Communication

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Pilot-scale production of polylactic acid nanofibers by melt electrospinning

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Abstract: Melt electrospinning has been used to manufacture fibers with diameters in the low micrometer range, but the production of submicrometer fibers has proven more challenging. In this study, we investigated the feasibility of fabricating polylactic acid nanofibers using polymer grades with the increasing melt flow rates (15–85 g/10 min at 210°C) by melt electrospinning with a 600-nozzle pilot-scale device featuring an integrated climate control system realized as a glass chamber around the spinneret. Previous experiments using this device without appropriate climate control produced fibers exceeding 1 µm in diameter because the drawing of fibers was inhibited by the rapid cooling of the polymer melt. The integrated glass chamber created a temperature gradient exceeding the glass transition temperature of the polymer, which enhanced the drawing of fibers below the spinneret. An average fiber diameter of 810 nm was achieved using Ingeo Biopolymer 6252, and the finest individual fiber (420 nm in diameter) was produced at a spin pump speed of 5 rpm and a spinneret set temperature of 230°C. We have therefore demonstrated the innovative performance of our pilot-scale melt-electrospinning device, which bridges the gap between laboratory-scale and pilot-scale manufacturing and achieves fiber diameters comparable to those produced by conventional solution electrospinning.

Keywords: fiber spinning, nanotechnology, upscaling, nonwoven, process development

Abbreviations

GPC	gel permeation chromatography
M_w	relative molecular weight
M_n	number average molar mass
MFI	melt flow index
PLA	polylactic acid
SEM	scanning electron microscopy

1 Introduction

Electrospinning is the most common method for the production of polymer fibers with diameters in the low micrometer to nanometer range (1,2). There are two main variants of the method (1,3). In solution electrospinning, the polymer is dissolved in an organic solvent that evaporates as the fiber is spun. In melt electrospinning, the polymer is heated to above its melting point and solidifies as the fiber is spun. In both cases, a strong electric field is applied during spinning to stretch the polymer, typically resulting in fibers that are several hundred nanometers in diameter after drawing (4). Solution electrospinning is the simplest of the two processes because it does not require high temperatures or coagulation chemistry, but a solvent recovery step must be included, and there is a risk that toxic solvents may be carried over into the final product (3,5). There are no such risks in melt electrospinning, and the entire process is therefore more environmentally sustainable (6). However, due to the high viscosity and low electrical conductivity of the polymer melts, the resulting fibers have a slightly larger average diameter than those produced by solution electrospinning (3,7).

Currently, industry favors solution electrospinning not only because it produces finer fibers but also because the method is more scalable than melt electrospinning, and fiber manufacturing processes are therefore more economical despite the need for solvent recovery (3,6,8,9). To address the limited scalability of melt electrospinning, recent innovations such as multiple-needle and needleless device configurations have demonstrated a roadmap to overcome low flow rates (typically in

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the $\mu\text{g/h}$ range) and thus increase the productivity (3,10). Prototypes with umbellate nozzles containing 60 spinnerets can achieve maximum product deposition rates of $\sim 36\text{ g/h}$ (11,12). A polymer melt differential electrospinning system with a linear slot spinneret achieved an output of 75.6 g/h , which was improved further by increasing the slot width (8). Until recently, the largest multineedle configuration was a device with 64 nozzles (13,14), but in our previous report, we described a prototype with 600 nozzles that provides the basis for the pilot-scale melt electrospinning (15).

Although our prototype addresses the limited scalability of melt electrospinning, the resulting fibers are still coarser than those produced by solution electrospinning. Using a commercial spinning-grade polylactic acid (PLA) resin (Ingeo Biopolymer 6201D) supplemented with 6% (w/w) sodium stearate to increase the electrical polymer conductivity, we were able to produce fibers ranging from 1.00 to $7.00\text{ }\mu\text{m}$ in diameter, which are the finest biobased fibers manufactured thus far using our pilot-scale melt electrospinning device (15). So far achieved fiber diameters in the PLA solution electrospinning are in the range of 0.14 to $2.1\text{ }\mu\text{m}$ (16–20) and that in the melt electrospinning are 0.2 to $50\text{ }\mu\text{m}$ (6,12,21–28). In individual cases, fiber diameters in the subnanometer range have already been achieved using solution electrospinning (29). However, it should be taken into account that mainly small-scale devices are used, and especially in both melt electrospinning and solution electrospinning, additional device modifications such as an integrated airflow are used to achieve fine fiber diameters and a higher process productivity (22,30).

