Melt Electrospinning of Low-Density Polyethylene Having a Low-Melt Flow Index

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ABSTRACT: Melt electrospinning is a cheaper, more environmentally friendly, and safer alternative to solution electrospinning. We have designed a novel melt spinning device which incorporates a reverse of the normal polarity, with the capillary grounded and the collector grid at positive potential. The apparatus is much simpler and more economical than conventional equipment because no syringe pump is required. Low-density polyethylene (LDPE) with a low-melt flow index of 2 g/10 min, which is not suitable for spinning using current commercial methods, was chosen to highlight the advantages of melt electrospinning in general, and our device in particular. The effects of varying the electrospinning parameters such as temperature, electrostatic field, spinning distance, and capillary inner diameter, have been studied. Although it was found that temperatures higher than normal processing temperatures had to be employed in our electrospinning system to reduce the viscosity of the polymer melt sufficiently, good quality fibers with smooth and even surfaces, most of which had diameters smaller than 15 μm, were electrospun successfully. It was observed that there was an optimum point for the spinning distance (14–15 cm) and the capillary inner diameter (0.4–0.6 mm) to get fine fiber. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 166–175, 2009

Key words: melt electrospinning; melt flow index; LDPE; thin fibers; DSC

INTRODUCTION

Recent years have seen a renaissance in interest in electrospinning as a means of producing microfibers in a way which is easier and more efficient than traditional commercial fiber manufacturing methods.[1–5] It has also been reported that nanofibers, i.e., fibers having a diameter smaller than 100 nm, can be produced through electrospinning.[2] The vast majority of research activity has been focused on solution electrospinning with far fewer studies of melt electrospinning.[3] There are, however, a number of disadvantages of solution electrospinning associated with the use of volatile solvents. First, there is a safety problem, particularly when the electrospun fibers are to be used in biomedical applications, such as tissue scaffolds, wound dressings, artificial skin, and drug delivery systems, since it is necessary to ensure that there is no residual solvent in the fibers. There is also a problem of very low-productivity since more than 90% of the mixture being electrospun evaporates during the process, so that productivities as low as 0.01 g/min are common. Furthermore, the capillary, an important component of an electrospinning device, frequently becomes blocked which interrupts the continuous process and further reduces productivity. These are probably the main reasons why commercial fiber manufacturers continue to shy away from using electrospinning,[1] but there are also a number of other problems. The toxicity and cost of most solvents used in solution electrospinning means that solvent recycling is necessary on both environmental and economic grounds; such processes can be both expensive and complex.[1] In the case of polymers such as polypropylene, one of the most widely used polymers in commercial spinning, and polyethylene, it is difficult to find a suitable solvent since the materials have limited solubilities in volatile solvents at room temperature.[4] Finally, evaporation of the solvent can affect the smoothness of the final fibers and porous fiber surfaces are commonly obtained which has an adverse effect on the mechanical properties of the fibers.

Melt electrospinning of fibers does not require the use of solvents and hence has been proposed as a cheaper, more environmentally friendly, and safer alternative to solution electrospinning. In spite of its potential benefits, however, there have been

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relatively few reported studies of melt electrospinning. There are two main reasons for this[6–8]: (i) The viscosity of a molten polymer is much higher than that of a solution so that larger fiber diameters, mostly submicron and micron, are obtained by melt electrospinning, whereas the smallest fibers produced in solution electrospinning are tens of nanometers in diameter, and the nanotechnology bandwagon has tended to ensure that funding has been directed toward solution electrospinning; (ii) The apparatus required for melt electrospinning is much more complicated than that for solution electrospinning because a temperature control system is required to melt the polymer, and there is interference between the high-voltage supply system and the sensors in the temperature control system.

In this article, we describe a device for melt electrospinning which is much simpler than those which have been previously reported.[1–19] There are two key features responsible for the simplification. First, a syringe pump, an expensive element which is generally used to control the flow rate of the molten polymer in melt electrospinning devices, is not required. Second, the electrodes have been reversed to avoid the interference noted above. Unlike a conventional melt electrospinning device where the collector grid is grounded, in our case the capillary is grounded so that the cylinder loaded with the polymer melt can be heated directly by means of an electrical heating ring, and the thermal sensor is located inside the cylinder where it is not subject to interference from the high-voltage system. In fact, this not the first time that reversing the electrodes has been reported[9] but it has not been widely used, probably because of the lower electrostatic forces generated. By raising the electrostatic voltage, however, it is possible to supply a sufficiently large electrostatic force.

The primary reason why the diameters of the fibers produced in melt electrospinning are much larger than those produced by solution electrospinning is the high-viscosity of the polymer melt. The viscosity of the melt depends on the melt flow index of the polymer, the higher the melt flow index, the lower the viscosity. Therefore, in commercial spinning, the melt flow index of the materials used is very high to minimize the viscosity. In the case of polypropylene, for example, materials with a melt flow index in the range 400–1200 g/10 min are commonly used in commercial spinning in the US. To highlight the advantages of melt electrospinning in general, and our device in particular, the material used in this work was low-density polyethylene (LDPE) with a melt flow index of 2 g/10 min; it is generally accepted that a polymer with such a low-melt flow index is not suitable for spinning by conventional methods. We report here a systematic study of the effects of varying the electrospinning parameters, including temperature, electrostatic field (varying voltage at fixed spinning distance and varying spinning distance at fixed voltage), spinning distance (in a fixed electrical field), and capillary inner diameter, on the properties of the electrospun LDPE fibers.

