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Electrospinning of Thermoplastic Polyurethane Microfibers and Nanofibers from Polymer Solution and Melt

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ABSTRACT: Electrospinning technique was used to produce ultrafine fibers from thermoplastic polyurethane (TPU). A direct comparison between melt and solution electrospinning of TPU was provided for the evaluation of process–structure relationship. It was found that the deposition rate of melt electrospinning (0.6 g h^{-1}) is four times higher than that of solution electrospinning (0.125 g h^{-1}) for TPU under the same processing condition. However, the average fiber diameters of solution electrospun TPUs (220–280 nm) were much lower than those of melt electrospun TPUs (4–8 μm). The effect of processing variables including collection distance and electric field strength on the electrospun fiber diameter and morphology was also studied. The findings indicate that increasing the electric field strength yielded more electrical forces acting on polymer jet and resulted in a decrease in fiber diameter as a result of more fiber drawing in both solution and melt electrospun fibers. It was also demonstrated that increasing the collection distance led to an improvement in the solidification of melt electrospun fibers and thus less fused fibers were obtained. Finally, a close investigation of fiber structures revealed that melt electrospun TPU fibers had smooth surface, whereas solution electrospun TPU fibers showed high intensity of cracks on the fiber surface. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

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INTRODUCTION

Polyurethane (PU) is composed of soft and hard segments connected by a urethane linkage. The soft segments impart flexibility, whereas the hard segments provide the rigidity and strength.[1] Thermoplastic polyurethane (TPU) typically includes 60–85% soft segments and shows desirable properties for particularly medical applications. These properties include elasticity, compatibility with tissue and blood, thrombo resistance, controlled degradation,[1] excellent hydrolytic stability, and resistivity to microorganisms and abrasion.[2,3] TPUs are also considered to be a good candidate for ultra-light protective clothing and synthetic biomaterials such as artificial vascular grafts.[3] However, such applications require high surface area to volume ratio and small pore sizes that could be obtained using submicron-to-nanofibers.

Electrospining is a robust and one of the most common techniques to produce fine fibers in the submicron-to-nanometer diameter range. Electrospinning technique[4–10] makes use of electrostatic forces instead of mechanical forces applied in conventional melt spinning technique to draw fibers. It involves applying an electric field between a polymeric solution or molten polymer and a collector. This applied electric field generates electrostatic forces at the surface of polymeric material. With the help of these forces, charged polymeric material can overcome the surface tension and able to travel from the feeding unit to the collector. When the polymer solution is used, solvent gradually evaporates and fiber forms during this movement. At the same time, fiber diameter is gradually decreased as a result of fast movement. When the polymer melt is used, attenuation and cooling take place simultaneously instead of solvent evaporation. Finally, continuous fibers are collected typically in the form of a nonwoven web.

Some of the advantages of electrospun fibers are their large surface area to volume or weight ratio and ability to provide small pore sizes in web structures. Application areas include but not limited to textile, filtration, electrical, optical, agricultural, composite, health care, biomedical, membrane technology, aerospace, defense, and security.[8,11] Therefore, electrospinning technique has gained great interest both from academia and from industry. Many of the studies on electrospinning have mainly focused on

understanding the process–property relationship and determining optimum conditions to obtain uniform fine fibers with desired morphology and functionality from various polymers.[12]

Demir et al.[13] conducted the first systematic study on the electrospinning behavior of segmented polyurethaneurea copolymer in solution. They investigated the effects of processing condition (electrical field) and solution properties (concentration, viscosity, and temperature) on the process and properties of electrospun fibers. They found that increasing the voltage resulted in higher flow rate with a relationship of flow rate $\sim$ (voltage)^3. Also, multiple jets were formed instead of one at higher electrical field and the number of jets formed linearly increased with increasing the electrical field. Their effort to reduce viscosity and lower surface tension by adding salt into solution yielded higher flow rates but did not reduce the average fiber diameter obtained. It was also demonstrated that fiber diameter is a strong function of solution concentration. It increased with increasing solution concentration and a power law relationhip was suggested for this particular electrospinning solution: fiber diameter $\sim$ (solution concentration)^3.

Demir et al.[13] also suggested that 3.8 and 12.8 wt % are the lower and upper concentration boundaries for the formation of polyurethaneurea fibers, respectively. At upper concentration value of 12.8 wt %, a wide range of fiber diameter distribution with three dominant population levels around 0.4, 1, and 1.4 μm were obtained. A close investigation on the morphology of these fibers revealed that fibers were not uniform instead they have wavy and curly structures. When the concentration was lowered to 5.2 wt %, the formation of beads was observed. It was suggested that increasing the distance or decreasing the electrical field could lower the occurrence of the beads on fibers. Similarly, raising the temperature of the solution resulted in uniform fiber structure in terms of diameter and morphology. Also, it led to an increase in the deposition rate.

