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Electrospinning of Poly(ether sulfone) and Evaluation of the Filtration Efficiency

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Abstract: To produce high heat-resistant air filter, filtration properties of poly (ether sulfone) (PES) made by various electrospinning conditions were evaluated. The PES webs of 0.4-1.1 μm average diameter fiber were obtained from 35-40 wt% PES / N,N- Dimethylacetamide (DMAc) solution. The diameter profile of electrospun PES web was clearly affected by PES concentration of the spinning dope and feeding rate of the dope, while the take-up speed effects little. The needle-collector distance affects the diameter profile for higher feeding rate conditions. The pore size of these webs was 1.3 - 5.6 μm, which was decided not only average fiber diameter but also fiber diameter variation. Both filtration efficiency and pressure loss were dropped steeply at about 3.0 μm of pore size. For the web having a pore size of 3.2 μm, the pressure loss decrease to 215 Pa, while the filtration efficiency for 0.3 μm particle kept 99.9998 %, which satisfied the HEPA requirement.

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1. Introduction

There are many methods to produce the ultra-fine fiber; conjugate spinning, melt-blowing, and flash spinning. Electrospinning, in which a polymer solution or a polymer melt is extended by electrostatic force, is one of the processes recently studied frequently. It have a long history, first patent was applied in 1934 [1], and the production of ultra-fine fiber of less than 1 μm diameter was reported in 1971[2]. With the increase of ultra-fine fiber's demand, it attracts attention again as a technique of making ultra-fine fiber from various materials recently. Followings are the basic researches; influence of the spinning-dope solvent to the spinning behavior [3-5], influence of the spinning-dope concentration to the spinning behavior [6-8], and influence of the spinning conditions to the mechanical properties of obtained fibers [9-12].

High performance filter is one of the most promising practical uses of electrospun webs because the fiber diameter is a very important factor to decide the filtration properties of web filter. There are some electrospun web filters already made in laboratory [13-16]. In this study, we have evaluated filtration properties of electrospun poly (ether sulfone) (PES) web of various diameter's fiber. Because the PES has almost the highest glass transition temperature (220 °C) and anti-creep property as an amorphous polymer, the electrospun PES web is expected to use as a heat-resistant high performance air filter.

2. Experimental

2.1 Electrospinning

Sumika Excel PES 3600G (SUMITOMO CHEMICAL Co.,Ltd.), of which intrinsic viscosity is 0.29 dL/g, was used in this study. The electrospinning dope was made by dissolving 15-40 wt% PES in m- Cresol, N,N- Dimethylacetamide (DMAc), N,N- Dimethylformamide (DMF), and N– Methyl– 2- Pyrrolidone (NMP). Fig. 1 shows the schematic diagram of the electrospinning system,

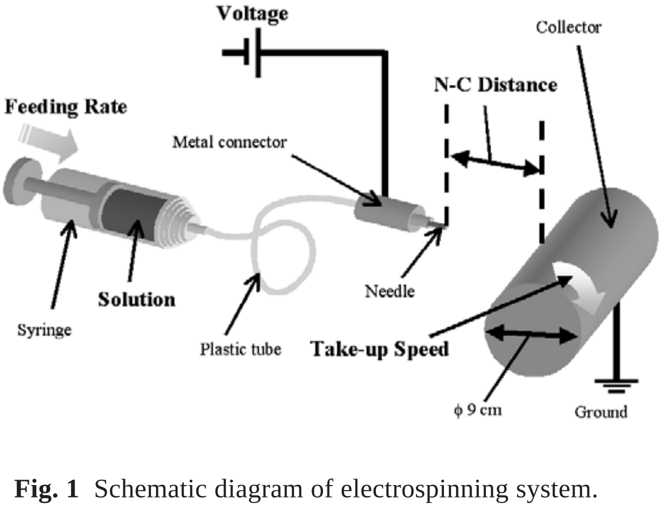

Fig. 1 Schematic diagram of electrospinning system.

composed of a needle, a power source, and a drum type collector. The electrospinning system and KDS-200 solution feed system used in this study were produced by nanoNC Co., Ltd. The spinning dope fed from the 0.65 mm diameter needle was drawn by the electrostatic force of electric field between needle and collector, and was collected by drum type collector. The drum collector was not only rotated at certain speed (take-up speed), but also traversed axially to keep the surface density uniform. The location of the drum was also changed to control the distance between needle tip and drum collector (N-C distance).

