전기방사기법에 의한 Poly(butylene terephthalate) 나노섬유 제조 및 특성

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Preparation and Characterization of Poly(butylene terephthalate) Nanofiber by Electrospinning

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1. Introduction

The electrospinning is a unique method that can prepare the nanofiber web because it use the difference of electromotive force between polymer solution and collector. The nanofber web offers as a crucial raw materials to electrical and electrochemical system by the enhancement of physical property. Recently, the polymers such as PAN, PBI were electrospun in the solvents like DMF and THF [1-3]. However, the preparation of web using PBT solution by elctrospinning was hard to find out elsewhere. The factors that affect to electrospinning are system, process, peripheral circumstance. Firstly, there are the concentration of polymer solution, viscosity, surface tension in the factor of system. Second, there are the voltage strength, distance between tip and collector, and flow rate of polymer solution in the factor of process. Finally, there are humidity and temperature in the peripheral circumstance. In this study, the nanofiber web was prepared to use PBT solution in tetrafluoroacetic acid with various concentrations. The electrospinning was examined with changing the process factor such as the voltage strength, distance between tip and collector, and flow rate of PBT solution. The morphology of web was observed by SEM.

2. Experimental

PBT (7-20 wt.%) was dissolved in trifluoroacetic acid. The solution was spun into a fiber web through the positively charged capillary using an electrospinning apparatus (NTSEE Co. Korea). The polymer solution was placed in a 10 ml syringe with a capillary tip of 0.838 mm diameter. The anode of the high voltage power supply was clamped to a syringe needle tip and the cathode was connected to a metal collector. The electrospun fiber was collected by attaching it to aluminum foil nwrapped on a metal drum rotating at round 500 rpm. The flow rate controlled in the range of 0.1 to 0.5 ml/h by syringe pump (Cole-Parmer, KDS 100), the

distance between tip and collector was 10 to 20 cm, and the web was prepared with changing the voltage through 15 to 35 kV. Both morphology and thermal characteristics of web was observed by Scanning Electron Microscopy (SEM, Model JSM-6460LV) and thermo gravimetric analysis (TGA), respectively.

3. Results and discussion

Fig. 1 shows a SEM image of the electrospun PBT nanofiber web with the concentrations of solutions at 20 kV. The diameter of fibers has a general tendency by the decrease of the instability of whipping because the bending and splitting of polymer was difficult in the electric field by the increase of the stress of polymer within polymer solution as the concentration increases. The PBT solution was not to be electrospun smoothly by the small globular accumulation of polymer solution because the concentration is too low and the surface tension was too large below 7 wt.% concentration.

Fig. 2 shows a SEM image by the change of fiber diameter with the voltage to use 8 wt.% solution shown the small, uniform tendency of the fiber diameter. As voltage increases the diameter of nanofiber decreases, but at a certain point it increases its diameter rather than decreasing it. The reason for this is that the polymer solution was to be electrospun smoothly as the globular shape at the optimum voltage.

The SEM image of Fig. 3 represents the change of fiber diameter with increasing the flow rate of PBT solution. When the flow rate of the solution was 0.1 ml/h, it was hard to make the uniform fiber even if the diameter of fiber was much thinner. On the contrary, the diameter of fiber was to be increased in over 0.1 ml/h flow rate. The reason for this is that the spinning time was to be reduced by the increase of the content of solution to be loaded to the constant electrostatic attraction.

Fig. 4 shows a SEM image of nanofiber formed with increasing TCD. The average diameter was decreased by the increase of whipping, splitting time, and by the easiness of the volatility of solvent as TCD was increased. However, the diameter of fiber is increased by the decrease of fiber strain in 20 cm due to the reduction of electrostatic attraction.

Fig. 5 shows thermogravimetric analysis (TGA) of web formed by electrospinning. The weight loss was examined to measure until 800° by 5° /min. The web was rarely the change of weight until 400° in the atmosphere of nitrogen and air.

4. Conclusions

The nanoweb of 280 nm average diameter was prepared uniformly in the conditions of 8 wt.% PBT solution, 20 kV voltage, 0.2 ml/h flow rate and 16 cm TCD. The fiber diameter decreases with increasing the applied voltage at the same concentration. The fiber shows the minimum average diameter at a certain voltage, and the average diameter of fiber increases again above voltage. The diameter of fiber was increased as the flow rate of the solution was increased at

the same voltage and concentration. The average diameter of fiber decreased as TCD increased. it was minimum at a certain voltage, and it increased again above voltage.

Acknowledgements

This research was supported by KOSEF under Grant number R01-2003-000-10100-0.

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Fig. 1. SEM microphotographs of PBTfibers electrospun at various concentrations.(V:16kV, TCD:16cm, F.R:0.4ml/h, R:500rpm)(a) X 5000, (b) X 50000

(b)
Fig. 2. SEM microphotographs of PBT fibers electrospun at various voltages.
(C:8wt%, TCD:16cm, F.R:0.4ml/h, R:500rpm)
(a) X 5000, (b) X 50000







Fig. 3. SEM microphotographs of PBT fibers electrospun at various flow rate. (V:20kV, TCD:16cm, C:8wt%, R:500rpm) (a) ×5000, (b) ×50000





Fig. 4. SEM microphotographs of PBT fibers electrospun at various TCD.(V:20kV, C:8wt%, F.R:0.2ml/h, R:500rpm)(a) X 5000, (b) X 50000



Fig. 5. TGA spectra of the web in atmosphere of both nitrogen and air.