Zhuo et al.[14] studied the effect of processing parameters such as applied voltage and feeding rate and solution concentration on the morphology and diameters of the electrospun PU fibers. They found that these parameters determine the final fiber structure. They were able to produce nanofibers in the diameter range of 50–700 nm. To obtain nanofibers without beads and curls solution concentration range of 5.0–7.0 wt%, PU in dimethyl formamide (DMF) was suggested along with processing parameters of 10–15 kV applied voltages and 0.06–0.08 mm/min feeding rates.

Pedicini and Farris[2] produced an electrospun fiber mat from TPU and DMF solution and determined mechanical properties of the mat. They found that electrospun TPU fiber mat showed a different stress–strain characteristic than a bulk TPU material. At same strain levels (60–300%), the electrospun TPU resulted in higher tensile stresses. This behavior was attributed to two factors: (i) strain-induced orientation of electrospun fiber and (ii) molecular orientation within the fibers that was induced by electrospining process.

Kampeerapappun[1] produced and examined a multilayer transdermal electrospun wound dressings made of PU. It was shown that electrospun PU dressing offers good barrier properties and oxygen permeability as well as less dressing change requirement. An antimicrobial dressing was also developed using single-step electrospinning approach and PU as the polymer.[1] This dressing exhibited very good uniformity and % extraction of antimicrobial agent. Kang et al.[3] produced and examined an artificial blood vessel made from electrospun TPU. They showed that electrospun TPU is thermally stable in the body temperature range and could perform as nondegradable blood vessels in the body system. Their findings also suggest that electrospun TPU-based artificial vessel yielded higher tensile properties than existing vessels made of polyester and expanded tetrafluoroethylene. Also, Chen et al.[15] explored the tissue engineering applications of TPU electrospun with collagen in the form of sheath/core bicomponent structure in which TPU was used as the core component. It was concluded that this material showed the characteristic of native extracellular matrix and could be used in tissue engineering applications and functional biomaterials.

All studies mentioned above used solution electrospinning technique to produce fine PU or TPU fibers. An alternative to solution electrospinning, melt electrospinning offers cheaper, more environmentally (solvent free), and safer process and products.[16] However, there are much less studies[16–23] on melt electrospinning compared to solution electrospinning in the literature.

In 1980s, Larrondo and Manley[17–19] conducted initial studies about melt electrospinning. They studied the effects of electric field potential and temperature on the diameter of electrospun polyethylene (PE) and polypropylene (PP) and found that the diameter of electrospun fiber decreases with increasing electric field strength and/or temperature of the molten polymer.[17] The fiber diameters obtained were $>$50 μm which is almost one to two magnitudes higher than those obtained using solution electrospinning. This was attributed to the difficulty to run melt electrospinning process owing to the high viscosity of the polymer melt.

Lyons et al.[16,20] studied the effects of tacticity and molecular weight of PP on the morphology and fiber diameter in melt electrospinning. It was shown that those two parameters have great influence in determining the fiber diameter. It was suggested using isotactic PP instead of atactic PP as the former resulted in smaller fiber diameter owing to the ability to form more oriented structure. The diameter of the fiber also decreased with decreasing molecular weight of the polymer. This was owing to the fact that lower molecular weight polymers have a lower viscosity. The average fiber diameters obtained at considerably large electric field strength values of 10–15 kV cm^{-1} ranged from 3 to 50 μm with high standard deviation values (up to 50%).

Dalton et al.[21] employed the melt electrospinning technique to develop tissue constructs from a blend of poly(ethylene oxide)-*block*-poly(ε-caprolactone) (PEO-b-PCL) with PCL for tissue engineering. Some processing parameters including spinneret diameter, flow rate, and collection distance were configured to obtain the highest quality fibers. Among these parameters, the flow rate was found to be the most critical parameter that influences the morphology and diameter of the electrospun fibers. In other study of Dalton et al.,[22] viscosity-reducing additives are used to decrease fiber diameters. For PP fibers, a considerable

Table I. Properties of TPU[24]

Properties	Hardness, (shore)	Specific gravity (g cm^{-3})	Tensile strength (MPa)	Tear strength, (N mm^{-1})	Elongation at break (%)
ASTM standard	D-2240	D-792	D-412	D-624	D-142
TPU (Elastollan®)	76 A	1.14	30	80	740

reduction from 35 to 0.8 μm was obtained. However, they also observed fusion on collected PP fibers owing to the insufficient cooling.

The effect of spinning temperature on diameter and morphology of polylactic acid (PLA) fibers was investigated by Zhou et al.[23] It was found that high spinning temperatures lead to a reduction in fiber diameter because of a strong whipping motion obtained at lower viscosity levels. It was also demonstrated that melt electrospun PLA fiber mats show an enhancement on the filtration efficiency and provide more environmentally friendly product.