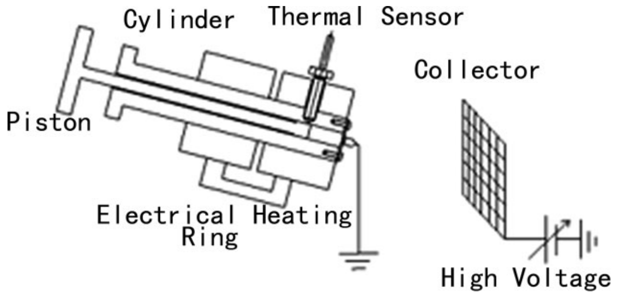

Figure 1 Schematic illustration of the experimental apparatus.

EXPERIMENTAL

Materials

Low-density polyethylene (LDPE), purchased from Beijing Yanshan Petrochemical Corp., China had the following characteristics: (i) Brand: 1I2A-1; (ii) Density: 0.9192 g/cm^3 (ISO/DIS 1183 : 1984); (iii) Melt flow index: 2 g/10 min (ISO 1133 : 1997); (iv) Elongation at break: 550% (ISO 527-1 : 1993); (v) Tensile strength: 15 MPa (ISO 527-1 : 1993); (vi) Vicat softening point: 85°C.

Equipment

Figure 1 is a schematic illustration of the equipment employed in this study. The cylinder and piston were manufactured of 45 steel and the capillary (not marked in the diagram) was made from stainless steel. The capillaries used in this article have a series of inner diameter (0.7 mm, 0.6 mm, 0.4 mm, and 0.2 mm) and have a fixed land length of 1 mm. The collector was a flat wire mesh cut to a specific size. The high-voltage supply device, purchased from Tianjin Dongwen High Voltage Power Supply Plant, had a maximum output of +60 kV and maximum current output of 2 mA. The electrical heating ring was custom-built with a power of 300 W. The Pt100 thermal sensor was purchased from Beijing Sailing Technology Company.

Electrospinning

The cylinder was first heated to the required temperature (in the range 315–355°C). (Instead of the oven, we used the electrical heating ring to melt the polymer. The temperature of the cylinder was controlled by a temperature control system. Each experiment was carried out after the temperature achieving the steady state). The piston was then extracted, the cylinder was loaded with LDPE, and the piston was replaced in the cylinder to compact the polymer and vent the air in the cylinder. After about 10 min, when the liquid flow began to emerge, the cylinder was gradually tilted until a cone (Taylor cone) formed at the apex of the capillary. The high-voltage supply device was then switched on and the voltage adjusted to the required value to complete the electrospinning process. It should be stressed that, unlike a syringe pump, the piston remained in the same position throughout because of the extremely small gap between the cylinder and the piston, and the polymer was drawn out purely by the electrostatic force rather than by movement of the piston. It was observed that as long as the cone appeared at the apex of the capillary, this process proceeded continuously until the cylinder was empty. The electrospinning process stopped immediately when the high-voltage supply device was turned off, confirming that it was the electrostatic force which overcame the viscosity forces in the polymer melt rather than gravity or any other force.

Characterization

The morphologies of the electrospun fibers were observed by scanning electron microscopy (SEM, Hitachi S4700, 20 kV accelerating voltage). The fiber samples were plated with a thin layer (several nanometers thick) of platinum. Fiber diameters were measured using the measuring tool in Adobe PhotoShop. First, obtain a pixel value by measure the scale and then measure the fiber to get another pixel value. The fiber diameter can be calculated by the following expression:

$$d = \mathrm{Fv}/\mathrm{Sv} \times S \tag{1}$$

where, Fv is the fiber pixel value, Sv is the scale pixel value, S is the scale, and d is the fiber diameter.

The average fiber diameter (AVG) and standard deviation (SD) were calculated from 100 measurements of random fibers at each spinning condition. The degree of crystallinity was estimated using differential scanning calorimetry (DSC, Perkin–Elmer Pyris 1) by heating from 25 to 150°C at 10°C/min, under an N_2 atmosphere.

RESULTS AND DISCUSSION

Effect of varying the heating temperature on the properties of the electrospun fibers

As discussed above, the high viscosity of the polymer melt is the primary reason why melt electrospinning cannot generally produce fibers as thin as those from solution electrospinning, although Möller and coworkers[13] have shown that polypropylene fibers with a diameter smaller than 1 μm can be prepared by adding viscosity-reducing additives during melt electrospinning. The viscosity of the polymer melt can be affected by many factors including the molecular weight of the polymer, the melt flow index of the polymer, the temperature of the polymer melt, the heating time of the polymer melt, as well as the presence of additives. For example, extensive studies by Lyons et al.[9] have shown that the molecular weight of the polymer has a significant influence on the feasibility of producing fibers, and that the lower the molecular weight, the thinner the fibers obtained.

Since we have deliberately chosen a polymer (LDPE) which presents a considerable challenge by virtue of its very low-melt flow index (2 g/10 min), we employed spinning temperatures in the range 315–355°C, just below the decomposition temperature of the polymer (360–400°C), to decrease the viscosity of the melt. At these temperatures, which are much higher than normal processing temperatures, LDPE flowed like a liquid and ultrathin fibers appeared readily and continuously under an applied field. This can be attributed to the microscopic chain structure of the polymer which favors the formation of macroscopic fibers.

Figure 2 shows SEM micrographs of electrospun fibers prepared with different melt temperatures in the range 325–355°C. In each case, melt electrospinning proceeded very successfully and all the fibers obtained had an average diameter smaller than 15 μm. The standard deviations (SD) indicated that the fibers were relatively uniform in diameter compared with the situation in Lyons priord work,[9] where the standard deviations were between 2.01 and 8.22. Figure 3 is an expanded image of Figure 2(D) and shows that the fibers had the smooth and even surfaces desired by all commercial fiber manufacturers, unlike the situation in solution electrospinning, where the surfaces of the fibers were porous commonly.[20] Figure 4 shows the porous surfaces of solution electrospun fibers. This phenomenon in solution electrospinning was unavoidable because of the evaporation of the solvent in the spinning process. As a result, it would affect the mechanical properties of the fibers greatly.