2.2 Electron microscope observation

Electrospun PES web was observed by field emission scanning electron microscope JSM-6330F or JSM-6340F (JEOL Ltd.). The PES webs were electrospun on aluminum foil, which covered on the collector drum, and was treated by platinum vapor deposition. The 0.1 μm step diameter distribution was obtained from the microscopic image of 60 μm x 40 μm area. For every image, diameter of all well-focused 50 - 100 fibers was measured by the image processing system Image-Pro Plus produced by Cybernetics Co. Ltd.

2.3 Pore size and filtration properties

Effective pore size and filtration efficiency of the web were evaluated. The sample web was electrospun on the poly (ethylene terephthalate) web substrate. Because pore size of the substrate, 150 - 200 μm, is far bigger than the pore size of samples, it acts almost no effect on the results. The pore size was measured by capillary flow porometer CFP-1200-AEL (PMI. Co. Ltd.). Starting from the state that all pores of the sample were fulfilled by a liquid, the pore size was estimated by the measurement of air pressure as the function of air flow rate. The Pore Wick, whose surface tension (τ) is 16 dyne/cm, was employed for the pore filler liquid in this study. Pore size (d) was estimated by equation (1) with constant C, surface tension τ, and the pressure at the airflow rate steeply increased (P). The constant C is 0.415 for the PSI pressure unit,

$$d = C\,\tau\,/P \tag{1}$$

The fractional efficiency filter tester TSI3160 (TSI Co. Ltd.) was used for the evaluation of filtration efficiency, the permeation percentage for certain size of particles. 0.15 μm and 0.3 μm size NaCl aerosol particles were used in this study.

3. Results and discussion

3.1 Electrospinning behavior

The effects of solvent, PES concentration, N-C distance and applied voltage to the electrospinning behavior and the resulting web were observed. For the case that PES / m-cresol solution was used for the electrospinning dope, electrospun web could not be obtained because the fiber had not solidified on the collector. For the case of PES / NMP solution, although the fiber could be obtained for 35 - 40 wt% of PES concentration and at least 15 cm of N-C distance, as shown in Fig. 2 (a), less uniform fibers and the fiber bonding points were observed. Also for the case

Table 1 Volatile properties of solvent.

Solvent	Boiling Point / ℃	Vapor Pressure /Pa
m-cresol	202	13
DMAc	165	330
DMF	153	492
NMP	202	66

