

Preparation of Poly(ester imide) Ultrafine Fibers by Gas-Jet/Electrospinning

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ABSTRACT: In this study, ultrafine fibers of poly(ester imide) (PEI) were produced by gas-jet/electrospinning of its solutions in mixtures of phenol and dichloromethane (DCM). The process parameters, including the solution concentration, gas flow rate, applied voltage, tip-to-collector distance (TCD), and inner diameter of the metal needle, were investigated by scanning electron microscopy. The results show that the solution concentration, gas flow rate, TCD, and inner diameter of the needle were the most important process parameters influencing the average diameter and morphology of the PEI gas-jet/electrospun fibers. An increase in the solution concentration resulted in a larger average diameter in the PEI gas-jet/electrospun fibers. Mixed-bead fibers were obtained when the concentration of PEI in phenol/DCM was below 20 wt % during gas-jet/electro-

spinning. A larger diameter of the capillary and a smaller gas flow rate favored the formation of ultrafine fibers with thicker fibers. Thinner and uniform PEI fibers with an average diameter of 298 nm were formed at a TCD of 25 cm. On the basis of the systematic parameters study, uniform PEI ultrafine fibers with an average diameter of 293 nm were prepared by this gas-jet/electrospinning with the following optimal process parameters: the concentration of the polymer solution was 20 wt %, the gas flow rate was 10.0 L/min, the applied voltage was 25.0 kV, the TCD was 25 cm, and the inner diameter of the metal needle was 0.24 mm. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 883–891, 2009

Key words: fibers; morphology; gas-jet/electrospinning; poly(esterimide)

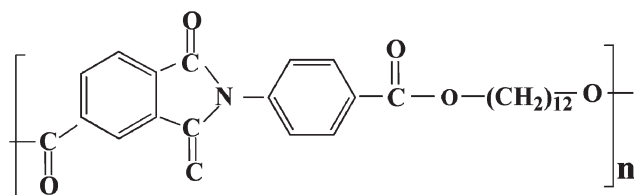
INTRODUCTION

Poly(ester imide) (PEI) is recognized as an important kind of high-performance polymer because of its outstanding properties, such as excellent mechanical properties, thermal stability, chemical resistance, and low melting viscosity.^{1,2} In addition, PEI with rigid and flexible segments in the main chain, which is represented in Scheme 1, very often forms a liquid-crystalline phase.³ It can form an enantiotropic nematic phase in addition to a crystalline solid state with a smectoid layer structure,⁴ so it possesses unusual rheological properties in addition to a high orientation in magnetic or electrical fields. Therefore, it is considered an advanced material and has been extensively used in insulating materials,^{5–10} sensor materials,¹¹ high-temperature resistant materials,¹² and so on.

Electrospinning is a straightforward, cost-effective, versatile technique that uses an electrostatic force to produce polymer fibers with diameters ranging from a few micrometers down to 10s of nanometers.^{13–18} Zelency first introduced this technique in 1914. It was patented by Formhals¹⁹ as a process for spinning fibers. However; the technique had not been received interest at the time. Recently, it has been revitalized and successfully applied for spinning ultrafine fibers.^{20,21} With large surface area to volume ratio,²² electrospun fibers have been used in filtration,²³ separation,²⁴ protective clothing,²⁵ tissue engineering,^{26,27} sensor,²⁸ catalyst,²⁹ and molecular recognition.³⁰ Over a hundred synthetic and natural polymers were successfully prepared into uniform fibers by this technique. However, liquid crystal materials are used as the material of electrospinning is very few. Until most recently, Krause et al.³¹ obtained fibers from the main-chain liquid crystal polymer by electrospinning in 2006. The average diameter of the good quality fibers was 1.5 μm . Wu et al.³² prepared fibers of liquid crystal polysiloxane with cholesterol by electrospinning in 2007. The fibers diameters ranged from 1 to 10 μm . The fibers above are micrometer thick in average diameter. In additional, up to now, studies on the liquid crystal

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Scheme 1 Chemical structure of PEI.

PEI electrospinning have hardly been reported in contrast to the many studies on the structure and property of PEI.^{1-3,33,34}

In our previous study, Yao et al. designed a gas-jet/electrospinning apparatus combining electrospinning with a gas-jet device and prepared polysulfone nanofibers with this apparatus.³⁵ It is different from the traditional electrospinning apparatus³⁶ in the spinneret, in which the capillary of spinning fluid is circled by the tube of the gas jet. Their experimental results show that gas-jet/electrospinning is a better method than conventional electrospinning, making finer and more uniform nanofibers and expressing higher efficiency because of the drawing force of the gas jet.³⁵ The gas-jet/electrospinning results of poly(ether sulfone) nanofibers by Lin et al. confirmed again that the stretching force of the gas jet on the polymer fluid jet played an important role during the spinning process.³⁷ In general, in the electrospinning process, the main factors that affect the morphology of the formed fibers are those including characters of polymers, solvent, value of voltage, distance between the tip and the collector, solution viscosity, surface tension, and net charge density induced by electrospinning.³² Therefore, it is significant to investigate the gas-jet/electrospinning process of liquid crystal PEI and prepare the thinner and uniform ultrafine fiber of PEI.