It should be noted that the ultrathin LDPE fibers we obtained are much smaller than those prepared by other workers using electrospinning of similar materials. For example, Larrondo and Manley[10–12] reported that high-density polyethylene (HDPE) with the same low-melt flow index gave electrospun fibers with diameters of several hundreds of microns. The reason why we can make much thinner

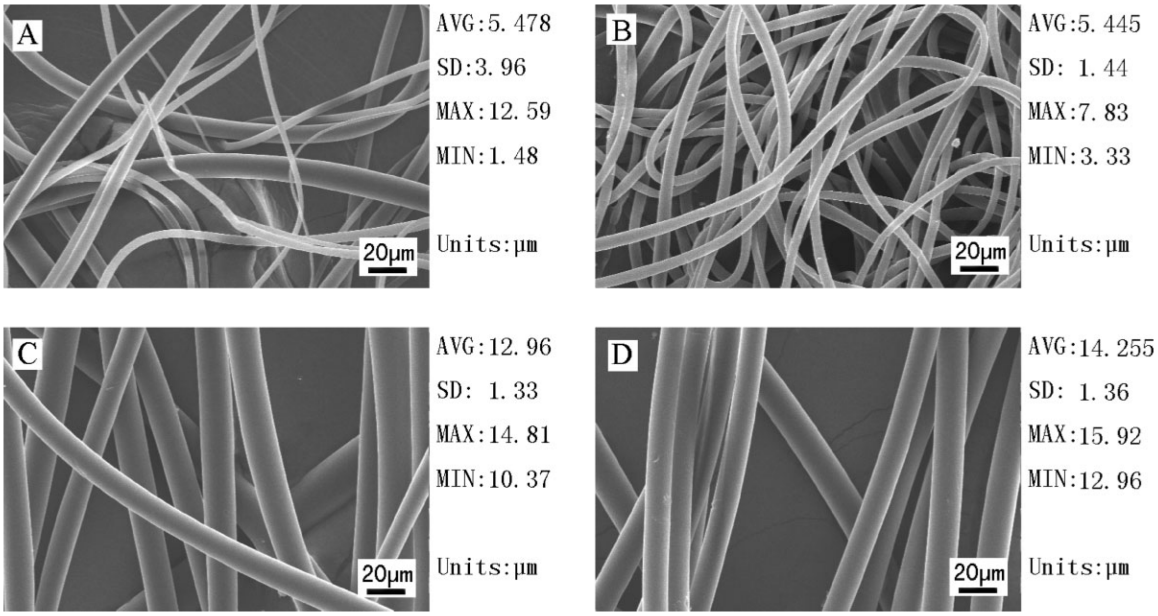

Figure 2 SEM images of LDPE melt electrospun fibers produced using a voltage of 40 kV, a capillary inner diameter of 0.6 mm, a spinning distance of 14 cm, and cylinder temperatures of (A) 355°C, (B) 345°C, (C) 335°C, and (D) 325°C. (N.B. The temperature given is the cylinder temperature because the thermal sensor was not in direct contact with the polymer melt, and the actual temperature of the polymer melt will be slightly lower than the values given). The average fiber diameter (AVG), standard deviation (SD), maximum (MAX), and minimum (MIN) fiber diameters are also given).

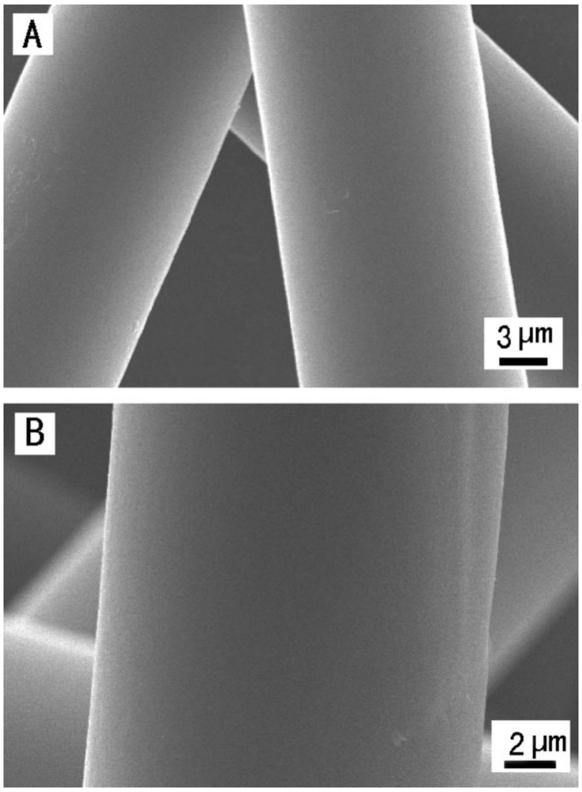

Figure 3 SEM images of LDPE melt electrospun fibers produced using a voltage of 40 kV, a capillary inner diameter of 0.6 mm, a spinning distance of 14 cm, and a cylinder temperature of 325°C.

fibers than those of Larrondo and Manley is that we operate at much higher temperatures to decrease the viscosity. At these high temperatures, the molten polymer was observed to flow like a liquid, allowing ultrathin fibers to be produced continuously and easily.

