

Psa composite fibers and membranes



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Polysulfonamide/nano titanium dioxide (PSA/nano-TiO₂) composite spinning solutions with various nano-TiO₂ mass fractions were prepared using the solution blending method. The corresponding composite fibers were developed by wet-spinning technology and the composite membranes were prepared using the digital spin-coating technique. The properties of PSA/nano-TiO₂ composite fibers and membranes were investigated by scanning electron microscope, Fourier transform infrared spectroscopy and X-ray diffraction, etc. The effects of nano-TiO₂ and its mass fractions on the mechanical properties, thermal stability and ultraviolet resistance of PSA composites were also analyzed. The experimental results showed that nano-TiO₂ with low mass fractions can be dispersed evenly in the PSA matrix; the blending of nano-TiO₂ had no obvious influence on the molecular structure and the chemical composition of PSA fiber; the crystallization in PSA fiber was promoted at low nanoparticles mass fractions because it can act as a nucleation agent; the mechanical properties and the thermal stability of PSA/nano-TiO₂ composites can be enhanced obviously by blending nano-TiO₂ into PSA matrix. The ultraviolet resistance of PSA composites can be improved significantly with the increasing nano-TiO₂ mass fractions and the 7 wt.% specimen showed the lowest UV transmittance.

Polysulfonamide (PSA) fiber is a new kind of hightemperature resistant material and it has outstanding heat resistance, flame retardancy, and thermal stability, therefore, it can be used to develop protective products used in aerospace, high-temperature environments and civil fields with the flame retardant requirements (Ren, Wang, & Zhang, 2007; Wang, 2009). However, raw PSA generally demonstrates poor ultraviolet resistance and

the amide groups in polymer molecular chains are prone to break down under the ultraviolet radiation; besides, the breaking tenacity of PSA fibers is low; these properties lead to some difficulties in its manufacturing procedures and limit its application in developing functional textiles. Therefore, it is a challenging work to improve the mechanical properties and ultraviolet resistance of PSA.

It has been proved that nano-TiO₂ is one of the ideal nano-enhanced materials and it has attracted great scientific attention because of its excellent mechanical properties in significantly improved properties of composites (Ali, Shadi, Shirin, Seyedeh, & Khademno, 2010; Han & Yu, 2005). Moreover, nano-TiO₂ is good semiconductor oxides and it has excellent ultraviolet scattering and absorption (Popov, Priezzhev, Lademann, & Myllylä, 2005). It is feasible to blend nanoTiO₂ into PSA matrix to improve the mechanical properties and ultraviolet resistance of PSA composites.

Experimental

Materials

The PSA polymer was used as spinning solution with intrinsic viscosity of 2.0–2.5 dL/g and relative molecular mass of 462. The rutile titanium dioxide (nano-TiO₂) was blended as functional particles with a diameter ranging from 30 to 50 nm and the rutile content of nano-TiO₂ was about 99%. The dimethylacetamide (DMAC) was selected as dissolvent in this study. The above materials were provided by Shanghai Tanlon Fiber Co. Ltd. All the chemicals used here were of reagent grade and they were used without further purification.

Preparation of PSA/nano-TiO₂ composites

A certain amount of nano-TiO₂ was predispersed in DMAC using ultrasonic vibration for 30min; and then added into the PSA solution. The PSA/nano-TiO₂ composite spinning solutions with various mass fractions of nanoparticles was prepared after mechanical stirring for 1 h and ultrasonic vibration for 2 h. The experimental data are shown in Table 1.

The pure PSA fibers and PSA/nano-TiO₂ composite fibers were developed by a small-scale and single screw wet spinning apparatus. Besides, the pure PSA membrane and PSA/nano-TiO₂ composite membranes were prepared using the SJT-B digital spin-coating instrument. The preparation procedures of nanofibers and membranes can be referred to the previous studies (Chen, Xin, Wu, Wang, & Du, in press; Xin, Chen, Wu, & Wang, in press).

Test methods

The dispersion of nanoparticles in PSA composites S-3400N scanning electron microscope (SEM) with a resolution of 4 nm was used to characterize the dispersion of nano-TiO₂ in PSA matrix. The machine was operated at 5 kV.

FTIR spectroscopy

Thermo Nicolet AVATAR 370 Fourier transform infrared spectroscopy (FTIR) was used to characterize the molecular structure and chemical composition of fibers; each spectrum was collected by cumulating 32 scans at a resolution of 4 cm⁻¹

X-ray diffraction

X-ray diffraction (XRD) measurements of the crystalline structure of fibers were recorded on k780 FirmV_06 X-ray diffraction using the $\text{CuK}\alpha$ radiation ($\lambda = 0.15406 \text{ nm}$). The spectra were obtained at 2θ angles range of 5°

-60°

with a scanning speed of $0.8^\circ/\text{s}$

step.