In this study, the gas-jet/electrospinning of solutions of liquid crystal PEI in mixtures of phenol/dichloromethane (DCM) was completed, and ultrafine fibers of PEI were prepared. During the gas-jet/electrospinning, the effects of various parameters on the diameters and morphology of the electrospun PEI ultrafine fibers were investigated.

EXPERIMENTAL

Materials

PEI was obtained from Aldrich Chemical Co. (Milwaukee, WI), and its intrinsic viscosity was 0.55 dL/g in the mixtures of phenol/DCM (1.5 : 1 w/w) at 298.2 K. DCM and phenol were purchased from Chengdu Kelong Chemical Plant (Chengdu, Sichuan Province, China) and the Joint Institute for Chemical Reagents (Chengdu, Sichuan Province, China),

respectively. They were used without further purification.

Preparation of the spinning solutions

The phenol was blended with DCM at a given weight ratio (1.5 : 1 w/w) as a solvent at room temperature. PEI was added to the mixture at fixed weight concentrations of 13, 15, 18, 20, and 22 and was then ultrasonically vibrated for 3 min at room temperature. The polymer solutions were further stirred for 1 h and were then placed in a water bath overnight to remove entrapped air.

Gas-jet/electrospinning

The PEI solutions for gas-jet/electrospinning were prepared in the mixtures of phenol/DCM at room temperature. The given concentrations of the prepared PEI solutions were 13, 15, 18, 20, and 22 wt %. The gas-jet/electrospinning was carried out in air. The gas-jet/electrospinning apparatus is shown in refs. 35 and 36. The spinning solution was placed in a syringe and was extruded to the spinneret fixed with a metal needle with inner diameters of 0.24, 0.37, 0.47, and 0.57 mm. The tip of the needle was cut flat. The spinneret was connected to a high-voltage supply, which could generate direct-current voltages ranging from 0 to 45.0 kV. A piston pump was used to feed the polymer solution at a flow rate of 3.0 mL/h. A high voltage was applied to the spinneret, through which the polymer solution passed to be spun with the help of an electrode. At the same time, nitrogen gas in storage was delivered through the pipe to the spinneret, and a gas jet occurred. The fibers were collected on clear stainless steel mesh (the collector) connected to a ground under the spinneret. The gas flow rates were 2.5, 7.5, 10.0, 12.5, and 15.0 L/min. The applied voltages were 15.0, 17.5, 20.0, and 25.0 kV. The tip-to-collector distances (TCDs) were 15, 20, 25, and 28 cm. The weight ratio of phenol to DCM was 1.5 : 1.

Scanning electron microscopy (SEM) characterization

A Hitachi (Tokyo, Japan) S-450 scanning electron microscope was used to investigate the morphology of the fibers. We determined the average diameters of the PEI ultrafine fibers by measuring the diameters of individual fibers from multiple SEM images using Adobe Photoshop 7.0 software.

RESULTS AND DISCUSSION

In this study, the following parameters were examined in detail: solution concentration, gas flow rate, applied voltage, TCD, and inner diameter of the needle. The influence of these properties on the average