Although higher temperatures lead to decreased fiber diameters, as shown in Figure 5, clearly the maximum temperature which can be employed is limited by the decomposition temperature of the polymer. In the case of a cylinder temperature of 355°C, the color of the polymer melt turned from

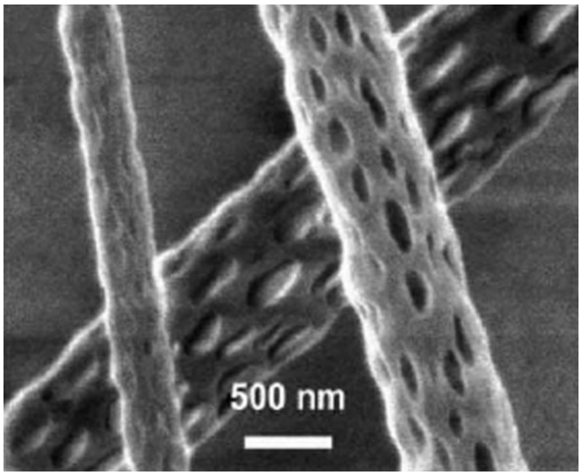

Figure 4 SEM image of solution electrospun fibers.[20]

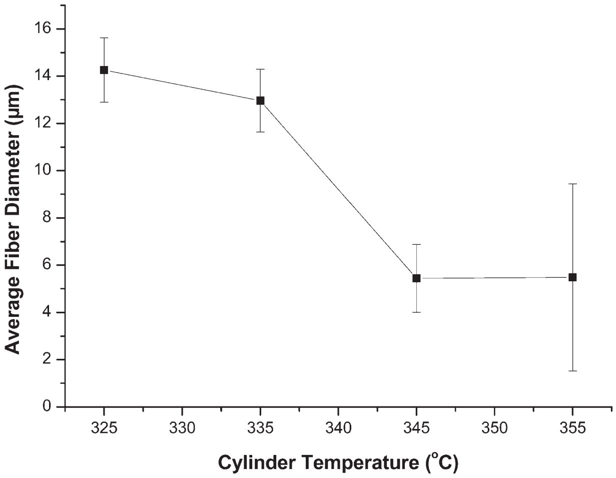

Figure 5 Plot of average fiber diameter as a function of cylinder temperature.

colorless to yellow after heating for 20 min, indicating the onset of decomposition of the polymer. Therefore, temperatures below 355°C were employed in the following experiments.

Effect of varying the electrostatic field on the properties of the electrospun fibers

The magnitude of the electrostatic force, which is the only driving force in electrospinning, can be controlled by varying the strength of the electrostatic field. Varying the voltage at fixed spinning distance (the distance between the two electrodes) and varying the spinning distance at fixed voltage both result in a change in the electrostatic field. The first method has been generally employed by other workers[1] but since the two processes may have different effects, both were carried out independently in the following experiments.

The voltage may be varied over a wide range. The minimum value is that which gives an electrostatic force which is sufficient to overcome the surface tension and viscosity forces in the molten polymer so that the melt jet can emerge from the capillary, with the maximum value being limited by the onset of the electric discharge phenomenon. Figure 6 shows the effect of varying the voltage in the range 30–60 kV with a fixed spinning distance of 18 cm.

At low-voltage (30 kV), the speed of the melt jet was very low, giving rise to fibers [Fig. 6(A)] with a large average diameter (>30 μm). At higher voltages, the speed of the melt jet was increased leading to the formation of fibers with a diameter of less than 15 μm [Fig. 6(B–D)]. In addition to the higher speeds of the melt jet, at high voltages a whipping motion of the melt jet was observed in the region near the collector. It has been suggested[4,14] that this whipping motion also contributes to the observed decrease in fiber diameter. The average fiber diameter showed a continuous decrease as the electrostatic field was increased by raising the voltage at fixed spinning distance as illustrated in Figure 7.

Figure 8 shows the effect of changing the electrostatic field by varying the spinning distance in the range 10–18 cm at a fixed voltage of 40 kV. As noted

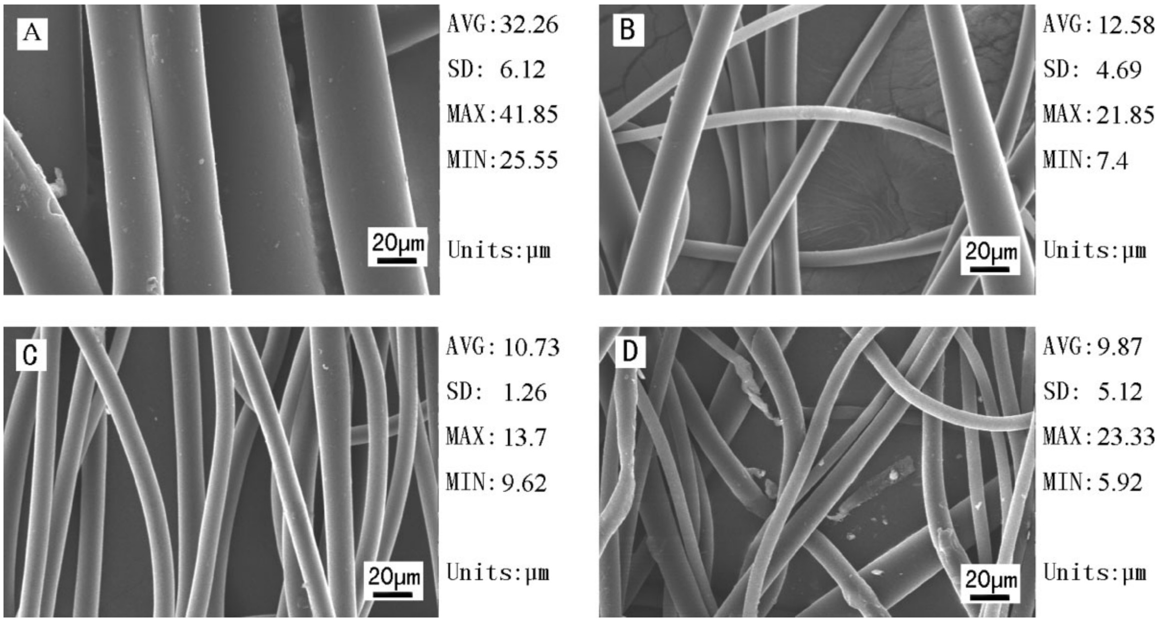

Figure 6 SEM images of LDPE melt electrospun fibers produced using a capillary inner diameter of 0.6 mm, a spinning distance of 18 cm, a cylinder temperature of 315°C, and voltages of (A) 30, (B) 40, (C) 50, (D) 60 kV.