Mechanical properties test

YG006 electronic single fiber strength tester was used to investigate the mechanical properties of fibers. The sample gage length was 10mm. The elongation speed was set at 20mm/min. The measurements for each sample were carried out 10 times and the average was taken. The thermal stability test The thermal stability of fibers was measured by Germany STA PT-1000 Thermal Gravimetric Analyzer (Linseis Inc., New Jersey, USA); the experiment was conducted under nitrogen atmosphere with a gas flow of 80–100ml/min; the samples were heated up to 700°C from the room temperature at a heating rate of $20^\circ\text{C}/\text{min}$.

Ultraviolet resistance test

Labsphere UV-1000F Ultraviolet Transmittance Analyzer (Labsphere, Inc., North Sutton, NH, USA) was used to test the UV transmittance of membranes. The instrument parameters were described as below:

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the absorbance was 0–2. 5A; scanning time was about 5 s; data interval was 1 nm and the diameter of beam was 10mm. The measurements for each sample were carried out for 10 times and the average was used for the result discussion.

Results and discussion

The distribution of nano-TiO₂ in PSA composites

As demonstrated in Figure 1, 1 wt.% of nano-TiO₂ can be dispersed evenly throughout the PSA matrix and the size of nanoparticles is about 50–60 nm; with the nanoTiO₂ mass fraction increased to 3 wt.%, a little aggregation can be observed; when the mass fraction of nanoTiO₂ increased to 5 or 7 wt.%, its dispersion in PSA becomes inhomogeneous because of their large specific surface and high surface polarity, and the aggregation size is about 100–300 nm. It is difficult for nano-TiO₂ with high mass fractions to distribute uniformly in the PSA blending system.

FTIR analysis of PSA/nano-TiO₂ composite fibers

As shown in Figure 2, the position and shape of characteristic peaks of PSA composites blending with nanoTiO₂ did not change obviously compared with the pristine PSA. The characteristic peaks of PSA composites exhibiting at about 3338. 99 cm₋₁ can be attributed to

the amide N–H stretching vibration and the peaks are flattened slightly with the mass fractions of nano-TiO₂ increased from 1 to 7 wt.%. It ascribes to the quantum size effect of nanoparticles (Zhang & Mou, 2001). In conclusion,

it shows no significant changes to the molecular structure and chemical composition of PSA fibers with the addition of nano-TiO₂.

XRD analysis of PSA/nano-TiO₂ composite fibers As depicted in Figure 3, the PSA composite fibers have diffraction peaks at 27.54°, 36.15°, 41.35°, and 54.40°, this is because of the blending of nano-TiO₂ (Chen, Liu, Zhang, Zhang, & Jin, 2003; Xia & Wang, 2002).

In addition, all the specimens have diffraction peaks at about 11.85° and 21.25°. The sharp diffraction peaks corresponding to 11.85° indicate that there are crystalline structures in PSA/nano-TiO₂ composite fibers (Yang, 2008).

Besides, the sharpness of the diffraction peaks at about 11.85° of composites enhances gradually with the nano-TiO₂ mass fractions increased from 1 to 5 wt.%. It suggests that the crystallization in PSA can be improved with the blending of nano-TiO₂, because it can act as a nucleation agent.

Moreover, the shape of diffraction peaks exhibiting at 21.25° of PSA composites broadens significantly with the increasing nano-TiO₂ mass fractions and it proves that the size of crystal region becomes smaller (Meng, Hu, & Zhu, 2007).

The mechanical properties of PSA/nano-TiO₂ composite fibers

As illustrated in Table 2, the breaking tenacity of PSA composite fiber with 1 wt.% nano-TiO₂ improved obviously; however, the improving degree of breaking tenacity begins to decrease with the continuous increase in mass fractions of nano-TiO₂ and the value of the 7 wt.% sample is lower than the pure PSA.

This is because nano-TiO₂ is an ideal nano-enhanced material; the blending of it into PSA can improve the mechanical properties of composites to some extent.

Moreover, nano-TiO₂ with low mass fractions can be distributed evenly in PSA matrix and it can form a good interface with PSA molecular chains.