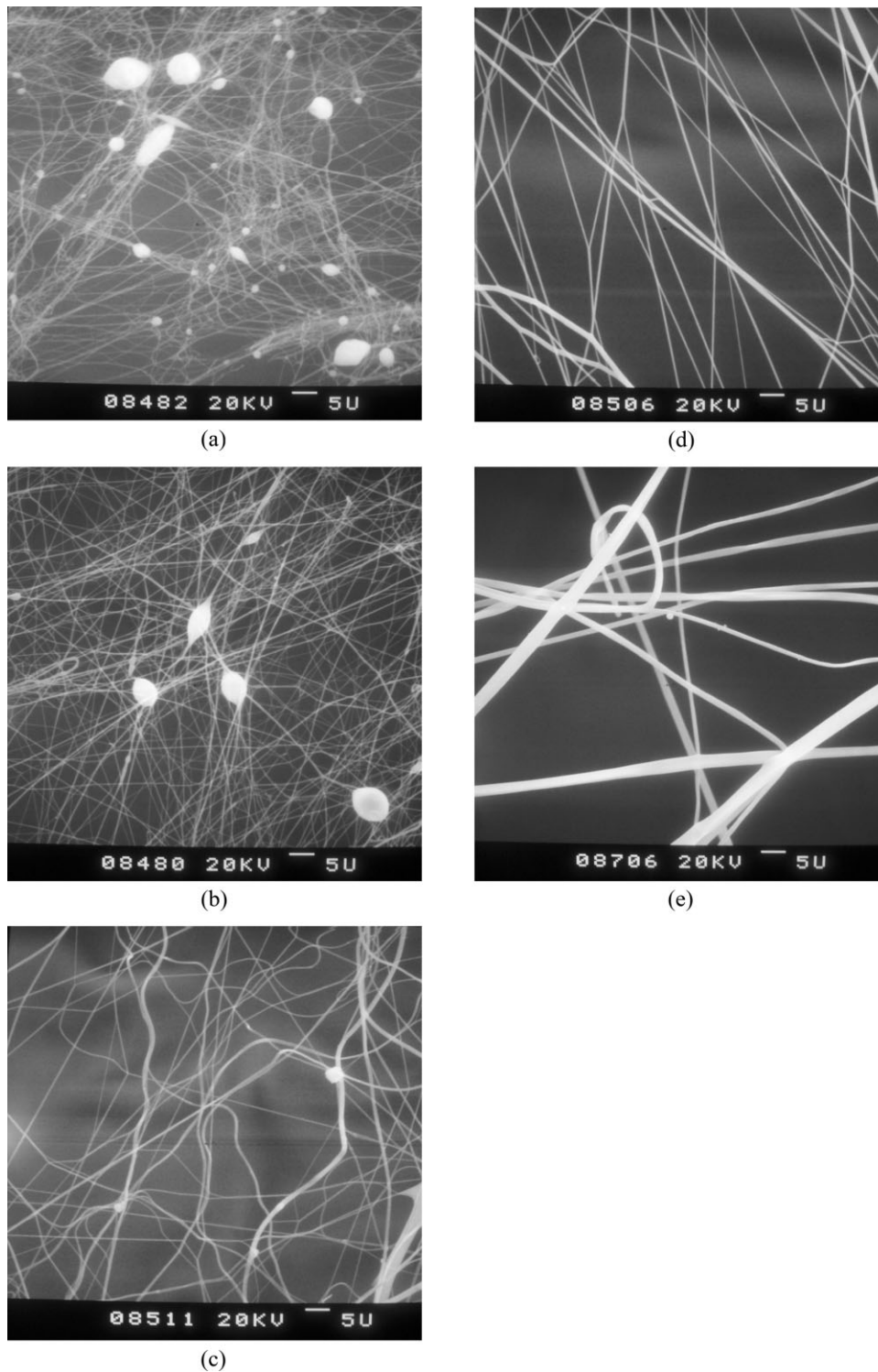


Figure 1 SEM images of the PEI gas-jet/electrospun fibers from (a) 13, (b) 15, (c) 18, (d) 20, and (e) 22 wt % PEI solutions (original magnification = 1000 \times).

diameter and morphology of the PEI ultrafine fibers is summarized and discussed next.

Solution concentration

Figure 1 shows SEM images of PEI gas-jet/electrospun fibers from different concentrations of PEI in

phenol/DCM (1.5 : 1 w/w). The concentrations of PEI were 13, 15, 18, 20, and 22 wt %. The other process parameters were as follows: the gas flow rate was 10.0 L/min, the applied voltage was 20.0 kV, the TCD was 20 cm, the inner diameter of the metal needle was 0.24 mm, and the solution flow rate was 3.0 mL/h. Table I shows the variation of

TABLE I
Average Diameter of the PEI Gas-Jet/Electrospun Fibers from PEI Solutions of Different Concentrations

Concentration (wt %)	13	15	18	20	22
Diameter (nm)	97	198	316	363	1289

fiber average diameter as the PEI concentration increased.

When the concentration increased from 13 to 18 wt %, fibers with beads were obtained, and the diameters and quantity of beads decreased. However, when the concentration of PEI was 20 wt % or more, the beads disappeared. In addition, the average diameter of the fibers gradually increased from 97 nm to 1.289 μm as the polymer concentration increased from 13 to 22 wt %. Apparently, the solution concentration played an important role in the average diameter and morphology of the gas-jet/electrospun fibers. The phenomenon was consistent with those previously recorded.^{35,37} It confirmed that concentration is an important process parameter during gas-jet/electrospinning and electrospinning.^{38,39}

The occurrence of beads in the gas-jet/electrospinning process was the result of the interaction between the surface tension and the viscoelastic force. Surface tension tends to minimize the surface area of the electrified jet and favor the formation of beads, whereas viscoelastic force favors the formation of fibers. At the low polymer concentration, the surface tension overcame the viscoelastic force. So beads appeared when the concentration was below 18 wt % [Fig. 1(a–c)]. At a high concentration, the viscoelastic force dominated the morphology of fibers, which was favorable for the prevention of the formation of beads and allowed the formation of smooth fibers. As a result, uniform PEI fibers without beads were obtained [Fig. 1(d,e)].

