PREPARATION OF BIODEGRADABLE NANOCELLULOSE COMPOSITE BY ELECTROSPINNING

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Introduction

Electrospinning is a simple and cost effective method for preparing nanofibrous polymers. The nanofibres produced by electrospinning show particular characteristics such as large surface area-to-volume ratio and high porosity with small pore size, which have potential use in tissue engineering [1].

Poly(lactic acid) (PLA) has been mostly used as a candidate for electrospinning because it has good mechanical properties, biodegradability and biocompatibility [2]. However, the low cell affinity is a restriction due to its hydrophobic nature.

The wettability of PLA nanofibrous mat can be improved by incorporating hydrophilic cellulose nano-whiskers during the electrospinning process. Moreover, cellulose nanowhiskers are biocompatible and have good mechanical properties [3]. The composite nanofibrous mats were successfully prepared by electrospinning the mixtures of cellulose nano-whiskers with PLA solution. The microstructure and morphology of pure PLA and composite mats were investigated in this study.

Experimental

Materials

Poly (lactic acid) (PLA, 2002 D), was purchased in pellet form from Natureworks Co., Minnetonka, USA. Bacterial cellulose pellicles for hydrolysis were kindly supplied by Tianjin University, China. N, N-dimethylformamide (DMF, Anhydrous, 99.8%) was purchased from Sigma-Aldrich, USA. Tetrahydrofuran (THF) was bought from ECP Ltd (Romil, UK). Sulphuric acid with concentration 95-97%, was purchased from Merck KGaA, Darmstadt, Germany.

Cellulose nano-whiskers were prepared by acid hydrolysis as previously reported [4].

Cellulose nano-whiskers were dispersed in DMF (ambient temperature, with stirring overnight), then appropriate amounts of THF and PLA were added and the mixture stirred for several hours. The final dispersion contained 2.5, 5.0, or 7.5 wt.% cellulose nano-whiskers, and 12% PLA.

Apparatus and Procedures

The electrospun mats were obtained by applying a high voltage between the tip of a needle and a grounded collector. The nanofibrous composite mat was collected on the aluminium foil by setting the distance of 80 mm between the collector and the needle tip, with applied voltage 8-10 kV and solution feed rate 0.8-1.0 ml hr^{-1}.

The morphology of the electrospun nanofibres was observed using field emission scanning electron microscopy (FE-SEM, FEI XL30s) with accelerating voltage 5 kV. Fourier transform infrared spectroscopy (FTIR, ATR-FTIR, Nicolet 8700, USA) was used to chemically confirm the presence of the nanocellulose in the matrices of PLA nanofibres. All spectra were collected with 4 cm^{-1} wave number resolution after 100 continuous scans at the wavelength range 4000 to 500 cm^{-1}.

The microstructure of pure PLA and composite mats was examined by X-ray powder diffractometry (XRD) using CuK_{α} radiation, at scan speed 0.02° s^{-1} from 5 to 50°.

Results and Discussion

Cellulose nano-whiskers produced by hydrolysis of bacterial cellulose pellicles were rod shaped with diameter about 20 nm and length 500-1000 nm [4]. The morphology of pure PLA and PLA/nanocellulose fibres in the mats produced by electrospinning is shown in Fig.1. The fibres with diameters of about 200 nm were relatively uniform. No cellulose nano-whiskers were observed on the outer surface of the PLA fibres, indicating that they were embedded in the PLA matrix.

To confirm the presence of nanocellulose in the PLA nanofibre mats, they were subjected to analysis with FTIR. In Fig.2, the bands between 3550 and 3200 cm^{-1}, which are typical of stretching O-H vibrations, are seen for the composite mats including 5.0 and 7.5 wt. % cellulose nano-whiskers, even though the peak is not strong. This means that cellulose nano-whiskers are incorporated into the PLA matrix during the electrospinning process. The weak diffraction peak at

2θ =22.6º, typical of the 200 plane of the cellulose, appears on the diffraction patterns of composites with 5.0 and 7.5 wt.% nanocellulose (Fig.3). Moreover this broad peak moves to a higher angle (from 13.5 to 14.5°) upon addition of cellulose nano-whiskers to PLA matrix (the peak at 14.5° is also one of the characteristic peaks of cellulose). Pure PLA and PLA composite mats show broad diffraction patterns, which indicate an amorphous structure. The addition of cellulose nano-whiskers produced sharper diffraction peaks, suggesting increased crystallinity of the PLA matrix.

