solution in water. Hence, it is necessary to modify the PP fibers surface to improve their affinity. Melt grafting, solid grafting, liquid-phase grafting and suspension grafting are main modification methods that are practically useful [16-19], but these methods have some drawbacks such as severe degradation of PP, high cost, non-fibrous nature of the modified product, and are also environmentally harmful. Plasma-induced grafting, an excellent technics of surface modification, has been developed recently. It has many outstanding features like mild process conditions, environmentally-friendly nature, and it also causes no damage to the internal structure of the material [20-22]. The principle of this technique is to produce active sites (free radicals) by bombarding the material surface with energetic ionizing particles, where in-situ grafting reaction of monomers takes place [23].

Polystyrene (PS) has a large number of styrene (St) units in its molecular chain, which is similar to the molecular structure of BTX, and their solubility parameters are also close [24]. So, PS has excellent adsorption and occlusion for BTX. However, the swelling and dissolution of PS in BTX causes deformation and loss of mechanical properties of PS fiber. Moreover, it is difficult to separate the BTX occluded inside the PS fibers, and its regeneration is difficult [25].

In this paper, by combining the advantages of PP and PS for adsorption of BTX, active macromolecular free radicals were generated on PP fiber matrix using argon (Ar) plasma irradiation. The in-situ grafting reaction of St monomers occurred and PP-g-St fibers were formed. The effects of reaction parameters including input power, irradiation time and grafting reaction time on grafting percentage were studied. Adsorption capacities of PP-g-St fibers for BTX emulsion and solution in water were evaluated, and their maximum adsorption capacities for pure BTX were measured as well. Finally, regeneration methods and regeneration adsorption efficiencies after maximum adsorptions were explored.

2. Experimental

2.1. Materials
PP melt-blown fibers (laboratory prepared, fibers diameter: 2-10 μm, specific area: 1.50 m²/g). St (CP grade), was distilled under reduced pressure prior to use. Benzene, toluene, xylene, acetone and anhydrous sodium sulfate were all analytical grade. Sodium dodecyl benzene sulfonate (SDBS) was of CP grade, carbon tetrachloride was specially produced for environmental protection, petroleum ether was of industrial grade. 500 W radio frequency power source, MSY-I and 2000 W radio frequency matcher, SP-II (plasma regenerator) were all provided by microelectronics institute of Chinese Academy of Sciences. Oil concentration analyzer ET1200 was purchased from Euro Tech Co., Ltd., Shanghai.

2.2. Preparation of PP-g-St fibers
The set-up for the preparation of PP-g-St fibers by plasma-induced grafting is shown in figure 1. The process can be described in following steps:

- PP fibers were immersed in acetone for 2 hours and dried to a constant weight at 50°C. then some PP fibers were taken as the sample and weighed. They were then transferred to reactor, the reactor was placed between electrodes of the plasma regenerator and connected to the system.
- Valve B was closed, Solution of the St monomers was taken in the separating funnel and Ar gas was bubbled into the solution to eliminate air.
- Valves A and C were then opened and air was removed until the pressure was 5 Pa. The air in reactor was replaced with Ar gas for 3 times. Ar gas was again pumped in and evacuated to 5 Pa. Valves A and C were closed, and the plasma generator was switched on to irradiate the fibers under set plasma conditions.
- The plasma generator was turned off and the valves B and C were opened to
guide the solution of St monomers into the reactor ③. Valve B ⑥ should be closed when fibers sample ⑩ is totally immersed in St monomers solution. This was the beginning of grafting reaction. The grafting reaction ended when valve B ⑥ was opened again to introduce air. After that, fiber sample ⑩ was taken out and extracted with acetone for 5 hours to get rid of the remaining unreacted St and the self-polymer (PS). Finally, fiber sample ⑩ was dried at 50°C till it attained constant weight.