Although melt electrospinning offers a solvent-free approach that is critical for the formation of biomaterials from TPU, we did not find any other study conducted for TPU in the limited melt electrospinning literature. Therefore, in this study, we focus on the melt electrospinning technique to produce fine TPU fibers. We also compare melt and solution electrospinning processes and products in terms of productivity and final fiber properties. In this regard, we examine the effect of processing parameters including electric field strength and collection distance on the morphology and diameter of electrospun TPU from solution and molten states.

EXPERIMENTAL

Materials

TPUs with the trade name of Elastollan® in granule form were obtained from Renko Textile (Istanbul, Turkey). Some typical properties of this polymer are summarized in Table I. DMF, the product of Merck Chemicals (Darmstadt, Germany), was used as a solvent for TPU. The concentration of TPU in the polymer solutions was ranged from 5 to 10 wt %. These solutions were prepared at the same days of electrospinning.

Electrospinning Setups and Parameters

Electrospinning system needs three main components to realize the process. They are high-voltage power supply, collector, and feeding unit. Gamma ES 100 type DC power supply which is able to apply voltage up to 100 kV was used in all experiments. For the collector, a circular-shaped galvanized thin metal plate coated with aluminum foil was chosen. The conditions for electrospinning were recorded as 33°C and 30% relative humidity (RH).

Irregular and discontinuous polymer flow has been a problem for the researchers in the solution electrospinning system. Some of them used micropumps to regulate the polymer flow. But, they are expensive. In the present study, this issue was solved in a simple manner by using a medical serum set. A medical serum set not only provides a continuous flow but also enables to adjust flow rate. The flow rate was adjusted to 0.05 mL min^{-1} manually by means of the flow adjuster. Other experimental parameters used for solution electrospinning were the electric field strength (2–4 kV cm^{-1}) and collection distance (6, 8, 10, and 15 cm).

In the melt electrospinning setup, the melt flow index tester (ATSFAAR, Segrate, Italy) was used as the feeding and heating unit because of its ability to melt thermoplastic polymers at desired temperature. It has two resistance sets and one PID digital thermo regulator for thermal control. Its working temperature is between 50 and 400°C with a sensitivity of 0.2°C. A spinneret with a 1 mm orifice diameter and a protector cabin were also designed and used to the melt electrospinning setup. The processing temperature which was the temperature measured from the spinneret tip ranged between 160 and 225°C. Other processing parameters were the electric field strength (4–6 kV cm^{-1}) and collection distance (6, 8, 10, and 15 cm).

Characterization and Measurements

The JEOL JSM-6335F, a cold cathode field emission scanning electron microscope, was used to characterize electrospun fiber diameters and morphology. Electrospun fiber diameters were measured using SEM images. Totally, 60 diameter measurements were carried out from each SEM image. The weights of the collected electrospun fibers were also measured with subtracting the initial weight from the final weight of the collector before and after the process using a sensitive weigh balance.

OBSERVATIONS

Critical Voltage and Temperature

The electrospinning process was initiated with a preset voltage value. Although the process was taking place, the voltage was gradually increased to determine the critical voltage value (V_c). In solution electrospinning experiments, V_c was found to be 5 kV at 5 wt % solution concentration and 6 cm collection distance. It was observed that single jet was formed at this voltage value and the number of jets increased with increasing the voltage. Multiple jet formation at relatively high voltage values was also reported in the literature.[13,25,26] It is owing to the fact that the increase in the charge per unit area of the initial jet causes instability and splitting of the first jet to reduce charge on the jet.

In melt electrospinning experiments, temperature values (the measured temperature at the spinneret exit) ranging from 160 to 225°C were tested. We observed that melt electrospinning did not occur until 200°C. Even at 200°C, we sometimes encountered that process was interrupted because of the insufficient viscosity of polymer melt. Above 210°C, in some instances the molten polymers did not solidify enough when it reached to collector. Therefore, the processing temperatures for the following experiments were kept between 205 and 208°C in which the molten TPU became viscous enough to be electrospun

without any process outage or solidification issue. V_c for melt electrospinning was then recorded as 30 kV at 6 cm collection distance and 208°C processing temperature. Contrary to solution electrospinning, only single jet was formed at all voltage values studied. We also observed some electrical discharge above 50 kV applied voltage values at or above 10 cm collection distance.

Effect of Solution Concentration on Fiber Formation

Of the solutions containing TPU at percentages of 5, 7.5, and 10 wt %, fibers could only be produced from the solution of 5 wt % (Figure 1). At concentrations of 7.5 and 10 wt %, we observed the formation of droplets at the tip of the needle which were neither stretched enough nor carried by the jet continuously. Instead, these droplets were landed on the collector by means of the weight gained by the droplets over the time and electrospraying effect rather than electrospinning. Similar droplet formation was also reported for PEO/water solutions with high concentrations by Deitzel et al.[25] It has been explained to be the limitation of electrospinning process at higher viscosities that can prohibit the continuous flow from the tip of the needle to the collector.