As can be seen in Table 2, the composite fibers have low elongation at break which is lower than the raw PSA; simultaneously, the initial modulus of composites increased significantly, however, the improvement begins to decrease with the mass fractions of nano-TiO₂ increased from 1 to 5 wt.% and the 7 wt.% sample has the minimum value of the initial modulus. It suggests that the blending of nano-TiO₂ with low mass fractions can improve the mechanical properties of PSA composite fibers to a certain extent.

The thermal stability of PSA/nano-TiO₂ composite fibers

TG curves and derivative thermogravimetric analysis (DTG) curves of PSA/nano-TiO₂ composite fibers are demonstrated in Figures 4 and 5, respectively. The main parameters of the curves are presented in Table 3.

In Figure 4, the thermal decomposition behaviors of specimens are divided into three regions.

The first region is a stage of small mass loss ranging from room temperature to 400°C. As depicted in Figure 4, each TG curve has a sharp decrease in the beginning and then reaches a platform with the temperature heating up to 350°C. However, the mass loss of PSA composites blending with nano-TiO₂ is always lower than the pure PSA during this process. As shown in

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Table 3, the T10wt of each PSA composite is high, whereas the mass loss of pure PSA reached 10% at 170. 19°C. This suggests that it is hard for the PSA composites to decompose and the thermal stability is significantly higher than PSA.

The second region is a stage of thermal decomposition process ranging from 400 to 600°C. According to the analysis of bond energy (Zhang, Cheng, & Zhao, 2000), the C-N section of amide in PSA macromolecular chains decomposes at 500–600°C (Broadbelt, Chu, & Klein, 1994a, 1994b) and the mass loss of PSA at this stage is attributed to the gases released such as SO₂, NH₃, and CO₂. In addition, as illustrated in Table 3, the T₀ of PSA composites blending with 1 and 3 wt.% nano-TiO₂ can be increased; therefore, its thermal stability can be improved correspondingly.

As exhibited in Figure 4, the mass loss of specimens accelerates steadily with the increasing temperature and each TG curve presents a rapid decomposition at about 500°C. Corresponding to the rapid decomposition, there is a peak in DTG curve shown in Figure 5 and the T_{max} can be determined according to the value of the maximum peak (Yang, 2008).

The third region is a high-temperature phase of carbon formation ranging from 600 to 700°C. As demonstrated in Figure 4, the PSA composites still show a slight decomposition during this stage; besides, the mass loss of pure PSA decreases obviously. As illustrated in Table 3, the residual mass of composites at the terminal temperature is higher than the pure PSA.

Therefore, it is concluded that the thermal stability of PSA composites blending with nano-TiO₂ can be improved significantly.

The ultraviolet resistance

As exhibited in Figure 6, the ultraviolet transmittance of specimens ranging from 390 to 400 nm decreases gradually with the increase in mass fractions of nano-TiO₂. This suggests that the nanoTiO₂ can improve the ultraviolet resistance of PSA composites significantly. This is because the refraction index (RI) of nano-TiO₂ is extremely high (2.73) and it has excellent ultraviolet scattering properties (Liu, Tang, Zhang, & Sun, 2007). In addition, electrons in nano-TiO₂ are transited from the valence band to the conduction band under the ultraviolet radiation; therefore, the nano-TiO₂ has outstanding ultraviolet absorption properties.

Conclusions

The PSA composite fibers and membranes with different mass fractions of nano-TiO₂ were developed.

The experimental results can be summarized as follows:

(1) The nano-TiO₂ with low mass fractions (1 or 3 wt.%) can be distributed evenly in the PSA blending system; however, it is difficult for nano-TiO₂ with high mass fractions (5 or 7 wt. %) to disperse homogeneously throughout the PSA matrix.

(2) The blending of nano-TiO₂ showed no obvious changes to the molecular structure and chemical composition of PSA composite fibers.

(3) The crystallization of PSA composite fibers can be improved by blending with low mass fractions of nano-TiO₂, because it can act as a nucleation agent.

(4) The breaking tenacity and initial modulus of

4

5

ance %

(a)

(b)

(c) PSA composite fibers can be improved obviously by blending with low mass fractions of nano-TiO₂; whereas the elongation at break of PSA composite was decreased with the particles mass fractions increased from 1 to 7 wt.%.

(5) The thermal stability of PSA composites can be increased significantly and the nano-TiO₂ has some influences on the T_0 , T_{10wt} , and T_{max} of PSA composites compared with the pure PSA.

(6) The blending of nano-TiO₂ can improve the ultraviolet resistance of PSA composites significantly and the 7 wt.% specimen had the lowest UV transmittance.