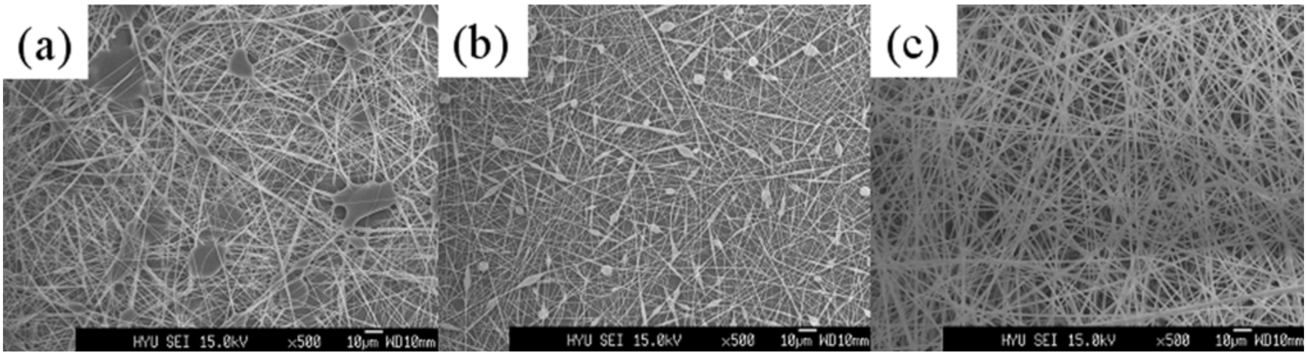

Fig. 2 PES nanofiber web obtained by the electrospinning condition of (a) 40 wt% NMP solution, 25 cm N-C Distance, and 15.9 kV applied voltage; (b) 35 wt% DMF solution, 25 cm N-C Distance, and 16.7 kV applied voltage; (c) 37.5wt% DMAc solution, 20 cm N-C Distance, and 15.8 kV applied voltage. Feeding rate was 1.0 ml/h for all.

of PES / DMF, fiber can be obtained for the range of 25-35 wt% PES concentration and at least 10 cm of N-C distance, less uniform fibers with beads were observed as shown in Fig. 2 (b). By contrast for the case of PES / DMAc, uniform fibers were obtained as shown in Fig. 2(c). The vapor pressure and the viscosity of solution strongly influenced to the evenness of electrospun web. For example, the vaporizing rate of m-cresol and NMP, which have higher boiling temperature and lower vapor pressure than the other solvent as shown in Table 1, are too low to solidify the spun fiber within the N-C distance. On the other hand, the low viscosity of DMF solution should cause the ununiform drawing and the capillary breakage of fiber.

The electrospinning behavior depends on the applied voltage [18]. If the voltage was too low, fiber cannot be made because the solution does not reach the collector and drop down. For the suitable voltage, several centimeter's stable jet of spinning dope spouted from the needle as shown in Fig. 3. The stable jet length was decreased with the increase of applied voltage, and at last the jet was vanished. Then the spinning dope scattered from the needle. Because the most uniform fiber was obtained when the stable jet length was the maximum, applied voltage used for the following measurements (Table 2) was decided as the minimum voltage to keep the stable jet stably.

3.2 Fiber diameter and pore size

Table 2 summarizes average diameter, pore size and filtration properties of the web samples produced from PES/ DMAc solution electrospun at various conditions. The average fiber diameter is increased with the increase of PES concentration and the increase of feeding rate, whereas there is no clear difference between the average diameter for take-up speed of 18 m/min and 90 m/min. This result suggests that the fiber running speed is far faster than 90 m/min. If there was no splitting occurred in the electrospinning process, fiber running speed should be run up to 6000 m/min for the condition No. 1. Then the jet splitting number should be less than several tens.

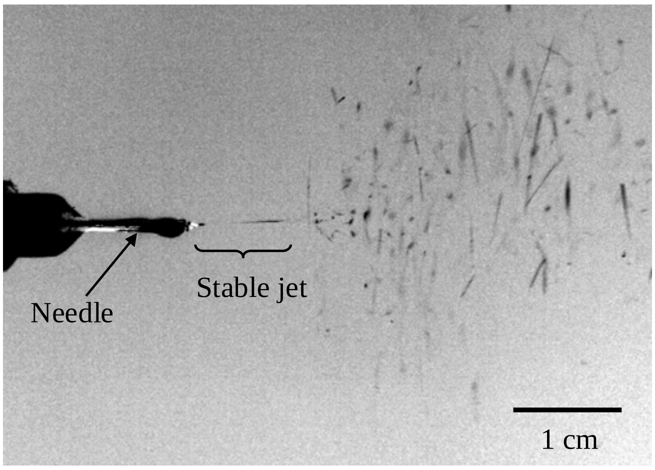

Fig. 3 Close-up photo around the spinning needle. Electrospinning conditions are equal to the No.4 in Table 2.