The fibers using our pilot-scale device were produced without a climate control system although climate control is common for other device configurations (31–33). Climate control prevents the rapid cooling of the fibers, so that diameters in the nanometer range can be achieved by drawing before the polymer solidifies. Therefore, we integrated a climate control system into our pilot-scale melt electrospinning device by surrounding the spinneret with a glass chamber. We investigated the influence of climate control on the temperature distribution below the spinneret, on the melt viscosity of three different PLA grades with increasing melt flow indices (MFIs), and ultimately on the diameter of the resulting fibers at different spin pump speeds. We were able to optimize our pilot-scale device to produce fibers with a diameter below $1\text{ }\mu\text{m}$ for the first time, thus offering an industrial solution for the preparation of melt-electrospun nanofibers.

2 Experimental

2.1 Materials

Melt electrospinning was carried out using three granular, thermoplastic PLA fiber-grade resins: Ingeo Biopolymer 6201D, 6260D and 6252D (NatureWorks LLC, Minnetonka, Minnesota, USA). The materials are characterized by different MFIs and crystalline melt temperatures. The standard fiber-spinning grade (Ingeo Biopolymer 6201D) has a MFI of $15\text{--}30\text{ g/10 min}$ at 210°C and a crystalline melt temperature of $155\text{--}170^\circ\text{C}$. The first melt-blown grade (Ingeo Biopolymer 6260D) has a MFI of 65 g/10 min at 210°C and a crystalline melt temperature of $165\text{--}185^\circ\text{C}$. The second melt-blown grade (Ingeo Biopolymer 6252D) has a MFI of $70\text{--}85\text{ g/10 min}$ at 210°C and a crystalline melt temperature of $155\text{--}170^\circ\text{C}$. All materials were vacuum dried at 60°C for 12 h before processing.

2.2 Melt-electrospinning equipment

We used our prototype pilot-scale melt electrospinning device including a spinneret with 600 nozzles, each 0.3 mm in diameter and spaced at 8 mm intervals. We integrated a glass chamber around the spinneret to achieve climate control. A schematic illustration (a) and an image (b) of the device including the glass chamber is shown in Figure 1. A constant supply of polymer melt was ensured by a speed-adjustable single-screw extruder with three heating zones based on integrated heating elements and a speed-controlled spinning pump. Spinning was carried out at an adjusted nozzle temperature of 230°C without the glass chamber and at 220°C and 230°C with the integrated glass chamber using spin pump speeds of 10, 5 and 2 rpm. Preliminary experiments have shown that at $220\text{--}230^\circ\text{C}$, a stable fiber formation is ensured over all 600 nozzles and no droplet formation occurs. The statistical relevance of the independent and in this study investigated factors spin pump speed and temperature on the fiber diameter was previously proven with a two-way analysis of variance (34). An aluminum collector with an uneven surface was used instead of a conventional plate collector. The adjustable collector pins can be placed 8 mm apart, and we used a narrow and offset pin order with a diagonal distance of 2.6 cm . With the nozzle/collector pairing installed, nonwovens up to 340 mm in width could be produced on a continuous basis. The distance

Figure 1: Schematic illustration (a) and picture (b) of our pilot-scale melt electrospinning device including the glass chamber.

between the collector and the nozzle plate was maintained at 11 cm to allow comparison with our previous results. A positive voltage of 60 kV was applied to the collector with simultaneous grounding of the spinneret.

2.3 Polymer characterization

The rheological properties of the materials, focusing on the frequency-dependent complex viscosity G^* , were characterized using a Discovery HR1 hybrid rheometer (TA Instruments, New Castle, USA). We carried out two frequency sweeps, the first from 628 to 10 rad/s and the second from 100 to 0.1 rad/s. For all experiments, we used a 25-mm plate-to-plate geometry. The gap distance was set to 1,000 μm , and the strain amplitude was maintained at 1%. Measurements were taken at environmental temperatures of 200, 230 and 250°C. To improve the comparability of the results, the viscosity of different PLA grades is presented at the same frequency of 10 rad/s.