The increase in the average diameter of the fibers was attributed to the increase in the solution viscosity.⁴⁰ Interaction of the large molecular chains was enhanced and the viscoelastic force increased with increasing concentration. An increasing viscoelastic force that resists rapid changes in fiber shape may result in uniform fiber formation and an increase in diameter.¹ As a result, the average diameters of the PEI fibers increased gradually with the increase in solution concentration [Fig. 1(a–e)]. This corresponded with the results that a higher viscosity often results in a thicker fiber.^{35,39}

Gas flow rate

Figure 2 shows SEM images of the PEI gas-jet/electrospun fibers from 20 wt % solutions of PEI in phenol/DCM (1.5 : 1, w/w). The gas flow rates were 2.5, 5.0, 7.5, 10.0, 12.5, and 15.0 L/min. The other

process parameters were as follows: the applied voltage was 20.0 kV, the TCD was 20 cm, the inner diameter of the metal needle was 0.24 mm, and the solution flow rate was 3.0 mL/h. The experimental results show that the average diameter of the gas-jet/electrospun PEI fibers decreased from 4800 to 363 nm after an initial increase in the gas flow rate from 2.5 to 10.0 L/min; the average diameter increased from 363 to 1279 nm when the gas flow rate increased from 10.0 to 15.0 L/min. In addition, when the gas flow rate was below 7.5 L/min, interfibrillar bonding in the junction of fibers was present [Fig. 2(a,b)]. Subsequently, the interfibrillar bonding disappeared when the gas flow rate was 10.0 L/min. This indicated that the gas flow played a significant role in the average diameter and morphology of the PEI fibers during the gas-jet/electrospinning process.

The effect of the gas flow on the average diameter of the fibers resulted from the drawing force of the gas jet and the volatilization of the mixed solvents. In general, the drawing force of a gas jet has both positive and negative influences on fibers. On the one hand, the increase of the gas flow rate resulted in an increase in the drawing force, which was favorable for obtaining thinner fibers. On the other hand, the drawing force reduced the flight time and split time of the charged jet. This was not favorable for the formation of thinner fibers. In addition, the increase of the gas jet rate accelerated the volatilization of solvents from the surface of the charged jet, which caused the fiber to be formed earlier. It was also not beneficial for the formation of thinner fibers. When the gas flow rate was increased in the beginning, the mixed solvents were inadequate to evaporate off before the solution jet arrived at the collector. Fibers presented bonding in the junction of the PEI fibers [Fig. 2(a,b)]. As the gas flow continuously increased, the bonding disappeared. This indicated that the gas jet sped up the evaporation of solvents and reduced the bonding of fibers. At the same time, the drawing force of the gas flow determined the formation of the PEI fibers. It produced thinner fibers with increasing gas flow [Fig. 2(b–d)]. However, the further increase of the gas flow heavily decreased the flight time and split time of the charged jet, which led to an increase in the average diameter of the PEI fibers [Fig. 2(e)].

Applied voltage

Figure 3 shows SEM images of the PEI gas-jet/electrospun ultrafine fibers from 20 wt % solutions of PEI in a mixture of phenol/DCM (1.5 : 1, w/w) at applied voltages of 15.0, 17.5, 20.0, and 25.0 kV. The other process parameters were as follows: the gas flow rate was 10.0 L/min, the TCD was 25 cm, the