Deposition of Electrospun Fibers

The collection of electrospun fibers was also investigated. In solution electrospinning process, electrospun fibers were dispersed homogeneously onto the collector. It was observed that electrospun fiber carrying jets could move in the course of time to deposit fibers from a certain part of the collector to another part of the collector as shown in Figure 2(a). It is owing to the fact that the first deposition part of the collector was isolated in the course of time and therefore the jet preferred the more conductive path or better grounding location and changed its path. On the contrary of solution electrospinning process, electrospun fibers were collected by accumulating one on top of the other in a way to form a hill-like structure in a very restricted area shown in Figure 2(b). This can be attributed to the limitation on the whipping of the jet owing to the high viscosity, fast solidification, and large mass or diameter. Therefore, the jet was not capable of changing its path in the course of time and restricted in a very narrow collection area.

RESULTS AND DISCUSSION

Comparison of Solution and Melt Electrospinning Deposition Rates

In the literature, there is a general belief that the production rate of the melt electrospinning is greater than that of the solution electrospinning.[23] However, there is not much scientific or empirical support for this implication. Therefore, in this section, we aimed to provide a direct comparison between deposition rates which were the amount of electrospun fibers deposited on a collector at certain time interval for both solution and melt electrospinning processes.

To compare the deposition rates, we kept the collection distance and time constant at 10 cm and 4 min, respectively. However, we varied the applied voltage values to determine the effect of electric field on the deposition rate. Figure 3 shows the deposition rate results for solution and melt electrospinning processes.

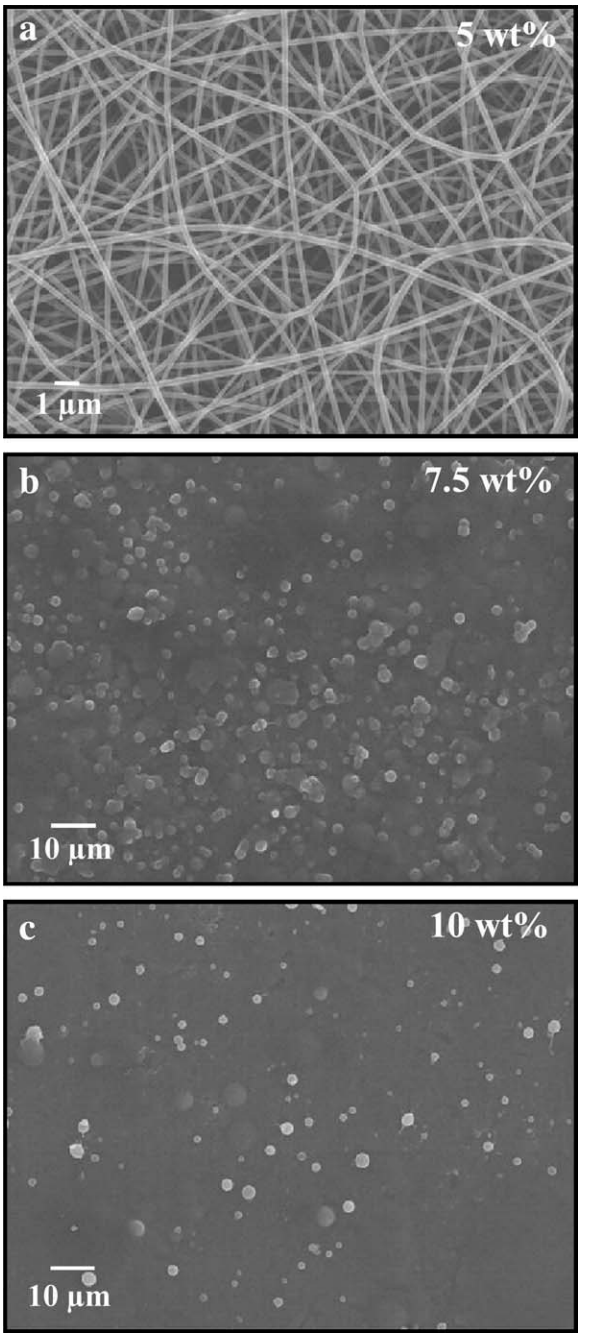

Figure 1. Effect of TPU solution concentration on fiber formation: (a) 5 wt %, (b) 7.5 wt %, and (c) 10 wt % (at 4 kV cm^{-1} electric field and 10 cm collection distance).

