

A Study on the Shape Control of Multiple Layers for PES Nanofiber Using Electrospinning Method of Top-down Spinning

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Abstract: In this study, we investigated the diameter and pore control of PES nanofibers by focusing on the process parameters such as concentration of polymer solution, applied voltage of spin column, and injection rate of polymer solution among various electrospinning parameters. The device for electrospinning was designed by modularizing to be able to cope with changes in test conditions and large area.

The detailed device consists of a fabric feeding part, a spinning part, a soaking part, a drying part, and a fabric transfer part, the spinning device is based on top-down spinning and the spinning column is composed of 12 columns with 10 nozzles per column. The results obtained from this study are as follows. The maximum pore diameter is less than 22 kV at an applied voltage of 20 kV, the standard deviation of nanofiber diameter at each applied voltage means that the fibers radiated at 20 kV from 18 kV to 245.84 nm, 20 kV at 183.52, and 22 kV at 232 kV were more uniformly emitted, it has been proved that the nanofibers are radiated unevenly due to the effect of the strong applied voltage at 22 kV.

Key words: Electrospinning, PES(Poly-Ether-Sulfone), multiple layer, nano fiber, SEM(Scanning Electron Microscope).

1. Introduction

Recently, there has been a surge of fine dust emissions and alarms according to the influence of long-distance movement of dust from China due to the indiscriminate industrialization in domestic and abroad. In addition, respiratory and cardiovascular diseases due to long-term human exposure of fine dusts are rapidly and markedly increasing [1]-[4].

Air pollution is mostly caused by fine dusts, such as gas from factories and automobile. This is divided according to particle size, fine dust (PM10) with a diameter of 10 μm or less, fine dust (PM2.5) with a diameter of 2.5 μm or less. Meta-analysis of the recent 24 articles has shown that exposure to short-term air pollution affects chronic obstructive pulmonary disease and asthma exacerbations, resulting in asthma-COPD overlap syndrome and the risk of poisoning increased.

Therefore, in order to reduce exposure of human dust to (micro) dust, various researches have been actively carried out such as protection products, a fine dust reduction technique, and an air purification technique [5]-[8].

In this paper, we have derived the parameters to control the diameter and pore diameter of PES nano

fiber in the process of fabricating filter fabric through electrospinning of polyether sulfone (PES) material.

In order to evaluate the structural effect of the PES filter fabric according to the electrospinning conditions, the density of the polymer mixed solution, the applied voltage of the electrospinning column nozzle, the injection rate of the polymer mixed solution, and the feed rate of the fabric were varied.

The analysis of the filter structure influence on electrospinning condition was confirmed by measuring the diameter, orientation and density of the nanofiber filter.

2. Experiment of Condition for Electrospinning

2.1. Configuration of Electrospinning System

The device for electrospinning was designed by modularizing to be able to cope with changes in test conditions and large area. The detailed device consists of a fabric feeding part, a radiation part, a soaking part, a drying part, and a fabric transfer part, as shown in Figure 1. The radiation device is based on top-down radiation and the radiation column is composed of 12 columns with 10 nozzles per column.

To verify the effect of the electric field generated in the electrospinning, we made it individually controlled. Collector substrate applied (-) voltage is made of stainless steel and the electrospinning is performed by transferring the PES fabric to the upper surface of the substrate or stainless steel substrate.

Polyether sulfone (PES), which takes into account physical properties such as tensile strength, elongation, and heat resistance, was used for electrospinning. Dimethylformamide (DMF), N-methyl- Pyrrolidone (N-Methyl-2-pyrrolidinone, NMP) was used at a constant ratio.

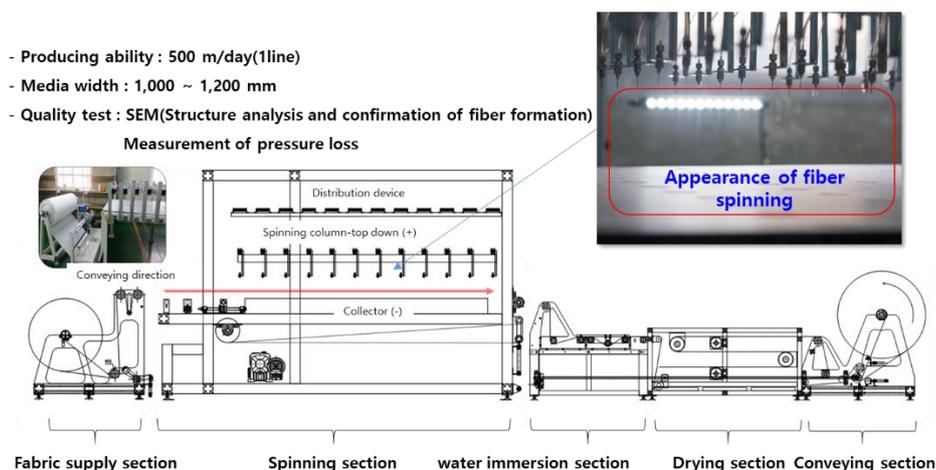


Fig. 1. Configuration of electrospinning system.