Figure 1. The set-up for the preparation of PP-g-St fibers by plasma-induced grafting. ①: vacuum pump, ②: plasma generator, ③: valve A, ④: mental air inlet pipe, ⑤: liquid separating funnel for St, ⑥: valve B, ⑦: valve C, ⑧: reactor, ⑨: electrodes, ⑩: fibers sample.

2.3. Determination of grafting percentage
The grafting percentage can be calculated using equation (1).

\[ \eta = \frac{(M_2 - M_1)}{M_1} \times 100\% \] (1)

Where, \( \eta \) (%) is the grafting percentage, \( M_1 \) (g) is the weight of PP fibers, \( M_2 \) (g) is the weight of PP-g-St fibers.

Scheme 1. Synthesis of PP-g-St fibers.
2.4. Adsorption for BTX

Adsorption capacities of PP-g-St fibers for BTX emulsion and solution in water were measured by following procedure. First, approximately 1 g of PP-g-St fibers sample was weighed and added into 2 L beaker containing 1.5 L deionized water, 20 ml BTX and 0.2 g SDBS, after stirring for 1 h and left to stand for 2h (25°C) to achieve well-distributed and stable, Excess amount of BTX floating on the water surface was removed and the initial concentration of adsorption solution was testified. Then, the PP-g-St fibers were fastened between the annular metal grids and immersed in the mixture for 1 h with low speed agitation using magnetic rotor to fully adsorb BTX emulsion and solution in water. After that, the PP-g-St fibers were fastened and transferred to a separating funnel containing a certain amount of CCl4 to extract the BTX adsorbed, the solution was adjusted pH≤2 and oscillated for 3 min. The water present in the extract of PP-g-St fibers was removed by adding 1.0 g of anhydrous sodium sulfate (extra addition needed if they all crystallize), then filtered the solid and the volume for extraction was adjusted to 200 mL and maintained for 3 h. Finally, the concentration of BTX in the extract was determined using the oil concentration analyzer. Adsorption capacity of PP-g-St fibers can be calculated using the equation (2) (the test was repeated for 5 times to obtain mean value and the adsorption capacity of original PP fibers was measure for contrast at the same time).

\[
Q' (mg/g) = \frac{(0.2 \times C)}{W} \times 100\%
\]  
(2)

Where, \(Q' (mg/g)\) is the adsorption capacity of PP-g-St fibers for BTX emulsion and solution in water, \(W (g)\) is the original weight of PP-g-St fibers before adsorption, \(C (mg/L)\) is the concentration of BTX in the water.

Adsorption capacity of PP fibers for BTX emulsion and solution in the water was also measured in the same manner stated above for comparison purpose.

2.5. Regeneration

In order to research the regeneration ability of PP-g-St fibers, it is necessary to investigate the regeneration adsorption efficiency of fibers reached maximum adsorption. Due to uniform dispersion and limitation in the concentrations of BTX emulsion and solution in water, it was difficult for fibers to attain saturation. So, by using pure BTX as adsorbate, adsorption could reach maximum in a short duration of time, and the maximum adsorption capacities of the fibers were determined by following way. About 1 g of PP-g-St fibers was weighed and then immersed into pure BTX (20 ml, 25°C) for 10 min to achieve maximum adsorption. After holding the fibers corner in the air until the liquid on the surface stopped dripping, the fibers were weighed again. Maximum adsorption capacity of PP-g-St fibers was measured by equation (3) (the test was repeated 5 times to obtain mean value).

\[
Q_s (g/g) = \frac{(W_1 - W_0)}{W_0} \times 100\%
\]  
(3)

Where, \(Q_s (g/g)\) is the maximum adsorption capacities for pure BTX, \(W_0 (g)\) is the original weight of PP-g-St fibers, \(W_1 (g)\) is the weight of PP-g-St fibers after maximum adsorption.

Maximum adsorption capacity of PP fibers in pure BTX was also determined in a manner similar to the above procedure for comparison purpose.