The fiber diameter profiles depend on the feeding rate, N-C distance, and PES concentration are shown in Fig. 4 and 5. For every PES concentration, the diameter profile of feeding rate 1.0 ml/h shows a long tail in the thicker side. In addition, although the web electrospun at 0.2 ml/h feeding rate have a sharp diameter distribution for all N-C distance, while the web electrospun at 1.0 ml/h feeding rate shows a longer tail for N-C distance of 20 cm N-C distance than the tail for N-C distance of 10 cm. These results indicate that the spun fiber solidified less than 10 cm from needle for feeding rate of 0.2 ml/h, while the fiber did not solidified at 10 cm from needle for feeding rate of 1.0 ml/h.

The pore size measured by capillary flow analysis is compared with the fiber diameter profile in Fig. 6. The larger fiber diameter causes the larger pore size, but the pore size is not simply proportional to the average fiber diameter. For example, sample No. 12 and No. 13 shows bigger pore size / fiber diameter ratio than the other. In particular the big pore size of No. 12 is interesting because the web density of the sample is also big. The pore size / fiber diameter ratio should be corresponding to the thicker-side tail of fiber diameter profile. That is, if the average fiber diameter is the same, the long thicker-side tail of diameter distribution leads bigger pore size, which indicates that the larger unevenness in fiber diameter make the bigger pore.

3.3 Filtration properties

The filtration properties of the samples shown in Fig. 6 are listed in Table 2. Over the pore size of 3.0 μm, both filtration efficiency and pressure loss are decreased steeply. However, the decreasing rate of pressure loss is smaller than that of filtration efficiency. Following the JIS standard, the web having more than 99.9995% of filtration efficiency for 0.15 μm particle and less than 245 Pa pressure loss is defined as ULPA (ultra low penetration air filter), and the web having more than 99. 97% of filtration efficiency for 0.3 μm particle and less than 245 Pa pressure loss is defined as HEPA (high efficiency particulate air filter) [19]. Filtration efficiency of No. 1, 4, 10 samples satisfy the ULPA requirement, but the pressure losses exceed the standard. And the sample No. 11, which has 3.2 μm pore size, satisfies the HEPA requirements.

Table 2 Electrospinning conditions and filtration properties for PES/DMAc system.

No.	PES Conc. / wt%	Feeding Rate / ml h^{-1}	N-C Distance / cm	Applied Voltage / kV	Take-up Speed / m min^{-1}	Avarage Diameter / μm	Avarage Pore Size / μm	Pore Size / Diameter	Filtration Efficiency x100		Pressure loss /Pa		Web Density / g m^{-2}
									Particle Size				
									0.12 μm	0.3 μm	0.12 μm	0.3 μm	
1	35	0.2	20	12.0	18	0.4	1.3	3.0	99.9998	99.9992	660	662	18.7
2	35	0.2	20	11.3	90	0.5	-	-	-	-	-	-	-
3	35	0.2	25	13.2	18	0.4	-	-	-	-	-	-	-
4	35	1.0	20	15.0	18	0.7	-	-	-	-	-	-	-
5	37.5	0.2	10	11.0	18	0.7	-	-	-	-	-	-	-
6	37.5	0.2	15	14.5	18	0.7	-	-	-	-	-	-	-
7	37.5	0.2	20	12.6	18	0.7	2.8	4.0	99.9998	99.9991	647	647	17.4
8	37.5	0.2	25	14.8	18	0.8	-	-	-	-	-	-	-
9	37.5	1.0	20	15.8	18	0.9	-	-	-	-	-	-	-
10	40	0.2	20	12.6	18	0.9	3.0	3.5	100.0000	99.9998	326	325	19.5
11	40	0.5	20	12.2	18	0.9	3.2	3.6	99.997	99.9998	215	215	23.3
12	40	1.0	10	11.3	18	1.1	5.6	5.0	96.7	97.8	135	134	30.9
13	40	1.0	20	12.8	18	1.0	4.3	4.3	99.4	99.7	184	182	30.8

4. Conclusion

The PES web satisfying the HEPA requirement was obtained from the electrospinning of PES / DMAc solution. The fiber diameter profile and filtration properties of the PES web produced by various electrospinning conditions are measured. The results show that the diameter profile was clearly affected by the PES concentration and feeding rate of the spinning dope, whereas almost no influence of take-up speed was observed. The needle-collector distance affects the diameter profile for higher feeding rate conditions. Pore size of web is affected not only average fiber diameter but also fiber diameter distribution. Both filtration efficiency and pressure loss were steeply decreased over the pore size of 3.0 μm.