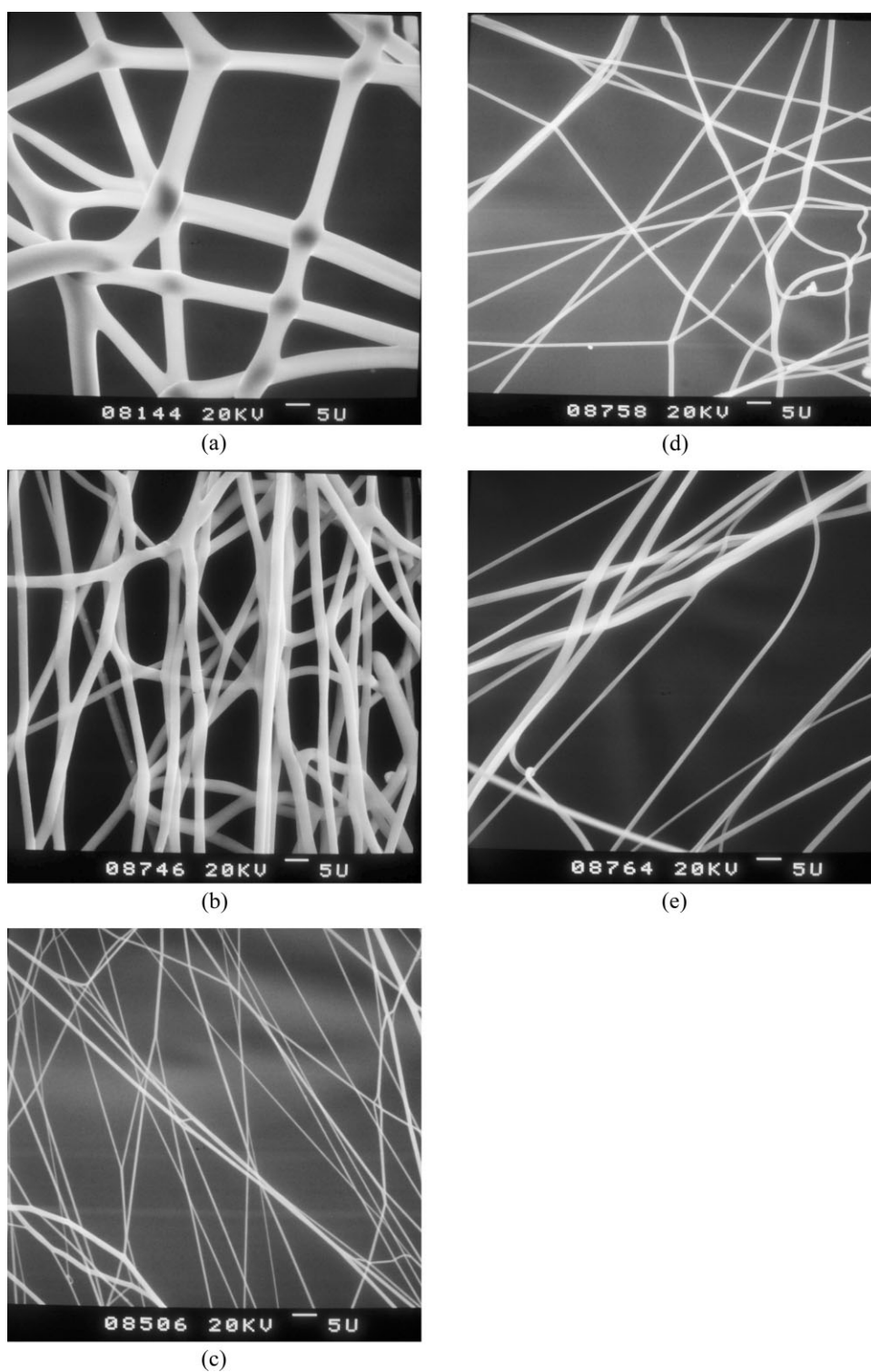


Figure 2 SEM images of the PEI gas-jet/electrospun fibers at gas flow rates of (a) 2.5, (b) 7.5, (c) 10.0, (d) 12.5, and (e) 15.0 L/min (original magnification = 1000 \times).

inner diameter of the metal needle was 0.24 mm, and the solution flow rate was 3.0 mL/h. Uniform PEI fibers without beads were obtained. The average diameters of the gas-jet/electrospun fibers changed slightly from 293 to 309 nm with increasing applied

voltage. Therefore, the voltage had no obvious influence on the average diameter and morphology of the fibers in the gas-jet/electrospinning process.

The effect of the applied voltage on the average diameter and morphology of the PEI fibers was