2.2. Experiment of Condition

In this study, we investigated the diameter and pore control of PES nanofibers by focusing on the process parameters such as concentration of polymer solution, applied voltage of spin column, and injection rate of polymer solution among various electrospinning parameters. Polyether sulfon (PES, BASF6020P) was used as a reagent for the test. Dimethylformamide (DMF), N-methyl-2-pyrrolidone (NMP) was used for organic solvent.

In producing of the polymer solvent, PES was added according to the polymer concentration (26, 28, 30 wt%) DMF and NMP were mixed with a certain amount of the organic solvent, and then S.T.P status in especially self-made impeller-type mixing reactor as shown in Fig. 2, and the mixture was stirred for 20 hours. For the analysis of the filter structure effect according to the electrospinning condition, a scanning electron microscope (SEM, Hitachi-S4700) was used to verify the diameter, the orientation degree and the

degree of integration of the nanofiber filter.

The diameter of the nanofibers was measured by analyzing 100 fibers at random using ImageJ an image analysis program, also to check the structural characteristics of the nanofiber filter pores, an automated capillary flow porometer (CFP-1200AEL) was used, the filtration efficiency and the pressure loss characteristics of the filter characteristics were measured through Fractional Efficiency Filter Tester-Neutrailzer type, TSI 3160



Fig. 2. Configuration of agitator and physical composition.

3. Results of Electrospinning Experiment

3.1. Analysis of Nanofiber Diameter by Concentration of PES

Electrospinning was carried out to the mixed polymer solution under the conditions shown in Table 1. Samples with different PES concentrations emitted in the same electrospinning process parameters were analyzed by scanning electron microscope for nanofiber diameter except for the fabric feed rate, the data measured and calculated by Capillary Flow Porometer and the data obtained by Automated Capillary Flow Porometer are shown on Table 2.

Table 1. Condition of Electrospinning for the Effect of PES Concentration

Concentration(wt%)	26, 28, 30
TCD(mm)	175
Applied voltage(kV)	20
Injection rate (cc/min)	0.5
Conveyingspeed(mm/min)	50, 100, 150

Table 2. Results of Capillary Flow Porometer

Concentration (wt%)	Fiber diameter (nm)	Capillary Flow Porometer		Filter characteristic		Quality Factor
		largest pore diameter (μm)	mean pore diameter (μm)	Differential pressure (mmAq)	Filter efficiency (%)	
26	449.60	2.89	1.79	11.95	95.88	0.27
28	626.26	4.58	2.42	8.99	93.57	0.31
30	643.48	6.28	3.11	4.88	60.16	0.19

The average pore diameter and the maximum pore diameter increased with the transfer rate of the fabric, as the result of spinning the 26 wt% polymer solution at the transfer rate of 50, 100, and 150, respectively. This is related to the density of the nanofiber web described above, and as the transfer speed of the fabric

increases, the density also decreases.

It was also confirmed that the polymer concentration increased from 26 wt% to 30 wt% at the same material transfer rate and the average pore size also increased. In addition, as a result of SEM photograph, shown in Fig. 3, as the concentration of the polymer increases from 26 wt% to 30 wt%, the fiber diameter tends to increase.

The average diameter was measured 449.60 nm at 26wt% of the feed rate of 50 mm / min, and the average diameter was 626.26 nm at 28wt% from the randomly sampling. The average diameter was gradually increased from 30wt% to 643.48 nm in Table 2 and Fig. 4.

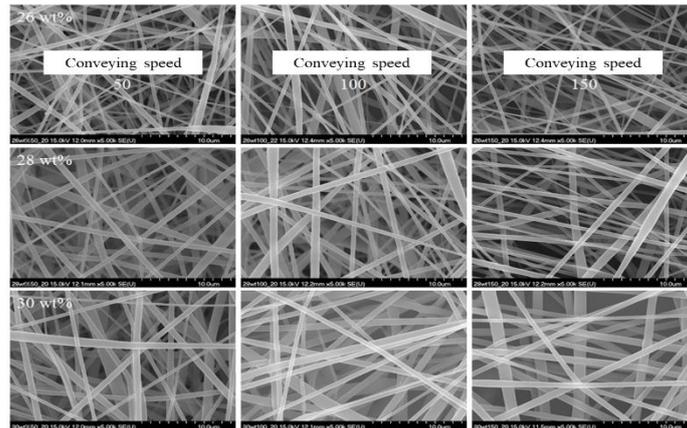


Fig. 3. Results of SEM image by concentration of PES.

