

# Development of Melt Blown Electrospinning Apparatus of Isotactic Polypropylene

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## ABSTRACT

Melt blown electrospinning is a method of combining melt blown with melt electrospinning. In this study, we developed the multi nozzle melt blown electrospinning machine. The significant point of this method is to use air blowing force as well as electrostatic force at the same time. Also, this machine has many processing parameters, such as applied voltage, spinning distance, temperature at polymer reservoir, temperature at nozzle, air flow rate at nozzle, nozzle diameter, nozzle length, flow rate of melt polymer, molecular weight of polymer, and additives, etc. We have successfully prepared nonwoven isotactic polypropylene fibers by controlling these parameters.

**Keywords:** melt blown electrospinning, polypropylene, fibers, air flow, nozzle

## 1 INTRODUCTION

In recent years, electrospinning is becoming of great interest because not only it can produce polymer fibers with diameters in the range of nano- to a few micrometers using polymer solutions or melts, but it also has the advantages of being simple, conventional methods with which a wide range of porous structures can be produced, and inexpensive as compared with conventional methods<sup>1-5</sup>. In many studies, this method has attracted considerable attention since setup is inexpensive and simple. In our previous study, we have firstly prepared a syndiotactic polypropylene (sPP) fibrous membrane from the solution via solution electrospinning at slightly elevated temperature [6]. SEM images and the frequency distribution of sPP fiber diameters of electrospun sPP fibers are shown in Figure 1. However, organic solvents used for electrospinning has serious issues which are mostly related to solvent recycling and environmental safety. Moreover, the mixed solvent system used for electrospinning of sPP is difficult for mass production. Among many studies, to figure out these problems, the use of molten polymers to produce electrospun fibers via melt-electrospinning becomes a subject of great interest. In spite of the potential benefits of melt-electrospinning, little progress has been made in the past twenty years. Fully understanding of the melt-electrospinning process, and its potential to replace solution electrospinning, has not yet been realized [8]. Meanwhile

melt spinning and melt blowing are the most commonly used processes for producing the nonwoven fibers. Melt blown process is known because it has higher productivity than melt spinning process. In general, melt blown commercial products are composed of fibers with average diameters exceeding 1-2  $\mu\text{m}$  [9].

In this study, we developed multi nozzle melt blown electrospinning machine. The significant point in this machine is able to use coincidentally two kinds of force, for instance, air blowing force and electrostatic force. We therefore strongly anticipate that air blowing force increases production rate and electrostatic force produces thin diameter of fibers, respectively. Here, we attempt to study the effects of air-flow rate, additive, and nozzle system on fiber diameter of the iPP nanofibers prepared by recently developed multi nozzle melt blown electrospinning apparatus.

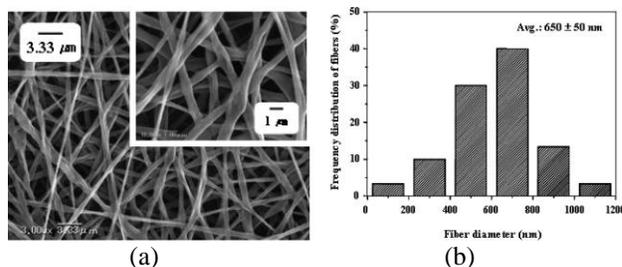


Figure 1: (a) SEM image and (b) frequency distribution of electrospun sPP fibrous membrane from solutions at a slight elevated temperature (35  $^{\circ}\text{C}$ )

## 2 EXPERIMENTAL

### 2.1 Materials

The iPP (isotactic polypropylene, 1200 melt flow rate (MFR) at 230 $^{\circ}\text{C}$ ) was kindly provided from Kuraray CO., LTD. (Japan). Irgatec CR 76 was obtained from Ciba Japan K.K. (Japan). LiCl was purchased from Wako (Japan). Two samples were used in this study. Sample A is prepared by mixing iPP and Irgatec CR 76 (200g/6g). Sample B is prepared by mixing iPP, Irgatec CR 76, and LiCl (200g/6g/2g).