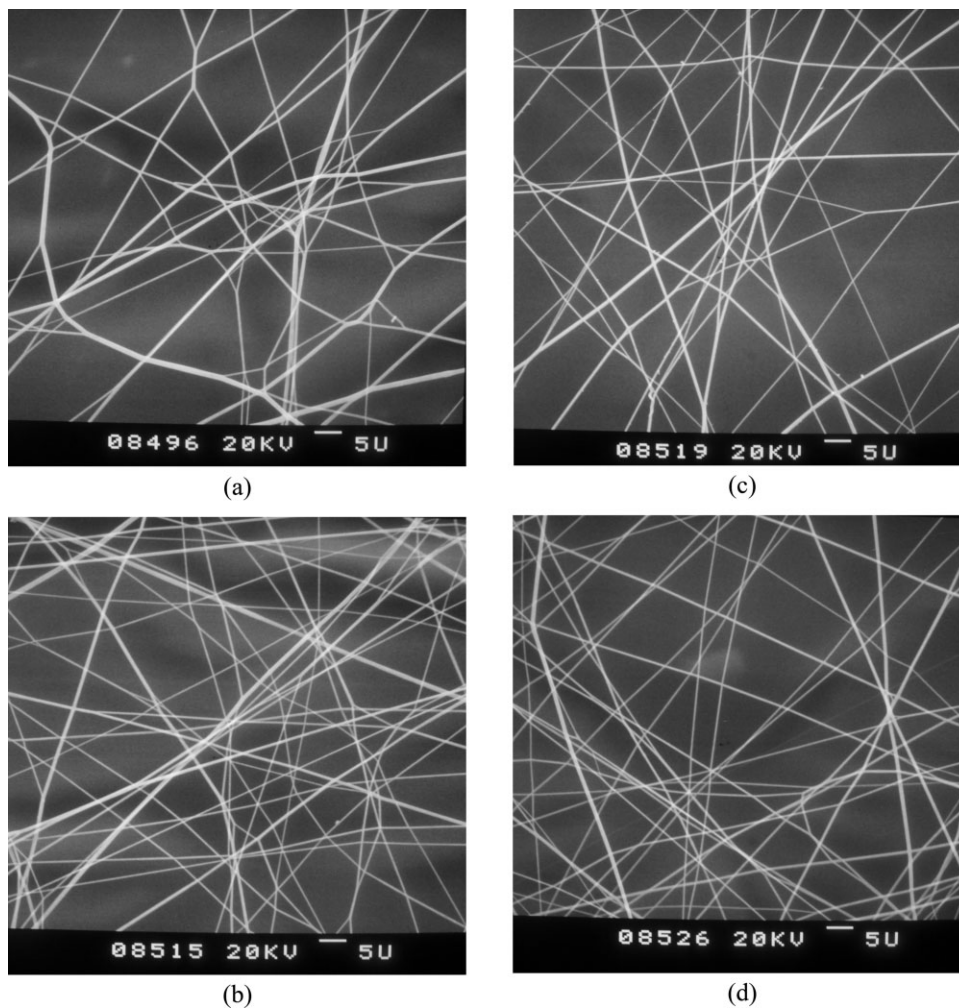


Figure 3 SEM images of the PEI gas-jet/electrospun fibers at applied voltages of (a) 15.0, (b) 17.5, (c) 20.0, and (d) 25.0 kV (original magnification = 1000 \times).

considered mainly as an incorporation of the flight time and the splittability of the charged jet. On one hand, a higher applied voltage led to a shorter flight time, which resulted in a shorter deformation to form fibers. This was disadvantageous for obtaining thinner fibers. On the other hand, the splittability of the charged jet in the electrostatic field increased with increasing voltage, and this led to the formation of fibers with a smaller average diameter. In the gas-jet/electrospinning process, the effect of the greater splittability of the charged jet was slightly beyond the effect of the shorter flight time of the charged jet, so these two opposite effects produced a slight change in the average diameter of the PEI fibers with increasing applied voltage.

TCD

Figure 4 shows SEM images of the PEI ultrafine fibers prepared at TCDs of 15, 20, 25, and 28 cm.

The concentration of the spinning solution of PEI in phenol/DCM (1.5 : 1, w/w) was 20 wt %. The other process parameters were as follows: the gas flow rate was 10.0 L/min, the applied voltage was 20.0 kV, the inner diameter of the metal needle was 0.24 mm, and the solution flow rate was 3.0 mL/h. The average diameters of the PEI gas-jet/electrospun fibers at TCDs of 15, 20, 25, and 28 cm were 1583, 363, 298, and 1218 nm, respectively. In addition, when the TCD was 15 cm, interfibrillar bonding in the junction of the fibers was present. As the TCD continued to increase, the interfibrillar bonding disappeared. Remarkably, the effect of the TCD on the average diameter and morphology of PEI fibers was significant.

In the gas-jet/electrospinning process, the electric field force played an important role in the stretching and splitting of the charged jet. When the TCD was 15 cm, the electrostatic force was so strong that the mixed solvents in the charged jet did not have