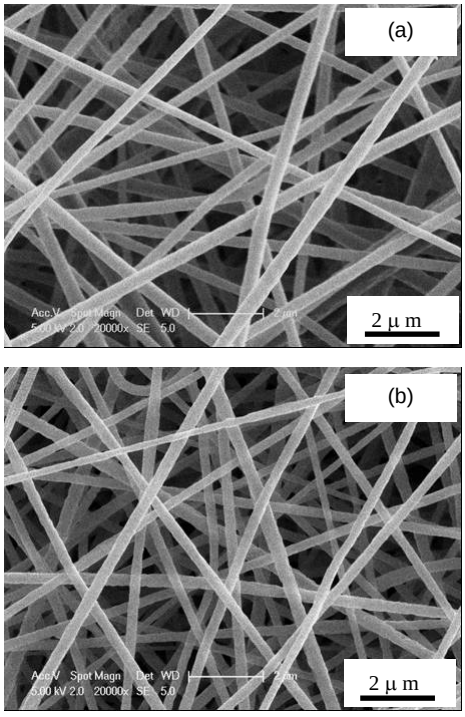

Fig. 1 SEM observations of electrospun fibres of (a) pure PLA mat and (b) PLA composite mats containing 7.5 wt.% cellulose whiskers.

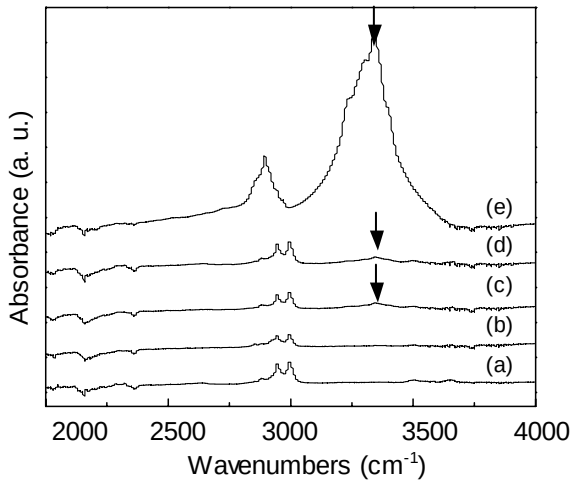

Fig. 2 FTIR spectra of (a) pure PLA, and PLA with (b) 2.5 wt.%, (c) 5.0 wt.% and (d) 7.5 wt.% cellulose nano-whisker composite mats, and (e) BC nano-cellulose whiskers.

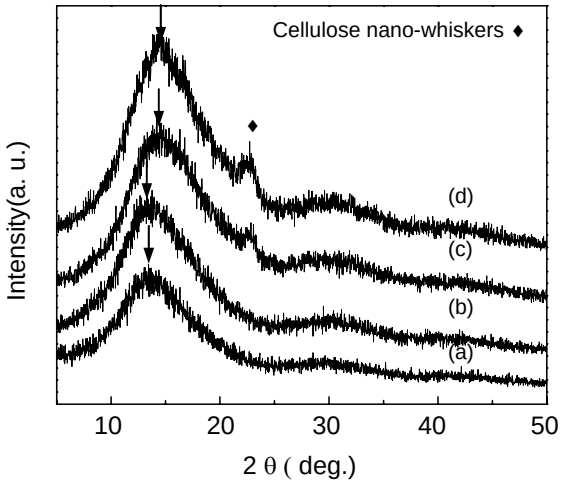

Fig. 3 X-ray diffraction patterns of (a) pure PLA, and PLA with (b) 2.5 wt.%, (c) 5.0 wt.%, and (d) 7.5 wt.% cellulose nano-whisker composite mats.

Conclusion

The PLA/cellulose nano-whiskers composite nanofibrous mats were successfully prepared by electrospinning. The fibres with diameters of 300 nm show no difference between pure PLA and PLA/cellulose nano-whisker composites.

References

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