At 4 kV cm^{-1} electric field strength, the solution electrospinning yielded around 0.125 g h^{-1} deposition rate, whereas the melt electrospinning provided approximately 0.6 g h^{-1} deposition rate for TPU. This suggests that melt electrospinning can produce considerably more amount of fibers than solution electrospinning in a constant time interval. This can be attributed to the lack of solvent evaporation during the melt electrospinning which results in more fibrous material onto the collector without any mass loss from the starting material. When the effect of electric field on deposition rate was

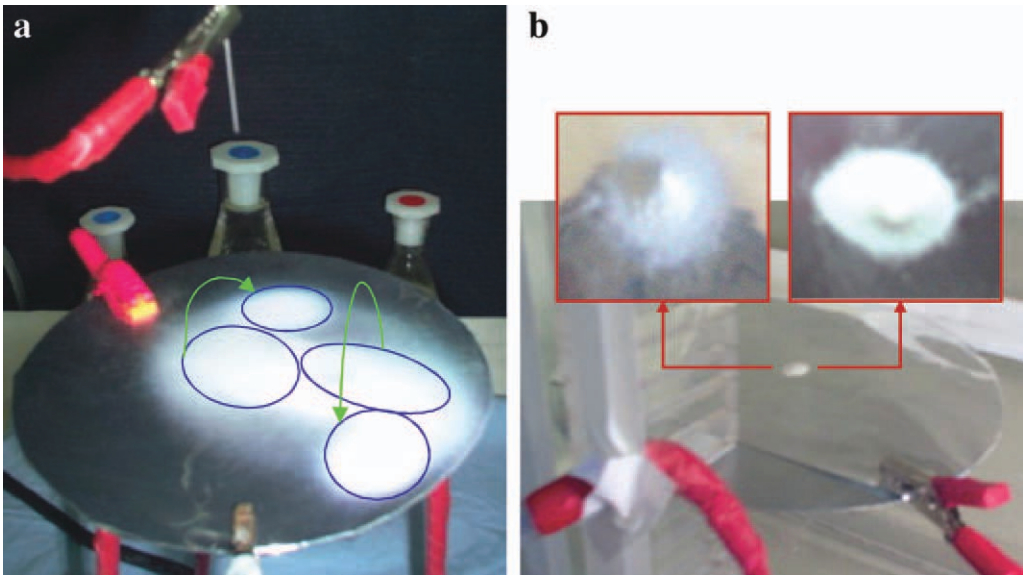

Figure 2. Fiber collection during (a) solution and (b) melt electrospinning processes. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

investigated, it was found that the deposition rate increased with increasing the electric field strength in both processes. This is mainly owing to the fact that more polymer solution or melt mass could be carried at higher electrical field as the electrical force acting on the jet is larger. This increase in the mass flow rate as a result of electric field strength increase was also reported in the literature.[25]

Effect of Processing Variables on Electrospun Fiber Diameter

Collection distance and electric field strength were chosen as the processing parameters for solution and melt electrospinning processes. Although the collection distance values were kept same in both processes, the electric field values used were different because of the necessity of higher electric field strength to carry out the melt electrospinning process. Thus, the electric field values tested ranged from 2 to 4 kV cm^{-1} for solution electrospinning and 4–6 kV cm^{-1} for melt electrospinning experiments. Under these processing conditions, electrospun fiber diameters for both processes were measured and discussed.

The results obtained for solution electrospun TPU fibers at varying collection distances (6, 8, 10, and 15 cm) and constant electric field value of 4 kV cm^{-1} are shown in Figure 4. The average fiber diameters obtained for different collection distances ranged between 220 and 240 nm and did not differ much with varying the distance. These results suggest that the electrospun fiber diameter is not dependent on the collection distance tested for solution electrospun TPU if electric field strength is kept constant. On the other hand, when the effect of electric field on the final fiber diameter was investigated (Figure 5), it appeared to be a tendency for a reduction in the fiber diameter with increase in the electric field strength. Fiber stretching during electrospinning is mainly caused by electrostatic forces (f_e) acting on the polymer jet which was defined as[5]

$$f_e = \frac{e(t)V}{h(t)} \quad (1)$$

where e is the charge on the jet, V is the applied voltage, and h is the distance from the tip of the jet to collector. According to this model [eq. (1)], the electrostatic forces acting on the polymer jet increase with increasing the electric field (V/h). Thus, more stretching could be achieved at higher electric field strength and this could result in a decrease on fiber diameter.

The effect of the collection distance on the fiber diameter in melt electrospinning of TPU was investigated at constant applied voltage (40 kV). The results are also shown in Figure 4. The smallest average fiber diameter of 4.01 μm (σ = 1.13 μm) was obtained at 6 cm collection distance, whereas the largest average fiber diameter of 8.21 μm (σ = 2.34 μm) was produced at 10 cm collection distance. This suggests that under constant applied voltage increasing the collection distance yields larger fiber diameter. It can also be associated with the change in electrostatic forces acting on polymer melt. That is to say, increasing the distance and maintaining the applied voltage could result in a decrease in electrostatic forces and therefore less fiber stretching was achieved.