### 2.2 Melt Blown Electrospinning

Conditions	Levels
Air flow rate at nozzle (l/min)	30, 60, 110, 130
Applied voltage (kV)	15, 28, 33
Tip to collector distance (cm)	3.0, 8.5
Temperature at polymer reservoir (°C)	260
Air temperature at nozzle (°C)	450

Table 1: Melt blown electrospinning conditions of iPP electrospun fibers.

This apparatus has many parameters: applied voltage (kV), spinning distance (cm), temperature at polymer reservoir (°C), air temperature at nozzle (°C), air flow rate at nozzle (l/min), nozzle diameter (gauge), nozzle length (mm), flow rate of melt polymer (ml/min). In addition, polymer characteristics such as molecule weight, melt index, and melt point notably can affect the spinnability and the resulting fiber diameter. In this work, we investigated only two important parameters. Firstly, we checked the effect of air flow rate at nozzle on fiber diameter. Two different conditions of air flow rate (at 60 l/min and 130 l/min) were tested, respectively, whereas other parameters were fixed. A high-voltage power supply was used to generate a potential difference of 28 kV between the needle (with a 19 gauge, 15 mm) and a grounded metallic rotating drum placed at the distance of 8.5 cm from the tip of the needle. Polymer was melt at 260°C, and the temperature at nozzle was controlled to be 350°C by blowing of hot air. Secondly, we investigated the effect of LiCl on fiber diameter. Polypropylene has a poor electrical conductivity. Generally, this property may not be appropriate for electrospinning. LiCl can help the enhancement of electrical conductivity of PP polymer and thereby improvement of electrospinning conditions [10].

### 2.3 Characterization

The morphology of the melt blown electrospun fibers was examined with scanning electron microscopy (SEM, VE-8800, Keyence Co., Japan) and also using a Digital microscope (VHX-900, Keyence.co.Japan)

To compare the crystal structure of melt blown electrospun fibers versus melt spun fibers were determined using a X-ray Diffraction Spectroscopy. (XRD, Dmax2500, Rigaku Ltd., Japan) operating at 50kV and 200mA using Cu- K $\alpha$  radiation. (wave length 1.5405nm) The diffracting intensities were recorded every 1 ° from 2 $\theta$  scans in range 5 ° ~ 30 °.

## 3 RESULTS AND DISCUSSION

### 3.1 Effect of Air Flow Rate At Nozzle on Fiber Diameter

Melt blown electrospun iPP nonwoven fibers shown in Figure 2. As shown in this figure, we could successfully

produce the melt blown electrospun iPP fiber mats (Figure 2), showing a good spinnability. We also investigated the effect of air flow rate at nozzle on fiber diameter. Figure 2 shows SEM images of melt blown electrospun fibers prepared at different air flow rates (Figure 2b: 60 l/min and 2c: 130 l/min, respectively). Table 2 summarizes averaged fiber diameter of each sample at different air flow rate, respectively. From SEM analysis, we found that the higher air flow rate at nozzle is, the thinner fiber diameter is formed. The diameter of the obtained fibers was found to be about 31.3  $\mu$ m. However, we couldn't observe narrower ultrafine fiber on the nano-scale.

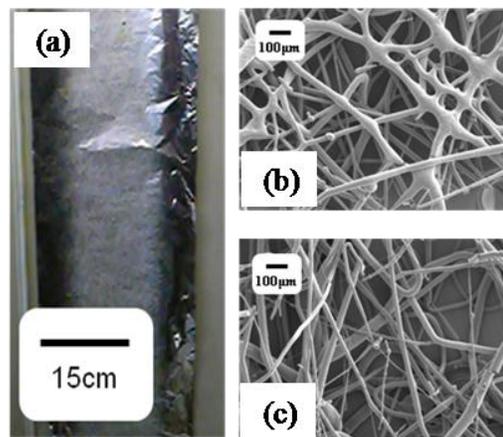


Figure 2: Melt blown electrospun iPP nonwoven fibers. (a) is Digital photograph, (b) and (c) are SEM images. Air flow rate at nozzle: (b) 60 l/min, (c) 130 l/min.