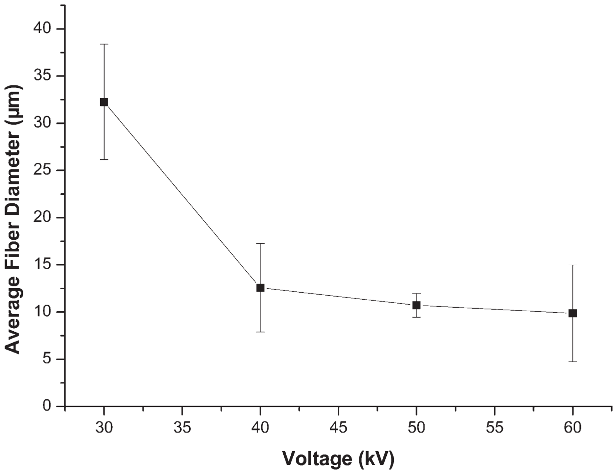

Figure 7 Variation of average fiber diameter with voltage at fixed spinning distance.

above, this process has not been widely studied. All of the fibers obtained in these experiments had an average diameter of less than 15 μm.

Decreasing the spinning distance at constant voltage leads to an increase in the electrostatic field. Interestingly, the average diameter of the fibers did not show the expected monotonic decrease as a function of increasing electrostatic field, as shown in Figure 9. Although the average fiber diameter initially decreased as the spinning distance was reduced from 18 to 14 cm, there was a pronounced increase in average fiber diameter at lower spinning distances.

These results were reproducible and we suggest that at short spinning distances, the melt jet does not have sufficient time to elongate, so that the final fibers are relatively thick. Increasing the spinning distance allows more time for the melt jet to elongate, resulting in thinner fibers. If the spinning distance is too long, however, the reduced electrostatic field results in thicker fibers. These results suggest that choice of the optimum spinning distance is crucial if high-quality melt electrospun fibers are to be obtained. This factor has not generally been taken into consideration in previous work.

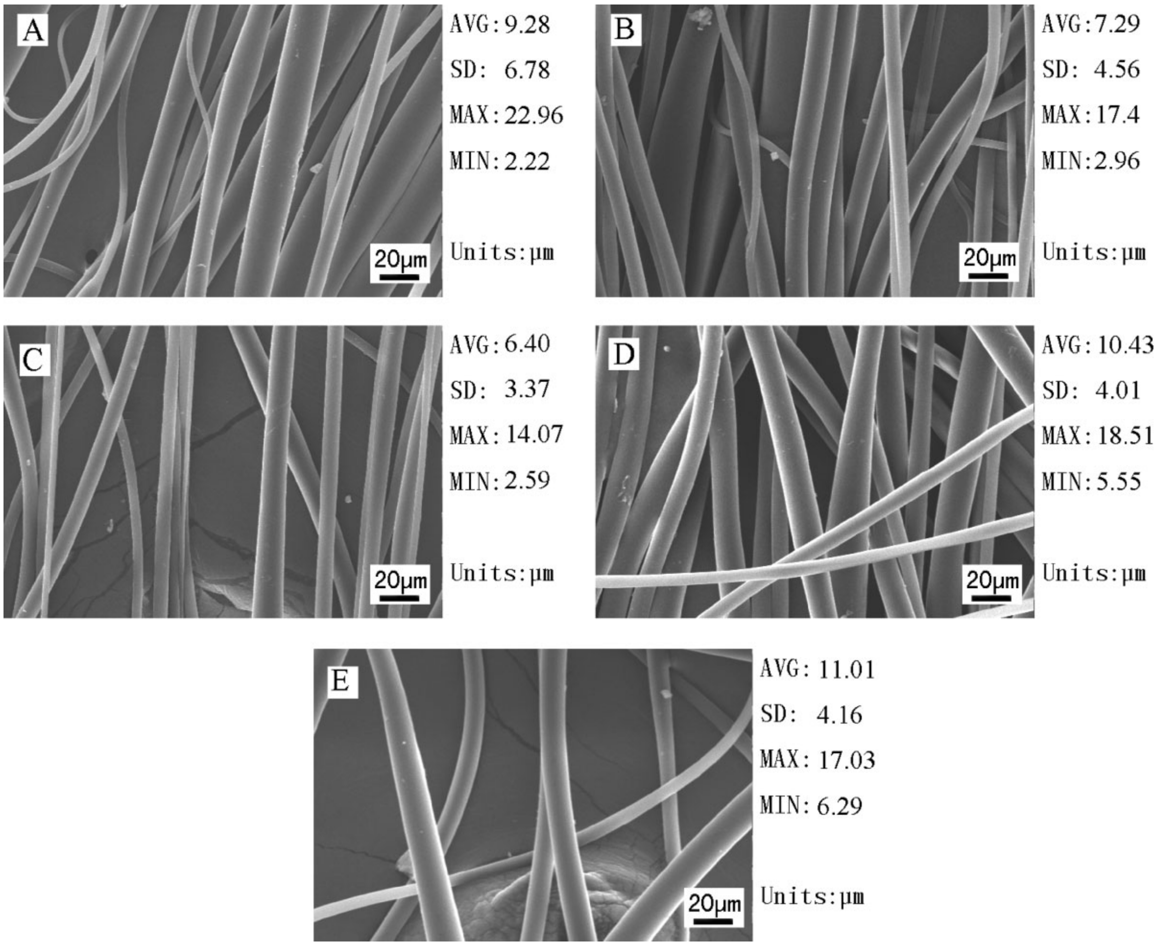

Figure 8 SEM images of LDPE melt electrospun fibers produced using a voltage of 40 kV, a capillary inner diameter of 0.6 mm, a cylinder temperature of 315°C, and spinning distances of (A) 10, (B) 12, (C) 14, (D) 16, and (E) 18 cm.