Gel permeation chromatography (GPC), using a 1260 Infinity GPC/SEC System (Agilent Technologies, Santa Clara, USA) with hexafluor-2-isopropanol containing 0.19% sodium trifluoroacetate as the mobile phase flowing at a rate of 0.33 ml/min, was carried out to determine the relative molecular weight (M_w) and the number average molar mass (M_n). The polymer sample (5 mg) was dissolved in hexafluor-2-isopropanol for 2 h before injection into a medium column with 7 μm particle size (Polymer Standards Service, Mainz, Germany). The experiment was calibrated against a standard polymethyl methacrylate polymer (1.0×10^5 g/mol).

2.4 Fiber characterization

Fiber diameters were measured using a DM4000M reflected light microscope (Leica Microsystems, Wetzlar, Germany) and by scanning electron microscopy (SEM) using a LEO 1450VP instrument (Carl Zeiss, Oberkochen, Germany). The

samples were attached to a stub with double-sided carbon tape and sputtered with gold prior to SEM to produce a conductive surface. In each case, the fiber diameter was measured 100 times at different sections in the nonwoven sample.

3 Results and discussion

3.1 Effects on viscosity

The complex viscosity curves of Ingeo Biopolymer 6201D, 6260D and 6252D are compared in Figure 2, corresponding to a frequency of 10 rad/s and set temperatures of 200, 230 and 250°C. There was little difference in viscosity between the two melt-blow grades (Ingeo Biopolymer 6260D and 6252D), but the viscosity of the standard spinning-grade PLA (Ingeo Biopolymer 6201D) was approximately three-fold higher at all set temperatures.

Table 1 presents the relative molecular weight (M_w) and the number average molar mass (M_n) of Ingeo Biopolymer 6201, 6260 and 6252. The decrease in molecular weight correlates with the increasing MFI values of the individual grades given by the polymer manufacturer.

3.2 Temperature distribution

At a set spinneret temperature of 230°C, the average surface temperature of the spinneret was 112°C after 2 h

Figure 2: Complex viscosity of Ingeo Biopolymer 6201, 6260 and 6252 determined at a frequency of 10 rad/s at set temperatures of 200, 230 and 250°C.

Table 1: Relative molecular weight (M_w) and number average molar mass (M_n) of Ingeo Biopolymer 6201, 6260 and 6252

Material	M_w (g/mol)	M_n (g/mol)
Ingeo Biopolymer 6201	1,47,700	86,170
Ingeo Biopolymer 6260	1,17,500	72,540
Ingeo Biopolymer 6252	1,03,400	66,730

of operation and 117°C after 4 h of operation without the glass chamber, as determined using an infrared thermometer. The polymer inlet was one sided, so temperature differences of up to 30°C were detected over the spinneret surface (31). Table 2 presents the temperature profiles at spinning temperatures of 220 and 230°C using the glass chamber. The heat accumulation within the chamber caused the surface temperature of the spinneret to increase by 30°C at a set temperature of 220°C and by 40°C at 230°C. As the distance to the spinneret increased, the temperature slowly declined until the temperature at the edge of the glass chamber reached 38 and 49°C at set temperatures of 220 and 230°C, respectively. This means that both temperature profiles fell below the glass transition temperature of the three PLA grades we used (55–60°C), thus avoiding the sticking and deformation of the fibers during deposition on the conveyor belt. Furthermore, the temperature remained above the glass transition temperature up to a distance of 4 cm from the spinneret, which allows further stretching of the fibers.

3.3 Fiber diameter

Next we investigated the processability of the three PLA grades and the influence of the temperature profile

Table 2: Temperature profiles at spinning temperatures of 220 and 230°C with the glass chamber installed

Distance from spinneret (cm)	Temperature below spinneret (°C) at set spinneret temperatures of	
	220°C	230°C
0	144	158
2	89	103
3	74	88
4	62	73
5	55	62
6	47	56
Outer edge	38	49

Table 3: Average fiber diameters ($\pm$ standard deviations) produced at 230°C without the glass chamber (GC) and at 220 and 230°C with the glass chamber installed, at spin pump speeds of 10, 5 and 2 rpm