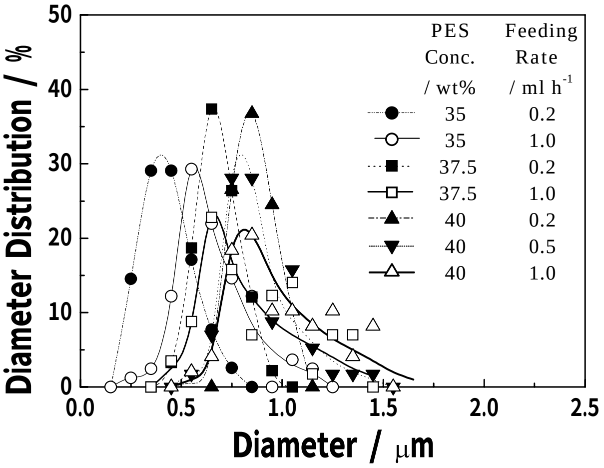

Fig. 4 Feeding rate dependence on fiber diameter profiles for No. 1, No. 4, No. 7, No. 9, No. 10, No. 11 and No.13 of Table 2. Every N-C Distance and Take-up Speed are 20 cm and 18 m/min respectively.

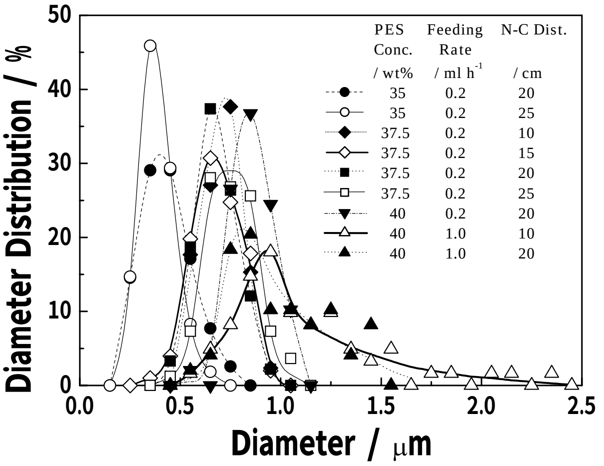

Fig. 5 N-C distance and PES concentration dependence on fiber diameter profiles for No. 1, No. 3, No. 5, No. 6, No. 7, No. 8, No. 10, No. 12 and No.13 of Table 2. Every take-up speed are 0.2 ml/h.

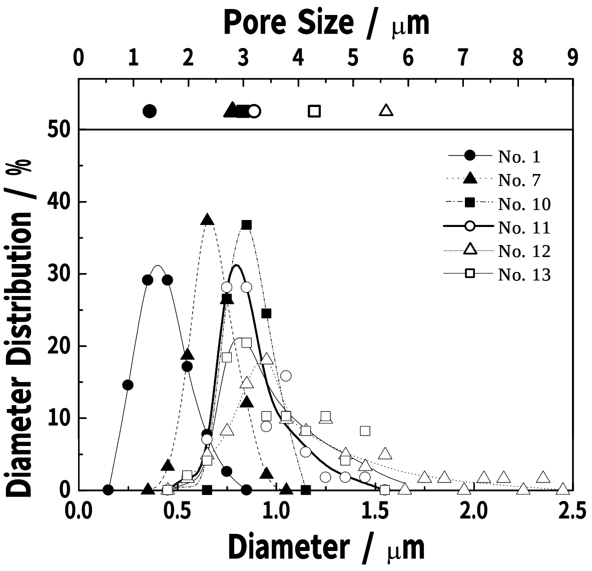

Fig. 6 Fiber diameter and Pore size of the PES web. Electospinning conditions are corresponding to No.1, No.7, No.10, No.11, No.12, and No.13 of Table 2.

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