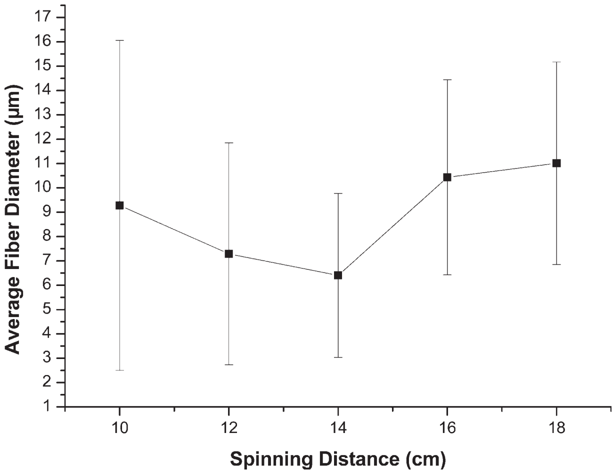

Figure 9 Variation in average fiber diameter with spinning distance at fixed voltage.

Effect of varying the spinning distance with a fixed electrical field on the properties of the electrospun fibers

To further investigate the effect of varying the spinning distance on the properties of the electrospun fibers, a series of experiments were carried out in which the spinning distance was varied over the range 5–20 cm, with the voltage being simultaneously adjusted to give a constant electric field of 3 kV/cm. SEM micrographs of the resulting fibers are shown in Figure 10.

Since the electric field was constant, the fibers were drawn out by the same magnitude of electrostatic force. At short spinning distance and low voltage, relatively thick fibers were produced as expected [Fig. 10(A)] since the velocity of the melt jet is low by virtue of the low voltage and the short spinning distance does not allow sufficient elongation of the fibers. As the voltage and spinning distance were increased, the average fiber diameter decreased as shown in Figure 11, reaching a minimum for 45 kV/15 cm. At further higher voltages and spinning distances, thicker fibers were obtained however. Since the earlier results (Fig. 7) have shown that the average fiber diameter decreases montonically with increasing voltage, the minimum in average fiber diameter can be ascribed to the variation in spinning distance. At too long spinning distances, the melt jet solidifies before reaching the collector so that thicker fibers result, despite the high voltage. These results are consistent with those in the previous section and highlight the importance of selecting the optimum spinning distance for successful melt electrospinning.

Effect of varying the capillary inner diameter on the properties of the electrospun fibers

Figure 12 shows the effect of varying the capillary inner diameter on the morphology of the electrospun fibers. It has previously been reported that the fiber diameter decreases as the capillary inner diameter decreases although, as Larrondo has pointed out, that the effect is generally not very large.[10] As shown in Figure 13, however, in our experiments

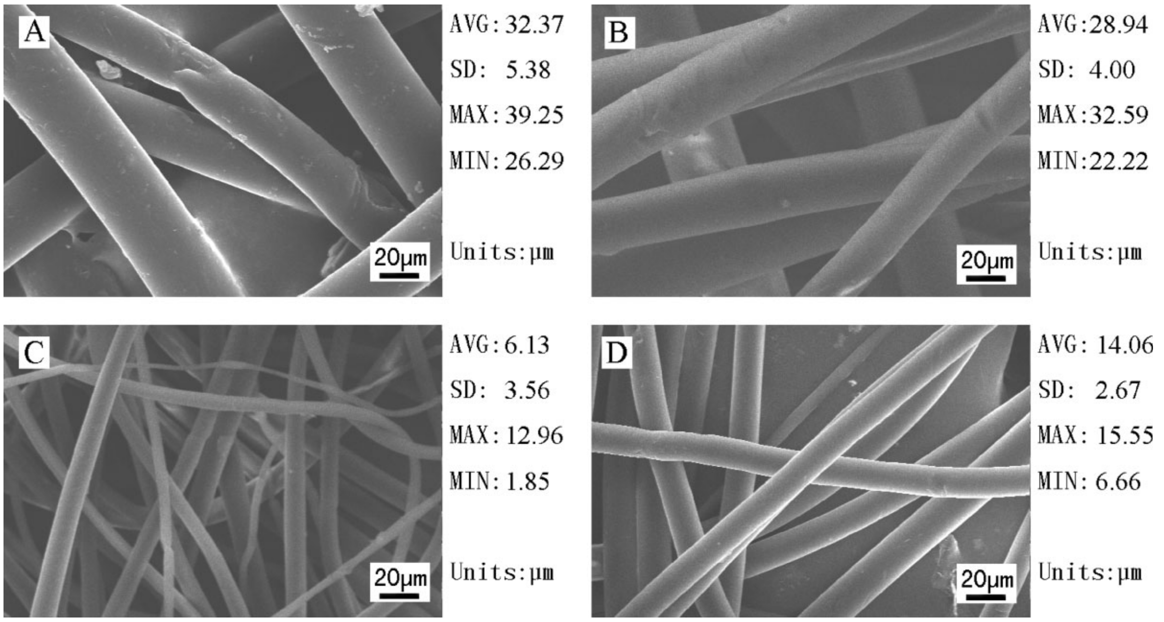

Figure 10 SEM images of LDPE melt electrospun fibers produced using a capillary inner diameter of 0.2 mm, a cylinder temperature of 325°C, and a fixed electric field of 3 kV/cm generated by different combinations of voltage/spinning distance (A) 15 kV/5 cm, (B) 30 kV/10 cm, (C) 45 kV/15 cm, (D) 60 kV/20 cm.