Centrifugation, gravity extrusion, and solvent extraction were chosen as the regeneration methods for maximum adsorption of PP-g-St fibers, and each cycle of regeneration – adsorption was repeated for 6 times. The conditions for each method were: centrifugation – 4000 rpm for 10 min, gravity extrusion - 18.9 KPa for 10 min, solvent extraction - soaking in petroleum ether for 10 min and then drying at 65°C for 2 h. Regeneration adsorption efficiency can be calculated by equation (4).

\[
K = \frac{Q_f}{Q_s} \times 100\%
\]  
(4)

Where, \(K (%)\) is the Regeneration adsorption efficiency, \(Q_f (g)\) is the maximum adsorption capacities after regeneration, \(Q_s (g)\) is the original maximum adsorption capacities.
3. Results and discussion

3.1. Surface profile and infrared spectroscopic analysis
The micro surface morphologies of PP fibers and PP-g-St fibers were observed by SEM (JSM-5900, produced by Japan Electronics Co., Ltd). The results are shown in figure 2.

![SEM images of PP fibers before and after plasma-induced grafting modification](image)

**Figure 2.** SEM images of PP fibers before and after plasma-induced grafting modification (A and B: PP fibers, C: PP fibers after plasma irradiation, D: PP-g-St fibers).

Figure 2 showed the images of PP fibers before and after plasma-induced grafting modification, whereas figure 2B revealed a smooth and flat surface of the PP fiber. After plasma irradiation (figure 2C), the surface of PP fibers almost remained unchanged which meant that besides creating active free radicals, plasma irradiation did not cause visible damage to the structure of PP backbone and had little effect on the mechanical properties.

The surface of PP-g-St fiber showed in figure 2D was obviously covered with a corrugated layer. Due to the unchanged smooth and flat surface of the PP fibers after irradiation, it can be inferred that St chains were introduced on the surface of PP fibers after grafting reaction. Moreover, the surface of PP-g-St fiber was rough and uneven, which could be attributed to the inhomogeneous grafting reaction. These depressions were uniformly distributed and had moderate depths, which contributed to the increased specific surface area of the fiber matrix. This was beneficial for the adsorption process, and had little effect on the mechanical properties.

The successful grafting of St onto the surfaces of PP fibers was further confirmed by FT-IR analysis (Nexus 670, Nicolet) of fiber samples. The results are shown in figure 3.

Compared to PP, the spectrum of PP-g-St showed almost the same absorption peaks except new peaks at 702 cm\(^{-1}\) and 758 cm\(^{-1}\), whereas the peak at 1642 cm\(^{-1}\) became stronger. The peaks at 702 cm\(^{-1}\) and 758 cm\(^{-1}\) could be attributed to the out-of-plane C-H bending vibrations of mono-substituted benzene ring [26]. The peak at 1642 cm\(^{-1}\) was also ascribed to the vibrations of the benzene ring. These results clearly indicated the grafting of St. Moreover, PP-g-St fibers were extracted in acetone to remove the unreacted St monomers and its homopolymer. Hence it became clear that a layer of St was grafted onto PP fibers to form PP-g-St fibers.
3.2. Effects of reaction parameters on grafting percentage

Above discussion proved that St was successfully grafted onto PP fibers. To study the extent of grafting of St on PP fibers, the influences of plasma reaction parameters on grafting percentage were investigated, as shown in figure 4.

**Figure 3.** FT-IR spectra of PP fibers before and after modification (A: PP fibers, B, C, D are PP fibers irradiated by plasma at 50 W, 3 min and 2 h of grafting reaction, 70 W, 3 min and 2 h of grafting reaction, 90 W, 3 min and 3 h of grafting reaction respectively).