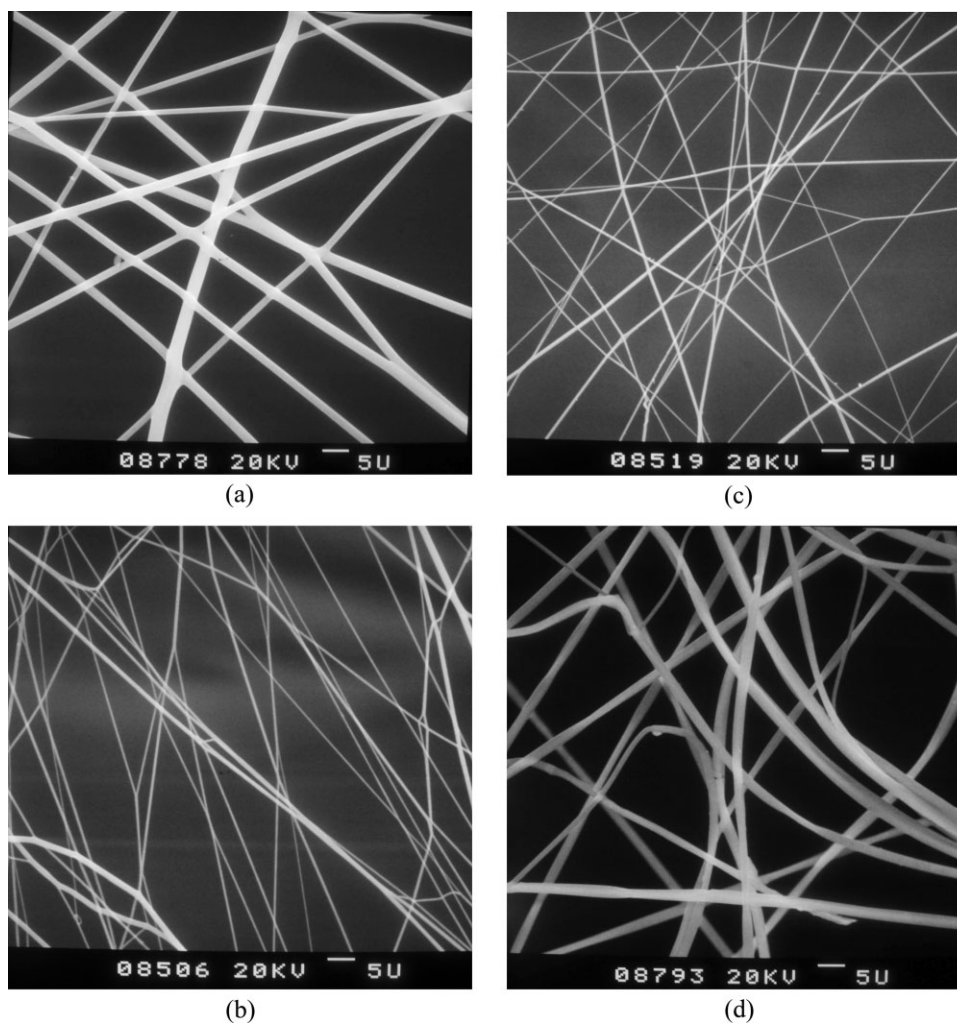


Figure 4 SEM images of the PEI gas-jet/electrospun fibers at TCDs of (a) 15, (b) 20, (c) 25, and (d) 28 cm (original magnification = 1000 \times).

enough time to evaporate fully. So the stretching and the splitting of the charged jet were not accomplished [Fig. 4(a)]. Therefore, the interfibrillar bonding in the junction of fibers appeared, and the average diameter of fibers was larger. When the TCD increased to 20 cm, the electrostatic force decreased, whereas the stretching and splitting time of the charged jet increased. At the TCD, the charged jet suffered from more sufficient stretching and splitting. As a result, thinner fibers were obtained, and the morphology of the interfibrillar bonding in the junction of the electrospun fibers disappeared. However, when the TCD was further, although it was enough to evaporate the solvents, the stretching and splitting of the charged jet declined. As a result, the average diameter of the fibers increased [Fig. 4(d)]. Therefore, the total effect of TCD on the average diameter of the PEI fibers was similar to that of the gas flow. That is, the average diameter of the PEI fibers reached a minimum

value after an initial increase at TCD and then became larger with increasing TCD.

Inner diameter of the needle

Figure 5 shows SEM images of the PEI gas-jet/electrospun ultrafine fibers from 20 wt % solutions of PEI in phenol/DCM (1.5 : 1, w/w). The inner diameters of the metal needle were 0.24, 0.37, 0.47, and 0.57 mm. The other process parameters were as follows: the gas flow rate was 10.0 L/min, the applied voltage was 20.0 kV, the TCD was 20 cm, and the solution flow rate was 3.0 mL/h. The average diameters of the PEI fibers were 363, 2187, 4017, and 4219 nm when the inner diameters of the metal needles were 0.24, 0.37, 0.47, and 0.57 mm, respectively. This indicated that the average diameter of these fibers systematically increased when the inner diameter of the metal needles was increased. The phenomenon was similar with what Lin et al. described.³⁷ In