Figure 6 shows the effect electric field on melt electrospun fiber diameter. Some data points are missing because of the challenges of collecting reliable samples or restrictions of applying voltages out of the range of 30–50 kV in melt electrospinning process. The general trend observed here was a decrease in average fiber diameter with increasing the electric field strength. This result is also in agreement with Reneker's model [eq. (1)] in which more electrostatic forces could act on molten polymer jet at higher electric field strength and thus, more drawing occur. Similar results were also reported for other melt electrospun polymers such as PE,[17] PP,[17,20] and PEO-b-PCL with PCL.[21]

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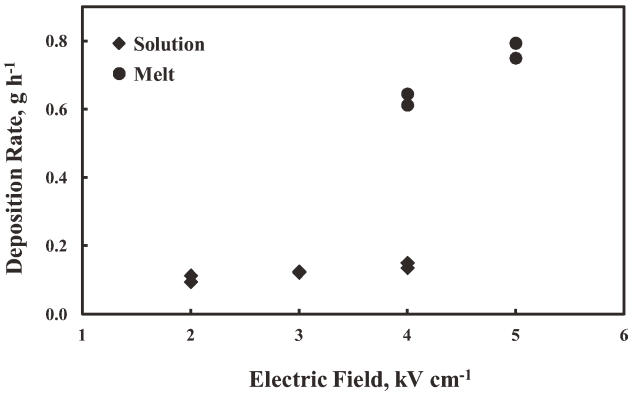

Figure 3. Effect of electric field on deposition rate (at 10 cm collection distance and 4 min collection time).

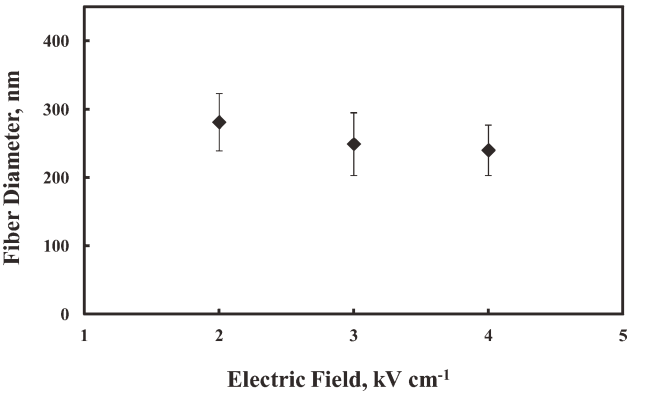

Figure 5. Effect of electric field on solution electrospun fiber diameter (at 8 cm collection distance).

Effect of Processing Variables on Electrospun Fiber Mat Morphology

When the SEM pictures of the solution electrospun TPU fibers were examined (Figure 7), it was observed that the electrospun fibers tended to stick each other with an increase in the electric field strength. For instance, at 4 kV cm^{-1} electric field strength the electrospun fibers formed a nonwoven web structure in which majority of the fibers created bonding spots with the nearest adjacent fibers [Figure 7(c)]. This can be attributed to insufficient solvent evaporation as a result of electric field strength increase which could result in higher throughputs (Figure 3) and less time for the solvent to evaporate. Thus, fibers were not dry enough when they reached to collector and formed bonds with other fibers. On the other hand, when 2 kV cm^{-1} electric field was used, collected electrospun fibers were more independent and formed fewer joints with other fibers [Figure 7(a)].

For melt electrospun TPU fibers, collection distance showed a great influence on the morphology of the collected fiber mat. In this regard, Figure 8 shows the results obtained for different collection distances under the same applied voltage value of 40 kV. It is clearly shown that increasing the collection distance resulted in more independent fibers and fewer joints between these fibers [Figure 8(c)]. This is owing to the fact that better cooling or solidifying effect could be achieved at the largest distance of 10 cm. It is also related to the achieved processing speed which was supposed to be the lowest at 10 cm and highest at 6 cm under the same applied voltage. Thus, the fibers electrospun at 10 cm had also longer time to cool and solidify, whereas the fibers electrospun at 6 cm had shorter time and were still slightly molten at collection. The increase in the quality of the melt electrospun fibers as a result of lowering the flow rates was explained by Dalton et al.[21] and could also be correlated with our findings. However, increasing the collection distance is a trade of in terms of final fiber diameter. Thus the lowest average fiber diameter was achieved when the lowest collection distance was used (Figure 4).