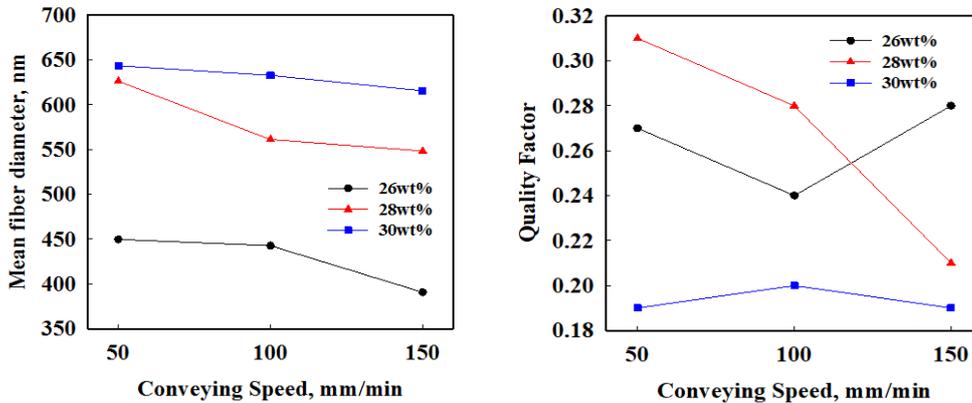


Fig. 4. Results of diameter change and quality factor by concentration of PES solution.

3.2. Analysis of Nanofiber Diameter by Applied Voltage

Table 3 shows the conditions of applied voltage for electrospinning. In Table 4 and Fig. 6, the average diameter of nanofibers tended to decrease linearly at 615.72 nm at 20 kV, 561.13 nm at 20 kV and 507.23 nm at 22 kV as the applied voltage increased from 18 to 22 kV.

Table 3. Condition of Electrospinning for an Applied Voltage

Applied voltage(kV)	18, 20, 22
TCD(mm)	175
Concentration(wt%)	28
Injection rate (cc/min)	0.5
Conveying speed(mm/min)	50, 100, 150

We can see the (SEM) photographs in Fig. 5. When the applied voltage is 18 kV, it can be confirmed that

the nanofibers are thick and thin, including beads that are elongated. The diameter deviation between the beads and each fiber gradually decreases as the applied voltage increases. The standard deviation of nanofiber diameter at each applied voltage is 245.84 at 18 kV, 183.52 at 20 kV, 232.43 at 22 kV, and 20.4 kV at 20 kV. It means more evenly emitted, it indicates that the nanofibers are unevenly radiated due to the effect of a strong applied voltage at 22 kV. Therefore, it is judged that the maximum pore diameter at 20 kV generally, uniformly radiated is smaller than the pore diameter at 22 kV.

Table 4. Results of Capillary Flow Porometer

Applied voltage (kV)	Fiber diameter (nm)	Capillary Flow Porometer		Filter characteristic		Quality Factor
		largest pore diameter (μm)	mean pore diameter (μm)	Differential pressure (mmAq)	Filter efficiency (%)	
18	615.52	18.33	3.33	2.83	55.30	0.28
20	561.13	7.29	3.20	3.31	59.78	0.28
22	507.23	9.01	2.48	2.74	50.26	0.25

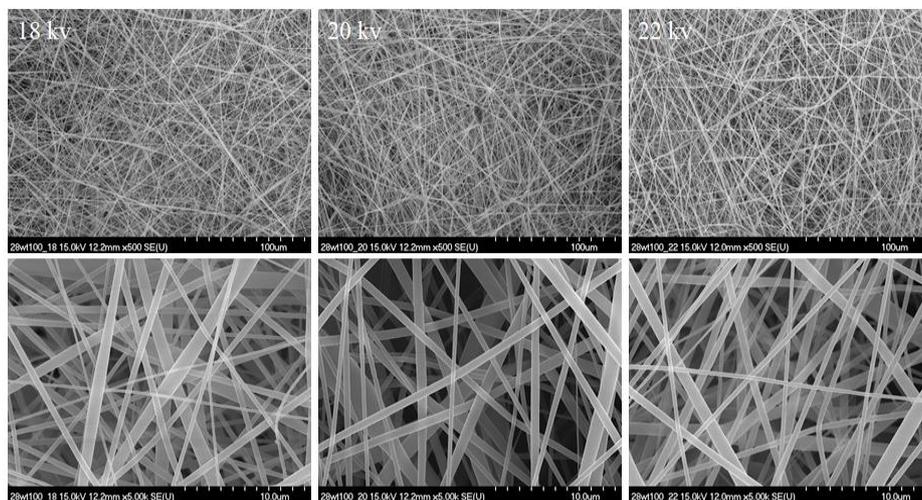


Fig. 5. Results of SEM image by applied voltage.