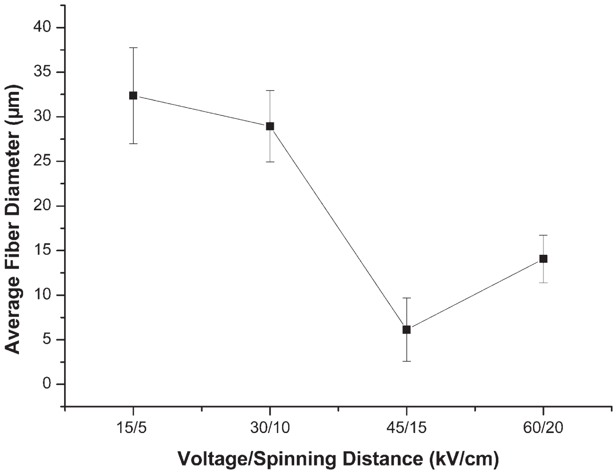

Figure 11 Variation of average fiber diameter with spinning distance at fixed electrostatic field.

although the average fiber diameter did show the expected initial decrease with decreasing capillary inner diameter, once the diameter was reduced below 0.4 mm the average fiber diameter increased significantly. It is likely that for very small capillary inner diameters, the extremely small orifice results in high pressures being required to form the Taylor cone, which in turn leads to increased polymer melt flow to the apex of the capillary. As a result, thicker fibers emerge. For our apparatus, the optimum capillary inner diameter was found to be in the range 0.4–0.6 mm.

Degree of crystallinity of the electrospun fibers

Crystal properties are some of the most important factors which determine the mechanical properties of the fibers. In particular, the elastic modulus of the fibers will increase as the degree of crystallinity increases and thus a higher degree of crystallinity will result in better mechanical properties. It has previously been reported by several authors that electrospun fibers are characterized by a lack of crystallinity.[5,18,19] In our case, however, the fibers we

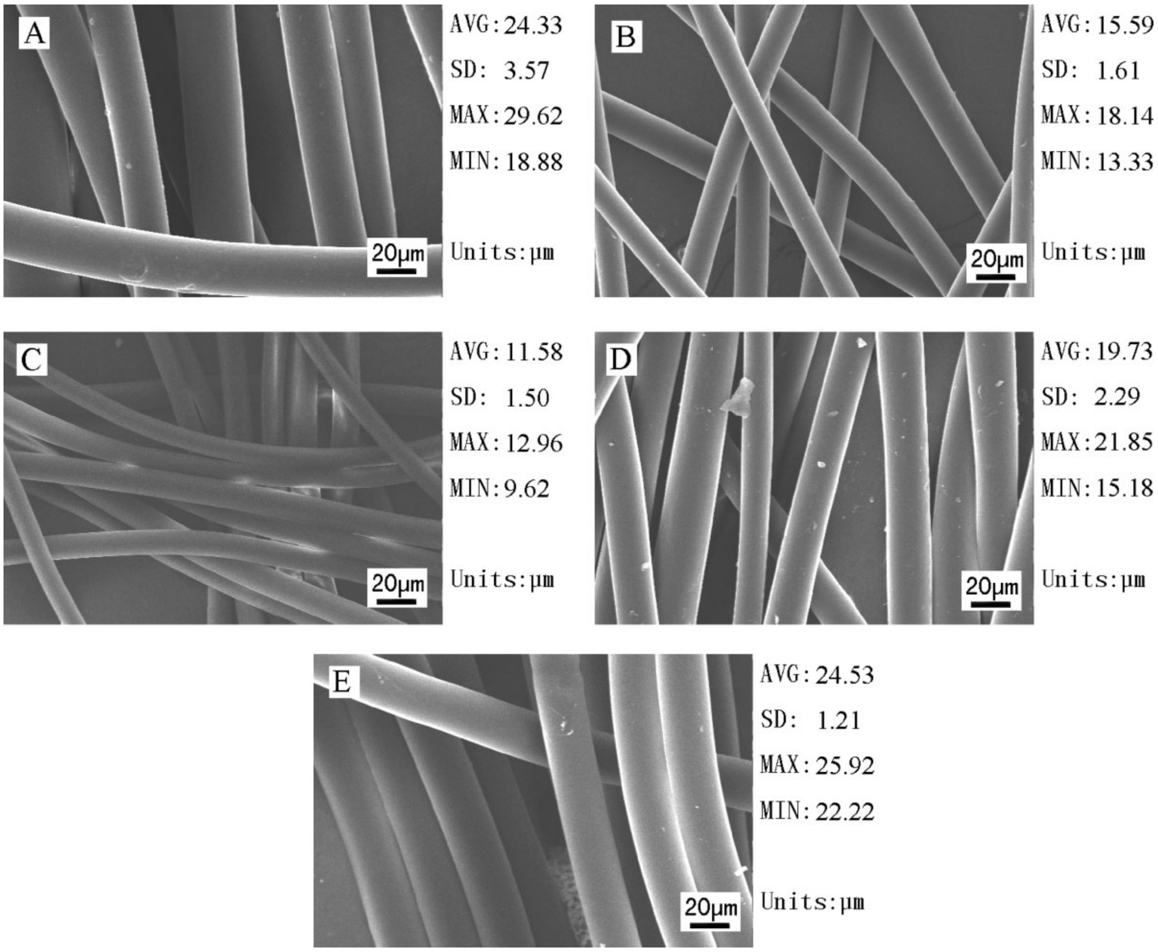

Figure 12 SEM images of LDPE melt electrospun fibers produced using a voltage of 40 kV, a spinning distance 14 cm, a cylinder temperature of 325°C, and capillary inner diameters of (A) 0.7 mm, (B) 0.6 mm,(C) 0.4 mm, (D) 0.34 mm, (E) 0.2 mm.