SEM images of enlarged fiber segments show that the melt electrospun fiber surface is smooth without cracks or other physical defects (Figure 8). However, solution electrospun TPU fibers showed high intensity of cracks (Figure 7). The reason for the formation of these cracks might be explained with the hypothesis suggested by Gentsch et al.[27] According to this approach, evaporation of the DMF from the fiber during and after electrospinning process may show a difference from the core to the skin of the fiber. The cracks on the fiber surface might form if the evaporation of this solvent is faster on the surface than in

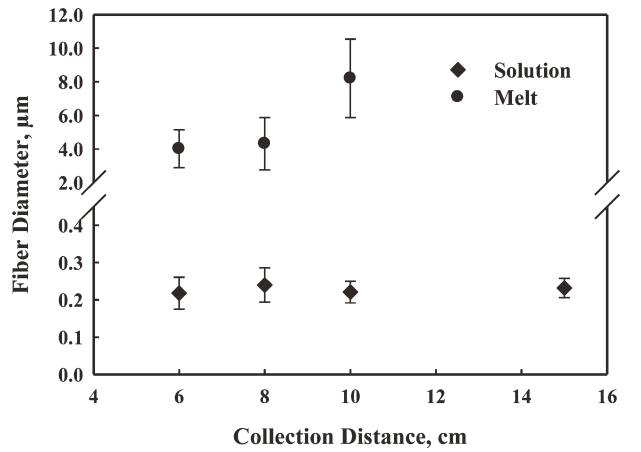

Figure 4. Effect of collection distance on electrospun fiber diameter.

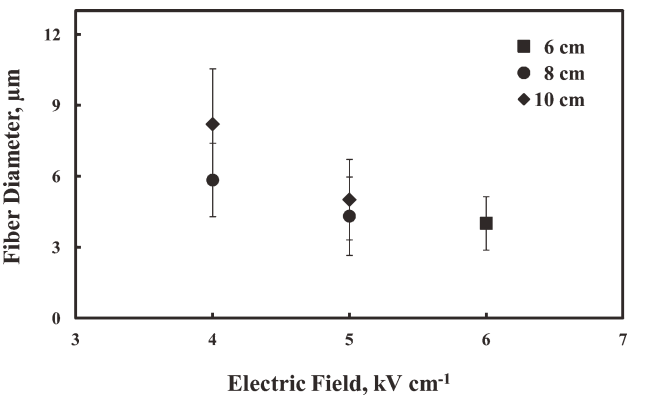

Figure 6. Effect of electric field on melt electrospun fiber diameter.

the fiber core. A tension against to fiber stretching could be generated on the fiber surface if the surface dries faster than the core. Also, environmental conditions for electrospinning might alter the rate of evaporation of the solvent which could trigger asymmetric drying on the fiber. For instance, increasing ambient temperature[28,29] or decreasing humidity[29,30] can increase the solvent evaporation rate. In our experiments, the recorded conditions were 33°C and 30% RH which could also facilitate the fast solvent evaporation compared to other electrospinning processes carried out at standard conditions.

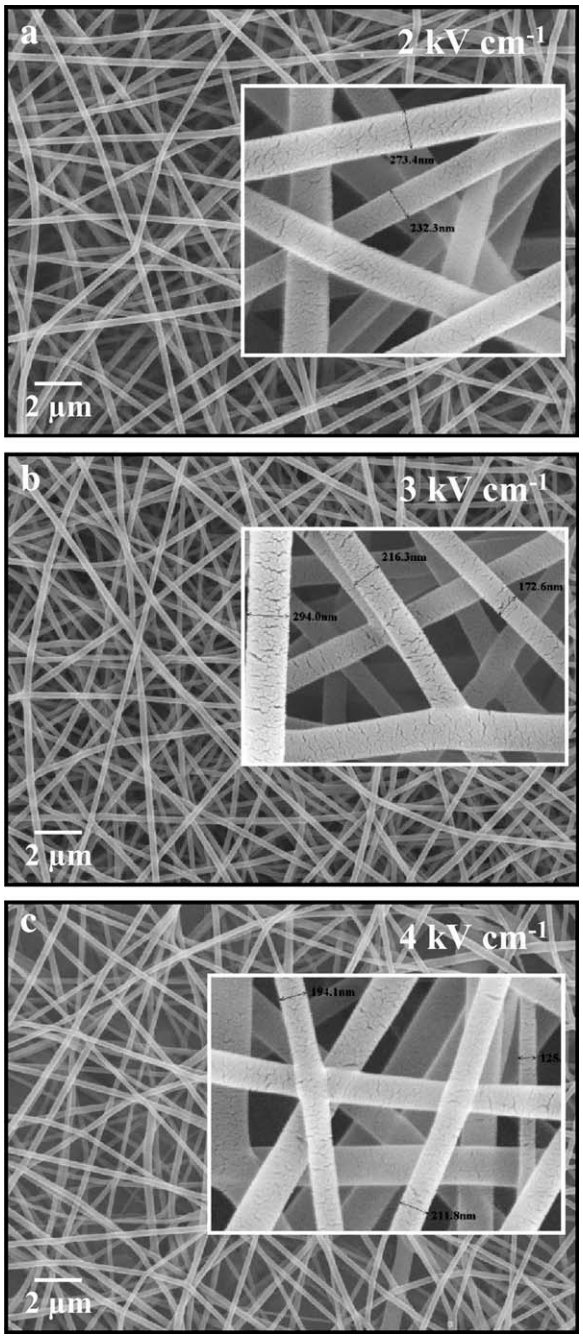

Figure 7. Effect of electric field on solution electrospun fiber morphology (at 8 cm collection distance): (a) 2 kV cm^{-1}, (b) 3 kV cm^{-1}, and (c) 4 kV cm^{-1}.