**Figure 4.** Effect of reaction parameters on grafting percentage of St (A: Effect of input power, B: Effect of irradiation time, C: Effect of grafting reaction time.).
First, the effect of input power of plasma generator on grafting percentage was investigated, as shown in figure 4A. It could be seen that by increasing the input power, grafting percentage increased initially and then decreased. Grafting percentage reached a maximum value of 3.01%, when input power was 90 W. This was due to the degree of dissociation of plasma atmosphere and increase in the velocities of active particles in the reactor with the increase of input power. In other words, active particles had higher density and kinetic energy, which increased the number of free radicals on the PP fiber surface and accelerated the in-situ grafting reaction. However, when input power exceeded 90 W, the grafting percentage decreased. This was due to the fact that when the density and kinetic energy of active particles were too high, the surface of PP fiber was bombarded and etched violently. This caused the fiber samples to lose weight, and so the grafting percentage decreased. Too much of etching renders the fiber locally incomplete or broken, which affects the mechanical properties of the material. Therefore, an optimal input power improves the grafting percentage, and in this paper the appropriate input power was found to be 90 W.

Secondly, the effect of irradiation time is shown in figure 4B. With increase in irradiation time, grafting percentage increased initially and then decreased. A maximum grafting percentage of 5.50% was reached when the irradiation time was 2 min. To some extent, the effects of extended irradiation time and increased input power were equivalent. Both these factors increased the number of free radicals on the surface of PP fibers and accelerated the grafting reaction. On increasing the irradiation time, the number of free radicals increased, and grafting percentage also increased. When irradiation time was long enough, the concentration of free radicals on the surface of PP fibers reached a saturation value, and the grafting percentage was maximal. However, when irradiation time was too long, the free radical concentration became too large, and the grafting percentage decreased. This was due to the fact that the activation energies of adjacent free radicals were very low, and the coupling reaction between the free radicals by collision became more favorable, which decreased the grafting percentage.

Lastly, the influence of grafting reaction time on grafting percentage was studied and the results are shown in figure 4C. It can be clearly seen that by extending the grafting time, the grafting percentage increased initially and then remained steady. The rate of grafting percentage was high in the first 1 h, then reduced, and almost reached zero after 3 h. The grafting percentage reached a maximum value of 5.7% after 3 h. In the first 1 h of reaction, the St monomers were fully in contact with the free radicals on the surface of PP fibers, which resulted in rapid chain growth reaction of St. On extending the grafting time, the active sites on PP fiber surface decreased, and the chain growth reaction gradually slowed down and then was terminated.

3.3. Adsorption of PP-g-St fibers for BTX

The adsorption capacity of PP fibers and PP-g-St fibers for BTX emulsion and solution in water were studied, and the results are presented in table 1.

| BTX emulsify and dissolve in water | adsorption capacities of PP fibers (mg/g) | adsorption capacities of PP-g-St fibers (mg/g) | Increasing rate of capacities (%) |
|----------------------------------|------------------------------------------|-----------------------------------------------|---------------------------------|
| benzene                          | —                                        | —                                             | —                               |
| toluene a                        | 142.30                                   | 612.70                                        | 336.89                          |
| xylene b                         | 115.45                                   | 513.26                                        | 344.57                          |

*a* initial concentration of toluene: 483 mg/L.

*b* initial concentration of xylene: 350 mg/L.

It can be seen from table 1 that compared with PP fibers, the adsorption capacities of PP-g-St
fibers for toluene and xylene emulsion and solution in water were increased, which were 336.89 % and 344.57%, respectively. Solubility parameters of PP-g-St fiber and BTX were closer, which increased their affinities [18]. BTX was tightly adsorbed on surfaces having St groups by Van der Waals forces, and was occluded by the swollen St chains on the fiber surface through solvation. Moreover, the surface of PP-g-St fibers was rough and had a larger specific surface area, which also increased the adsorption capacity of PP-g-St fibers for BTX emulsion and solution in water.

The adsorption capacity of fibers for benzene in water could not be tested accurately due to the volatile nature of benzene in water. Hence, the amount of benzene volatilized could be much higher than adsorbed over a limited time period.

3.4. Regeneration of PP-g-St fibers

Both PP fibers and PP-g-St fibers are all full of meshes—web structure, which makes a quick adsorption and great adsorption capacity for pure BTX. So, research their maximum adsorption capacities for pure BTX to compare with the adsorption capacity for it in water seems to be necessary. Meanwhile, regeneration adsorption efficiencies can be obtained too basing on this foundation. Figure 5 shows the maximum adsorption capacities of PP fibers and PP-g-St fibers for pure BTX.