Material	Fiber diameter (μm) at spin pump speeds of		
	10 rpm	5 rpm	2 rpm
Ingeo Biopolymer 6201 (230°C, without GC)	15.12 ± 2.68	11.29 ± 1.15	8.29 ± 1.49
Ingeo Biopolymer 6201 (220°C, with GC)	25.20 ± 5.92	5.66 ± 1.42	1.75 ± 0.54
Ingeo Biopolymer 6201 (230°C, with GC)	24.31 ± 5.71	3.06 ± 1.15	1.04 ± 0.15
Ingeo Biopolymer 6260 (230°C, without GC)	13.67 ± 3.16	11.03 ± 1.50	7.74 ± 1.26
Ingeo Biopolymer 6260 (220°C, with GC)	22.88 ± 5.27	2.57 ± 0.86	1.78 ± 0.50
Ingeo Biopolymer 6260 (230°C, with GC)	5.21 ± 1.23	1.60 ± 0.40	1.12 ± 0.43
Ingeo Biopolymer 6252 (230°C, without GC)	13.99 ± 2.30	11.72 ± 1.12	7.25 ± 1.16
Ingeo Biopolymer 6252 (220°C, with GC)	13.70 ± 4.52	2.10 ± 0.76	2.58 ± 0.94
Ingeo Biopolymer 6252 (230°C, with GC)	5.14 ± 1.16	0.81 ± 0.24	1.13 ± 0.40

induced by installing the glass chamber on the fiber diameter. Table 3 presents the average fiber diameters ($\pm$ standard deviations) produced at a set spinneret temperature of 230°C without the glass chamber and at 220 and 230°C with the glass chamber, at three different spin pump speeds. Comparing the diameter of fibers produced from Ingeo Biopolymer 6201 (Figure 3), Ingeo Biopolymer 6260 (Figure 4) and Ingeo Biopolymer 6252 (Figure 5) revealed no significant difference between the PLA grades at a set spinneret temperature of 230°C without the glass chamber, but there was a 50% reduction as the pump speed was reduced from 10 rpm ($\sim 15\mu\text{m}$) to 2 rpm ($\sim 7\mu\text{m}$). Figure 6 shows the microscopic images of the melt-electrospun fibers at a set temperature of 230°C without the glass chamber at a spin pump speed of 10 and 2 rpm: Ingeo Biopolymer 6201 (a) at 10 rpm and (b) at 2 rpm; Ingeo Biopolymer 6260 (c) at 10 rpm and (d) at 2 rpm; and Ingeo

Figure 4: Diameters of Ingeo Biopolymer 6260 fibers produced at 230°C without the glass chamber (GC), at 220 and 230°C with the glass chamber installed, and at spin pump speeds of 10, 5 and 2 rpm.

Figure 3: Diameters of Ingeo Biopolymer 6201 fibers produced at 230°C without the glass chamber (GC), at 220 and 230°C with the glass chamber installed, and at spin pump speeds of 10, 5 and 2 rpm.

Figure 5: Diameters of Ingeo Biopolymer 6252 fibers produced at 230°C without the glass chamber (GC), at 220 and 230°C with the glass chamber installed, and at spin pump speeds of 10, 5 and 2 rpm.

Figure 6: Microscopic images of the melt-electrospun fibers at a set temperature of 230°C without the glass chamber at a spin pump speed of 10 and 2 rpm: Ingeo Biopolymer 6201 (a) at 10 rpm and (b) at 2 rpm; Ingeo Biopolymer 6260 (c) at 10 rpm and (d) at 2 rpm; and Ingeo Biopolymer 6252 (e) at 10 rpm and (f) at 2 rpm.

Biopolymer 6252 at (e) 10 rpm and (f) at 2 rpm. Finer fibers were anticipated at lower spin pump speeds and melt flow rates, as observed during previous studies (3,32,33). Figure 7 shows the nonwoven of Ingeo Biopolymer 6260 melt-electrospun without the glass chamber at a set temperature of 230°C and a spin pump speed of (a) 10 rpm and (b) 2 rpm. A droplet formation could not be observed with or without the glass chamber. The nonwovens were deposited on a blue cellulose substrate. At lower spin pump speeds, the PLA fibers are attracted more strongly by the collector pins at which the electric field lines emerge, resulting in slight accumulations that leave a visible structure in the PLA nonwoven. To overcome this phenomenon and improve

the overlap, the distance between the pins can be reduced in future studies. The differing viscosities of the three materials appeared to have no effect. Contrary to expectations, the diameter of Ingeo Biopolymer 6201 fibers increased significantly at a pump speed of 10 rpm and at set temperatures of 220 and 230°C following the integration of the glass chamber, possibly because the higher melt flow rate resulting from the lower viscosity does not give sufficient time for drawing. The same phenomenon was observed for Ingeo Biopolymer 6260 at a set temperature of 220°C. However, the diameter of Ingeo Biopolymer 6252 fibers at 220°C remained almost the same with or without the glass chamber. Nevertheless, the diameter of Ingeo Biopolymer 6260 and 6252