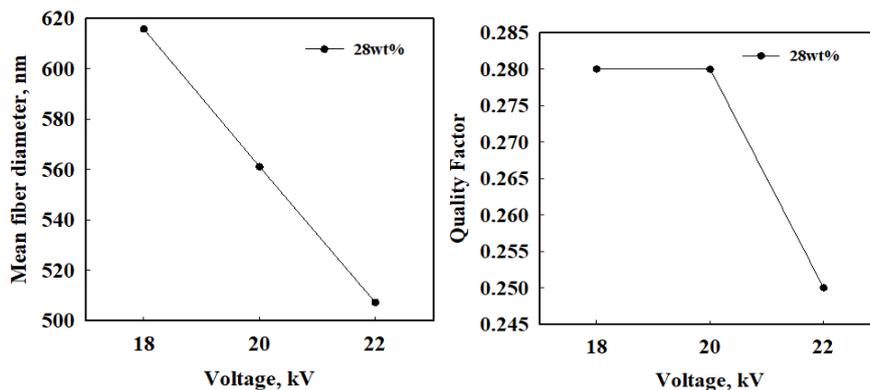


Fig. 6. Relation of diameter change and quality factor by applied voltage.

3.3. Analysis of Nanofiber Diameter by Injection rate

Table 5 shows the conditions of applied injection rate for electrospinning. SEM Analyzed in Fig. 7, the nanofibers were wholly reduced in diameters at feed rates of 50, 100 and 150 when the discharge rate per hour decreased from 0.5 cc to 0.3 cc.

This is due to the thinning of the fibers as the fiber diameter decreases, it is considered that the fibers are dense to the PES fibers, which are the substrate in Fig. 8, and the large pores of the fabric are not sufficiently

covered. As table 6 shows, The average pore size was increased from 3.20 μm to 4.26 μm by 1.06 μm when the amount of polymer solution was decreased from 0.5 cc / hr to 0.3 cc / hr at the fabric transfer rate '100 mm / min.,' the maximum pore diameter increases from 7.29 μm to 12.58 μm . As the pore diameter increases, the differential pressure decreases as compared with 0.5 cc / hr, and the overall filter filtration efficiency also decreases. In the case of Quality Factor, when the feed rate of the polymer solution was 50 and 100, the injection rate of 0.3 cc / hr was lower than that of 0.5 cc / hr, which is the same as the analyzed in a tendency. As shown in Fig. 9~ Fig. 10, it is believed that this is due to the fact that it is dense to the PES fiber which is the substrate and cannot sufficiently cover the large pores of the fabric.

Table 5. Condition of Electrospinning for an Injection Rate

Injection rate(cc/min)	0.3, 0.5
TCD(mm)	175
Concentration(wt%)	28
Applied voltage(kV)	20
Conveying speed(mm/min)	50,100, 150

Table 6. Results of Capillary Flow Porometer

Injection rate (cc/min)	Fiber diameter (nm)	Capillary Flow Porometer		Filter characteristic		Quality Factor
		largest pore diameter (μm)	mean pore diameter (μm)	Differential pressure (mmAq)	Filter efficiency (%)	
0.3	435.89	7.64	3.18	2.95	46.68	0.21
0.5	613.29	4.58	2.42	8.99	93.57	0.31

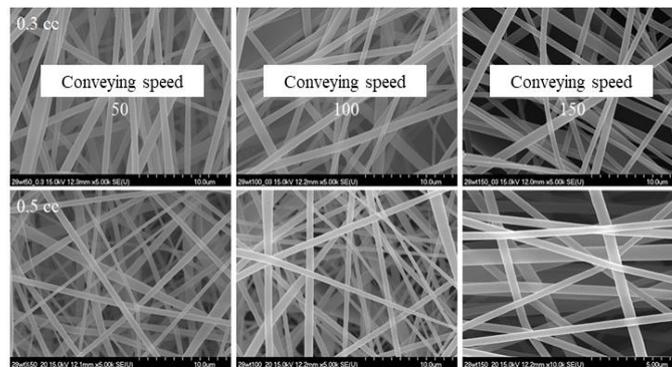


Fig. 7. Results of SEM image by injection rate.