![Figure 5. Maximum adsorption capacities of PP fibers and PP-g-St fibers for pure BTX.](image)

From figure 5, it was clear that compared with PP fibers, maximum adsorption capacities of PP-g-St fibers for pure benzene, toluene, and xylene showed considerable improvement, from 14.4 g/g, 13.5 g/g, and 10.9 g/g to 18.6 g/g, 16.8 g/g, and 13.4 g/g, respectively. As mentioned previously, this was due to the fact that St was grafted onto the fiber surface and the specific surface area of fibers was increased.

The differences in increasing rates of adsorption capacities of PP fibers and PP-g-St fibers for pure BTX, BTX emulsion, and solution in water have been discussed. Firstly, maximum adsorption capacities of PP fibers and PP-g-St fibers for pure BTX were large, and they could quickly adsorb BTX, more than 10 times their own weights. Meanwhile, their adsorption capacities and adsorption rates for BTX emulsion and solution in water were small. This was due to the web-like structures of PP fibers and PP-g-St fibers that allowed them to absorb large amounts of pure BTX quickly through their gaps by capillary effect. However, for BTX emulsion and solution in water, due to the dispersion of BTX and the flow of water, BTX could not be properly absorbed inside the PP and PP-g-St fibers through the gaps.

Secondly, compared with pure BTX, the increasing rate of adsorption capacities of PP-g-St fibers
for BTX emulsion and solution in water were larger than those of PP fibers. This could be explained by surface adsorption and occlusion of PP-g-St fibers, which dramatically enhanced its adsorption capacity for BTX emulsion and solution in water. Besides, the maximum adsorption capacities of PP fibers and PP-g-St fibers for benzene, toluene, and xylene showed a decreasing trend. This phenomenon could have occurred due to reduction in surface tension and surface energy, since an adsorbate with lower surface tension and surface energy is more likely to be adsorbed onto styrene in case of PP-g-St fibers [27].

Generally, synthetic fibers have better renewability than natural adsorbents. Based on the above discussion, PP-g-St fibers had a remarkable adsorption capacity for BTX by adsorption and occlusion, due to St groups present on the fiber surface. Also both, The desorption of adsorbed BTX and the regenerative efficiency of maximum adsorption of PP-g-St fibers, are extremely important for practical applications. It could be seen from figure 5 that the adsorption capacity of PP-g-St fiber for toluene was much higher. So, using pure toluene as adsorbate (benzene was too volatile to calculate the regenerative adsorption efficiency), regenerative adsorption efficiencies of PP-g-St fibers by regeneration methods like centrifugation, gravity extrusion, and solvent extraction were tested, as shown in figure 6.

![Regeneration adsorption efficiencies of PP-g-St fibers in three different ways.](image)

It is evident from figure 6 that amongst the three regeneration methods, centrifugation was found to be the most efficient one. The high efficiency of this method could be attributed to the strength of centrifugal force. BTX absorbed inside the web structure could be thrown out. Moreover, BTX absorbed and occluded in St chains by Van der Waals forces could also get desorbed. But with gravity extrusion and solvent extraction methods, desorption was difficult.

After six cycles (adsorption - regeneration - adsorption) using centrifugation process, the adsorption capacity remained stable, and the regeneration adsorption efficiency was still more than 90%. Most of the BTX absorbed on the surface of fibers was thrown out, but only a little amount of BTX, which was occluded by the St chains could not get desorbed. A moderate grafting percentage was very important. High grafting percentage could increase the adsorption capacity of PP-g-St fibers for BTX, but it would also cause problems for desorption or regeneration process.

4. Conclusion

- The optimum parameters for plasma treatment of PP fibers for grafting of St were: input power of 90 W, irradiation time of 3 min, reaction time for grafting of 3 h, to achieve a
grafting percentage of 5.7%.