Figure 7: Nonwoven of Ingeo Biopolymer 6260 melt-electrospun without the glass chamber at a set temperature of 230°C and a spin pump speed of (a) 10 rpm and (b) 2 rpm.

fibers could be reduced by more than 50% at a spinning temperature of 230°C and a spin pump speed of 10 rpm by installing the glass chamber. Otherwise, the fiber diameter declined with slower spin pump speeds, and the lowest average fiber diameter of 1.04 μm was achieved for Ingeo Biopolymer 6201 at a spinning temperature of 230°C with the glass chamber installed and a spin pump speed of 2 rpm. For the first time, individual fibers with diameters of less than 1 μm were observed, improving our previous results for PLA supplemented with 6% (w/w) sodium stearate, which generated individual fibers with a minimum diameter of 1.23 μm (15).

For all three PLA grades, we achieved the lowest fiber diameters at a spinning temperature of 230°C with the glass chamber installed. The diameter of Ingeo Biopolymer 6260 fibers could be reduced by 86% at a spin pump speed of 2 rpm compared to spinning in the absence of the glass chamber. In this case, we also observed individual submicrometer fibers. The minimum average fiber diameter was 0.81 μm , which was achieved using Ingeo Biopolymer 6252, a spinneret temperature of 230°C, a spin pump speed of 5 rpm, and the glass chamber installed (Figure 8). The finest individual fiber

was 0.42 μm in diameter. Therefore, we were able to achieve the production of nanofibers as fine as any previously achieved by solution or melt electrospinning on a device, which comes closer to industrial-scale melt electrospinning than any described in the literature. The scalability of our process therefore brings industrial melt electrospinning one step closer. The installment of the glass chamber did not appear to affect the electric field during spinning, although this should be examined more closely in future experiments.

4 Conclusion

We have previously reported the use of our prototype melt electrospinning device for the production of biobased thermoplastic submicrofibers and nanofibers. The finest PLA fibers we were able to produce were $\sim 1 \mu\text{m}$ in diameter, but this was achieved without comprehensive optimization of the apparatus, the process parameters, or the polymer substrate. Now we have integrated a climate control system realized as a glass

Figure 8: SEM image of (a) Ingeo Biopolymer 6201, (b) Ingeo Biopolymer 6260 and (c) Ingeo Biopolymer 6252 fibers at a set temperature of 230°C and a spin pump speed of 5 rpm using the glass chamber.

chamber, which generated a temperature gradient to ensure efficient heating and therefore prolonged the drawing of the fibers. We tested three different PLA grades and produced individual fibers with diameters in the nanometer range when the glass chamber was integrated, although the average fiber diameters remained predominantly above the threshold of 1 μm . Nevertheless, an average fiber diameter of 0.81 μm was achieved with Ingeo Biopolymer 6252, and the finest individual fiber (0.42 μm in diameter) was produced from this polymer at a spin pump speed of 5 rpm and a spinneret set temperature of 230°C. In contrast to other systems, our device does not require (hot) compressed air or additional heating of the glass chamber. Furthermore, no additives, as usual in melt electrospinning, were used, which enables easier approval for pharmaceutical and biomedical products. We have therefore demonstrated the innovative performance of our pilot-scale melt electrospinning device, which bridges the gap between laboratory-scale and pilot-scale manufacturing and achieves fiber diameters comparable to those produced by conventional solution electrospinning.

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Availability of data and material: The datasets used and/or analyzed during this study are available from the corresponding author on reasonable request.

Conflicts of interest: The authors declare that they have no competing interests.

Authors' contributions: KK performed the spinning trials, material analysis and validation, and wrote the manuscript. FL and GS supervised the study and helped to design the device. All authors read and approved the final manuscript.

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