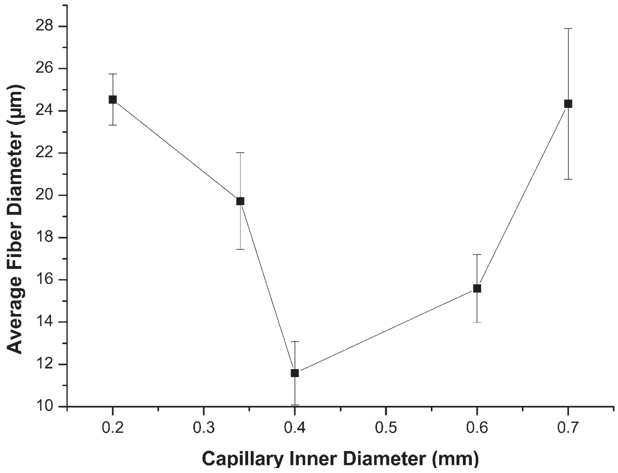

Figure 13 Variation of average fiber diameter with capillary inner diameter.

obtained do exhibit crystallinity. As shown in Figure 14, a melt peak was observed at 106.0°C. The presence of a melt peak confirms that crystallinity is present, because the temperature of the sample does not rise until all of the crystals have melted. The two points (X1, Y1) and (X2, Y2), at the ends of the peak baseline (not marked in the diagram), can be used to calculate the peak area which corresponds to the heat of fusion, ΔH. The degree of crystallinity c can be obtained by the following expression:

$$c = \Delta H/\Delta H_0 \times 100\% \qquad (2)$$

where, ΔH_0 is the heat of fusion for 100% crystalline PE, 287.3 J/g.[21] The calculated value of c (41%) indicates that a high-degree of crystallinity exists in the electrospun fibers.

Figure 15 shows the DSC curve of LDPE resin which was used in this article. The melt peck and the heat of fusion are 105.7°C and 115.403 J/g which close to those in the DSC cure of LDPE melt electrospun fibers (106°C,117.410 J/g). This means that the LDPE processing property is not significantly changed in the spinning process. However, because of the high temperature, there might be some chemical changes like decomposition occurred in the process. The degree of crystallinity c calculated by expression (2) is 40.17% which is a little lower than that of LDPE melt electrospun fibers. The higher degree of crystallinity in melt electrospun fibers might owe to the high-drawing process in spinning process.

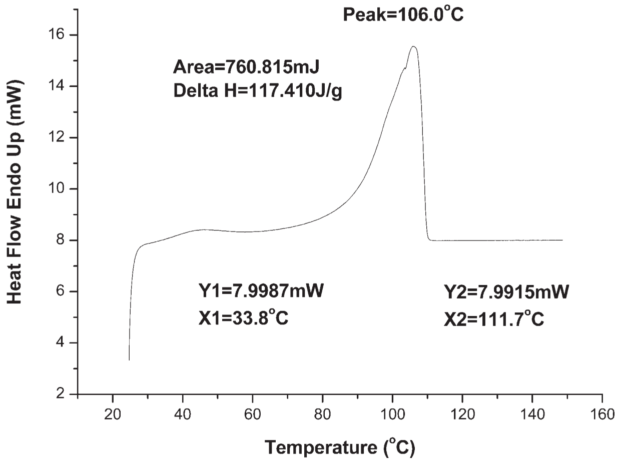

Figure 14 DSC curve of LDPE melt electrospun fibers produced using a voltage of 40 kV, a capillary inner diameter of 0.6 mm, a spinning distance of 14 cm, and a cylinder temperature of 335°C.

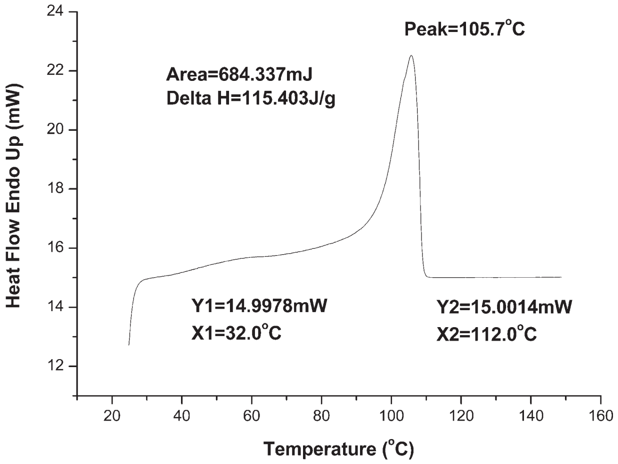

Figure 15 DSC curve of the as-received LDPE resin.

CONCLUSIONS

A simple melt electrospinning apparatus incorporating a reversed electrode polarity with the capillary being grounded and the collector grid at positive potential has been constructed. The apparatus is much simpler and more economical than conventional equipment because no syringe pump is required. LDPE with a low-melt flow index of 2 g/10 min, making it unsuitable for electrospinning by current commercial methods, has been successfully electrospun using the apparatus. Ultrathin fibers, most of which were smaller than 15 μm in diameter, were obtained. The resulting fibers were of good quality, having relatively smooth and even surfaces and a high-degree of crystallinity. By studying the effect of varying the experimental parameters, it was shown that minimizing the viscosity is the key to successful melt electrospinning. Thus, the melt flow index (or molecular weight) and the spinning temperature are the most important factors in melt electrospinning. To obtain a sufficiently low viscosity, a much higher temperature than the normal processing temperature was employed. In addition, the values of the applied voltage, the spinning distance, and the capillary inner diameter also have significant effects on melt electrospinning. The apparatus described here is eminently suitable for electrospinning of a wide range of materials, including those with a high-melt flow index of about 1200 g/10 min which are currently widely employed in commercial spinning, and these results will be summarized in a future publication.

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