- Maximum adsorption capacities of PP-g-St fibers for pure benzene, toluene, and xylene showed a considerable improvement, from 14.4 g/g, 13.5 g/g, and 10.9 g/g to 18.6 g/g, 16.8 g/g, and 13.4 g/g, respectively. The increasing rate of adsorption capacity of PP-g-St fibers for toluene and xylene emulsions and solutions in water were 336.89% and 344.57%, respectively.
- The adsorption capacity of PP-g-St fibers was stable, and the regeneration adsorption efficiency was still more than 90% after six repeated cycles, using centrifugation regeneration method.

References

[1] Asadpour R, Sapari N B, Isa M H, Kakooei S and Orji K U 2015 Fiber. Polym. 16 1830
[2] Wu D X, Fang L L, Qin Y M, Wu W J, Mao C M and Zhu H T 2014 Mar. Pollut. Bull. 84 263
[3] Dong T, Xua G B and Wang F M 2015 J. Hazard. Mater. 296 101
[4] Carvalho M N, Motta M da, Benachour M, Sales D S C and Abreu C A M 2012 J. Hazard. Mater. 239 95
[5] Lin S H and Huang C Y 1999 J. Hazard. Mater. 70 21
[6] Chen Y, Knappe D R and Barlaz M A 2013 Chemosphere. 90 251
[7] Szala B, Bajda T, Matusik J, Zięba K and Kijak Beata 2015 Microporous. Mesoporous Mater. 202 115
[8] Ranck J M, Bowman R S, Weeber J L, Katz L E and Sullivan E J 2005 J. Environ. Eng. 131 434
[9] Altare C R, Bowman R S, Katz L E, Kinney K A and Sullivan E J 2007 Microporous. Mesoporous Mater. 105 305
[10] Simpson J A and Bowman R S 2009 J. Contam. Hydrol. 108 1
[11] Guo M L, Luo Z W and Fan C H 2016 Asia-Pacific J. Chem. Eng. 11 778
[12] Korholen J T, Kettunen M and Robin H A 2011 Mater. Interfaces. 3 1813
[13] Li H Y, Wu W F, Bubakir M M, Chen H B, Zhong X F, Liu Z X, Ding Y M and Yang W M 2014 J. Appl. Polym. Sci. 10 40080
[14] Rengasamy R S, Dipayan D and Praba K C 2011 J. Hazard. Mater. 186 526
[15] Jin R G and Hua Y Q 2002 Polymer Physics (Beijing: Chemical Industry Press) vol 2 chapter 3 pp 58-9
[16] Shi D A, Yang J H, Yao Z H, Wang Y, Huang H L, Wu J, Yin J H and Costa G 2001 Polymer. 42 5549
[17] Sari A, Biçer A and Alkan C 2017 s 161 219
[18] Wang X Y, Yue X H, Wang X and Gou Q J 2011 Journal of Macromolecular Science. 50 1882
[19] Liu W T, Wang L S, Mai J Z and Zhang A Q 2014 Polym. Bull. 71 3111
[20] Li Y, Manolache S, Qiu Y P and Sarmadi M 2016 Appl. Surf. Sci. 364 294
[21] Khlyustova A, Galmiz O, Zahoran M, Brablec A and Cernak M 2015 J. Mater. Sci. 50 3504
[22] Samieyan E, Rahimi H and Ershad Langroud A 2013 Plast. Rub. Compos. 42 256
[23] Wavhal D S and Fisher E L 2003 Langmuir. 19 79
[24] Li S N and Wei J F 2012 J. Polym. Res. 19 9841
[25] Wu J Y, Alicia K A, Guo J X, Lee E J, Farid M U and Jeong S 2016 Chem. Eng. J. 12 010
[26] Li Y and Chu L Y 2004 Ind. Eng. Chem. Res. 43 2643
[27] Guo M L, Liang Z Y, Luo Z W, Chen Q G and Wei W J 2016 Fiber. Polym. 17 257