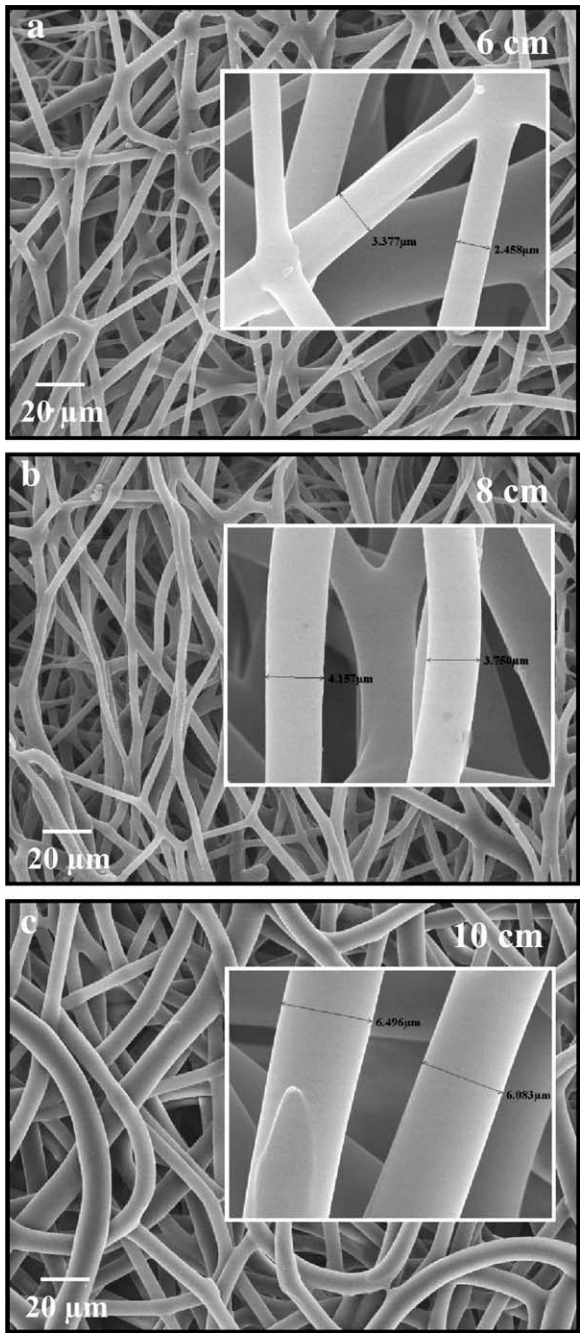

Figure 8. Effect of collection distance on melt electrospun fiber morphology (at 40 kV applied voltage): (a) 6 cm, (b) 8 cm, and (c) 10 cm.

SUMMARY AND CONCLUSIONS

Electrospinning of TPU from solution and melt states was studied to form fine fibers in the micron to nanometer diameter range. The solution and melt electrospinning processes were initially compared to determine critical processing variables and productivity levels. Our findings revealed that the deposition rate of melt electrospinning (0.6 g h^{-1}) is four times higher than that of solution electrospinning (0.125 g h^{-1}) under the same processing conditions. However, the average fiber diameters obtained for the solution electrospun TPU ranged between

220 and 280 nm, whereas they were in the range between 4 and 8 μm for the melt electrospun TPU. This can be related to the higher viscosity levels encountered in electrospinning of molten TPU compared to solution state. The effects of processing variables including collection distance and electric field strength on the final fiber diameter were then determined for both states. It was shown that varying the collection distance did not change the final fiber diameter significantly for solution electrospun TPU when the electric filed strength was kept constant. However, increasing the electric field strength resulted in a decrease in fiber diameter for both solution and melt electrospun TPU. In the electrospinning of TPU solution, the electric field strength was found to be the determining factor on electrospun fiber mat morphology. It was shown that increasing the electric field yielded more nonwoven web-like structure in which fibers were bonded at the connection points as a result of insufficient solvent evaporation because of higher throughput. For melt electrospun TPU fiber mats, increasing the collection distance resulted in more independent fibers and fewer joints between the fibers (less-fused fibers). This was attributed to the longer cooling or solidifying time and path achieved at the highest collection distance of 10 cm. On the other hand, increasing the collection distance and maintaining the same applied voltage caused an increase in the final fiber diameter.

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