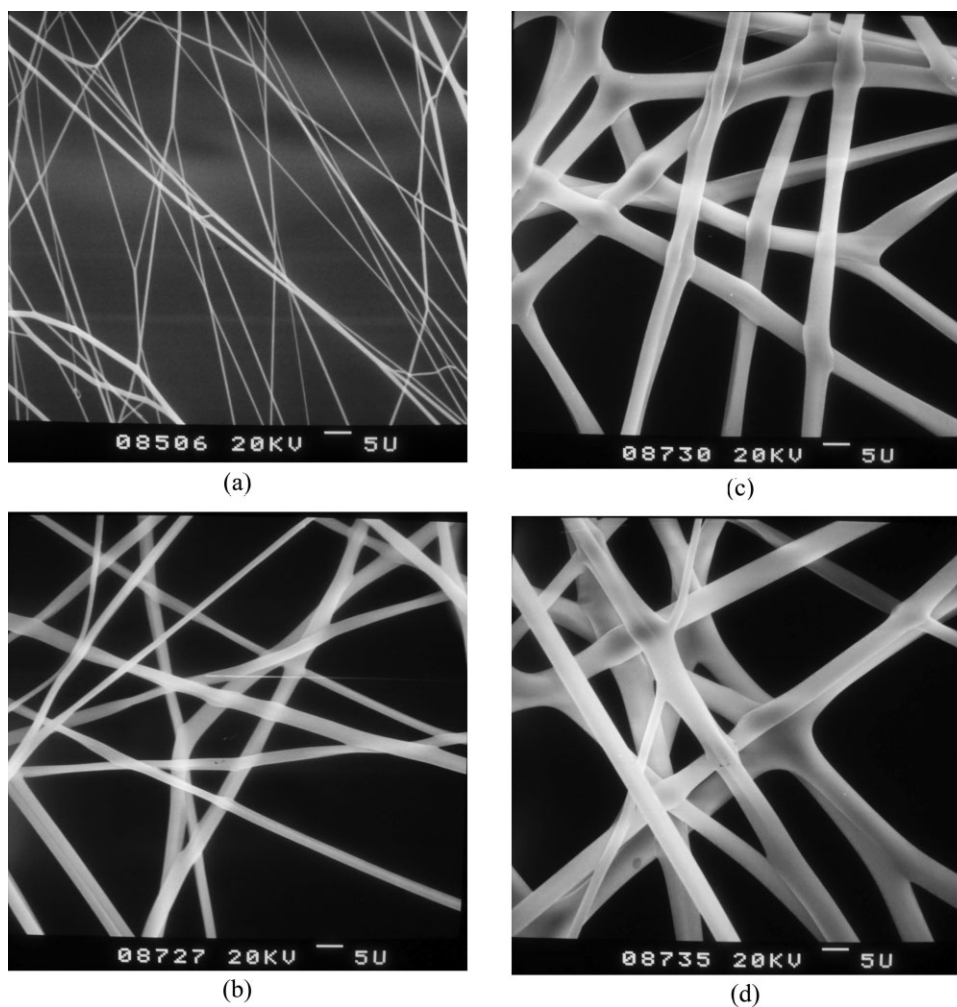


Figure 5 SEM images of the PEI gas-jet/electrospun fibers with inner diameters of the metal needle equal to (a) 0.24, (b) 0.37, (c) 0.47, and (d) 0.57 mm (original magnification = 1000 \times).

addition, interfibrillar bonding in the junction of the fibers appeared.

An interesting variation in the average diameter and morphology of the fibers was given when we considered the stretching and splitting of the charged jet in an electric field. A larger inner diameter of the needle resulted in a thicker initial charged jet. The thicker charged jet possessed a higher surface area, which led to a lower charge density in the surface of the charged jet. The lower charged density was not propitious for further stretching and splitting of the charged jet. In addition, the larger charged jet carried the larger quantity of solvent. Under the same conditions, it was harder to dry before it reached the collecting mesh. Consequently, the interfibrillar bonding in the junction of fibers appeared, and larger diameter fibers were obtained as the inner diameter of needle increased.

According to this research, different parameters had different roles in determining the average diameters and morphology of the gas-jet/electrospun PEI

fibers. It was reasonable to put all of these parameters together to produce desirable fibers. Bead-free, fine fibers with an average diameter of 293 nm were prepared successfully under the following conditions: the concentration of the polymer solution was 20 wt %, the gas flow rate was 10.0 L/min, the applied voltage was 25.0 kV, the TCD was 25 cm, the inner diameter of the metal needle was 0.24 mm, the flow rate of the solution was 3.0 mL/h, and the ratio of phenol/DCM was 1.5 : 1 (w/w).

CONCLUSIONS

In this study, ultrafine fibers of PEI were successfully prepared by the gas-jet/electrospinning of PEI/phenol/DCM solutions. On the basis of the experimental results, we suggest that the solution concentration, gas flow rate, TCD, and inner diameter of the needle had the most significant effects on the average diameters and morphology of the gas-jet/electrospun ultrafine fibers. The applied voltage had no

obvious influence on the average diameter and morphology of the gas-jet/electrospinning PEI fibers. An increase in the polymer concentration in spinning solutions resulted in the formation of ultrafine fibers with a larger diameter. The use of a 13 wt % polymer solution gave PEI ultrafine fibers with an average diameter of about 100 nm. However, the morphology of mixed bead fibers was formed when the concentration of PEI in phenol/DCM was below 20 wt % during gas-jet/electrospinning. The larger inner diameter of the needle and the smaller gas flow rate led to ultrafine fibers with a larger average diameter. The effect of TCD on the average diameter of the PEI fibers was similar to that of the gas flow rate. The average diameter of the PEI fibers reached a minimum value of 298 nm after an initial increase in TCD and then became larger with increasing TCD. Typically, uniform PEI fibers with an average diameter of 293 nm were prepared by gas-jet/electrospinning with the following process parameters: the concentration of the polymer solution was 20 wt %, the gas flow rate was 10.0 L/min, the applied voltage was 25.0 kV, the TCD was 25 cm, the inner diameter of the metal needle was 0.24 mm, the ratio of phenol to DCM was 1.5 : 1 (w/w), and the flow rate of the solution was 3.0 mL/h.

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