Sample	(b)	(c)
Average fiber diameter	31.3 $\pm$ 12	25.0 $\pm$ 10
Air flow rate at nozzle (l/min)	60	130
Applied voltage (kV)	28	28
Tip-to-collector distance (cm)	8.5	8.5

Table 2: The average diameters of melt blown iPP electrospun fibers produced from different air flow rate at nozzle.

### 3.2 Effect of LiCl on Fiber Diameter

As expected, the addition of small amounts of salt (about 1 wt.% in solution) was found to increase the fiber diameter and thereby decrease the jet length. [10] Consequently, to produce nanoscaled fibers, we added an additive, such as LiCl, which will increase the conductivity of molten iPP and results in an improved spinnability and thinner fiber diameter. The digital microscope images of melt blown electrospun iPP nonwoven fiber mats of sample B (with 1% LiCl) is shown in Figure 3 (left). Figure 3 (right) shows SEM image of melt blown electrospun iPP nanofiber with the smallest diameter of about 260 nm.

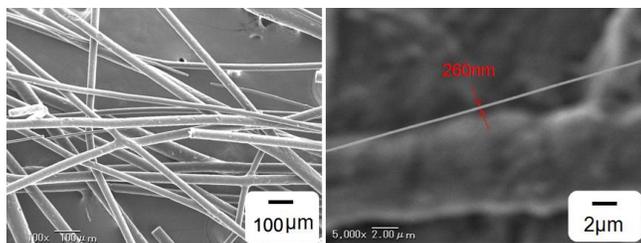


Figure 3: SEM images of melt blown electrospun iPP nonwoven fibers incorporating 1% LiCl.

### 3.3 XRD

The WAXD profiles of the melt blown electrospun iPP nonwoven fibers are shown in Figure 4. Sample A is melt electrospun iPP nonwoven fibers, sample B is melt blown iPP nonwoven fibers. It clearly shows the (110), (040), (130), and (131) crystal reflections at  $2\theta=14.0^\circ$ ,  $17.0^\circ$ ,  $18.5^\circ$  and  $22^\circ$ , indicative of form I with an antichiral helical conformation [7,11]. Specifically, as seen in Figure 4, the melt blown electrospun iPP nanofibers (Figure 4A) exhibited sharper peak than that of melt blown fibers (Figure 4B), suggesting the enhanced crystalline structures.

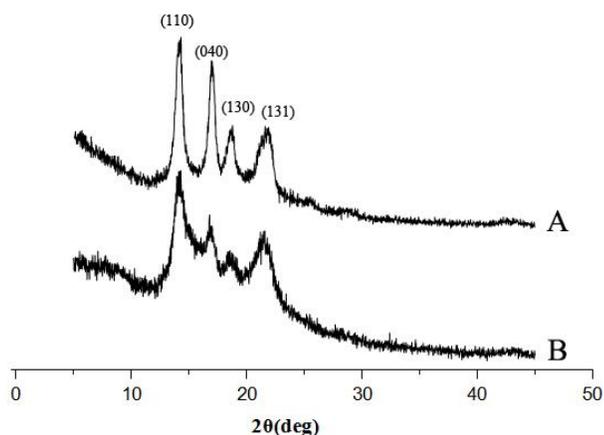


Figure 4: WAXD profiles of (A) melt electrospun iPP nonwoven fibers (B) Melt blown iPP nonwoven fibers.

### 3.4 Effects of Nozzle System on Fiber Diameter

Based on our previous results, melt blown electrospun iPP nonwoven fibers with the diameter of ca. 25 nm was successfully prepared by controlling the air flow rate and an addition of LiCl. But, we have faced on the difficulty in controlling the temperature of nozzle system, which will therefore give rise to poor spinnability and broader fiber diameter. Thus, we further improved the nozzle system to control the fine temperature during melt blown

electrospinning. Firstly, we changed the thickness of the walls of molten polymer tank in nozzle system for improving the thermal transfer. Secondly, we attached the heater on nozzle system for a stable heating environment. As a result, as seen in Figure 5, after changing the nozzle system, the temperature could be well-controlled, but the spinnability was not stable at higher air flow rate of 130 l/min.