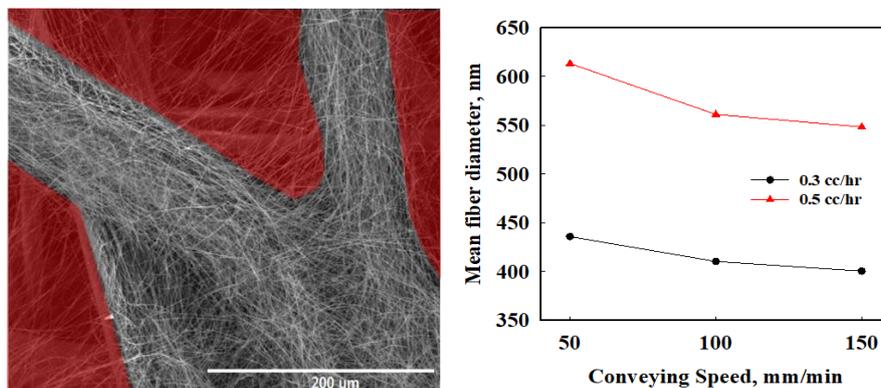


Fig. 8. Relation of diameter change by injection rate.

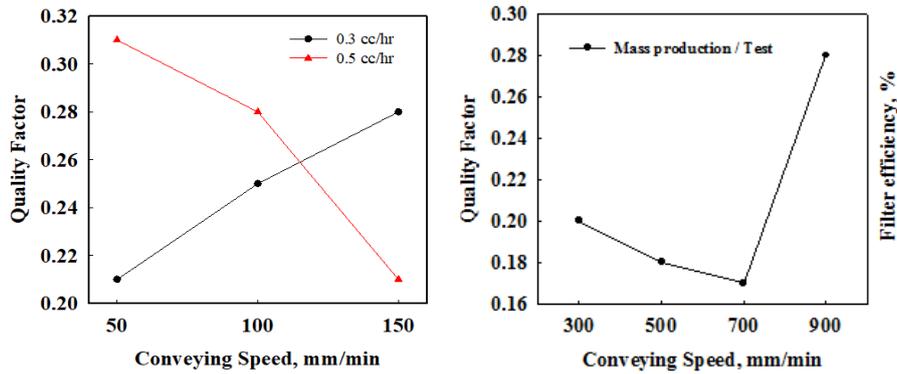


Fig. 9. Results of quality factor by injection rate and PES Media.

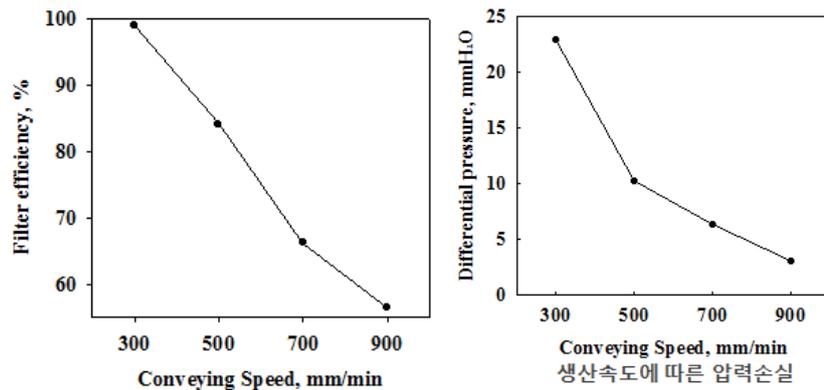


Fig. 10. Results of filter efficiency and differential pressure by PES media.

4. Conclusion

The results obtained from this study are as follows.

It is related to density of nanofiber web, as the feed rate of fabric increases. The density is also lower, it was also confirmed that the polymer concentration increased from 26 wt% to 30 wt% and the average pore size also increased at the same material transfer rate.

The maximum pore diameter is less than 22 μm at an applied voltage of 20 kV, the standard deviation of nanofiber diameter at each applied voltage means that the fibers radiated at 20 kV from 18 kV to 245.84 nm, 20 kV at 183.52, and 22 kV at 232 kV were more uniformly emitted, it has been proved that the nanofibers are radiated unevenly due to the effect of the strong applied voltage at 22 kV. The average pore size increased 1.06 μm much from at 3.20 μm to 4.26 μm , when the feeding rate of the fabric was 100, in the case the amount of the polymer solution was decreased from 0.5 cc / hr to 0.3 cc / hr, it is verified that the maximum pore diameter increased from 7.29 μm to 12.58 μm .

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