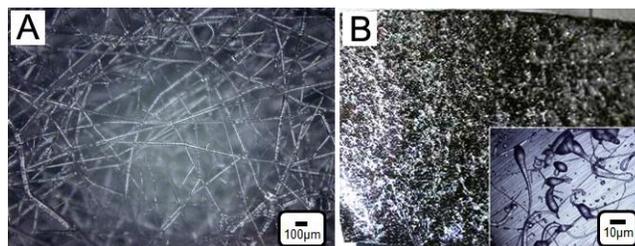


Figure 5: Digital microscope images of (A) previous nozzle system and (B) new nozzle system (air flow rate: 130l/min, TCD: 8.5cm, applied voltage: 28kV, air temperature at nozzle: 230 °C).

Thirdly, the number of tips in nozzle system was also changed from 6 metal tips to 3 metal tips. In principle, when the metal tips get closer, the electric filed between the metal tips will be interrupted each other during electrical charging. As a result, such interaction will interrupt a full whipping and extension of polymer chains. The results are shown in Figure 6, and indicate that the more stable spinnability was observed than that of the previous nozzle system with 6 metal tips. In addition, Figure 7 shows SEM images of the corresponding melt blown electrospun iPP nanofibers.

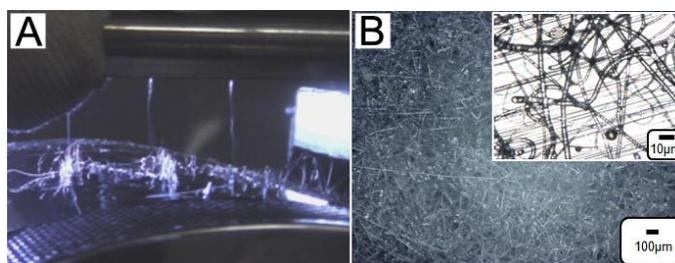


Figure 6: (A) Digital camera image of changed nozzle system and (B) digital microscope image of iPP melt blown electrospinning at the changed nozzle system. (air flow rate : 30l/min, TCD : 3.0cm, applied voltage: 15kV, Air temperature at nozzle: 230 °C)

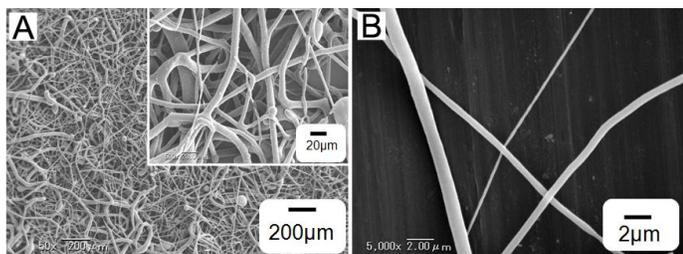


Figure 7: SEM images of iPP melt blown electrospinning at the changed nozzle system (air flow rate : 30l/min, TCD : 3.0cm, applied voltage: 15kV, Air temperature at nozzle: 230°C)

## 4 CONCLUSIONS

We developed the multi nozzle melt blown electrospinning machine. The crucial point of this method is to use both air blowing force and electrostatic force at the same time. Also iPP nonwoven fiber mat was successfully prepared by Melt blown electrospinning. We found that the higher air flow rate at nozzle is, the thinner fiber diameter is produced. Moreover, by adding LiCl into polypropylene, we could obtain the nanofiber with the smallest diameter (~260 nm) on melt blown electrospinning.

## 5 ACKNOWLEDGEMENTS

This work was supported by project for “Innovation Creative Center for Advanced Interdisciplinary Research Area” in Special Coordination Funds for Promoting Science and Technology from the Ministry of Education, Culture, Sports, Science and Technology of Japan. The authors acknowledge the support of Shinshu University Global COE Program “International Center of Excellence on Fiber Engineering”.

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