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Filtration Properties of Electrospun Ultrafine Fiber Webs

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Abstract−Electrospun fiber webs were prepared at various spinning conditions. The effect of electrospinning parameters on fiber morphology and filtration performance of electrospun webs was investigated. The processing variables considered were only the applied voltage and rotation speed of a drum type collector. The fiber diameter and mean pore size of the electrospun webs decreased with increasing applied voltage and collector speed. Pressure drop and aerosol collection efficiency of the electrospun fiber webs were increased with decreasing fiber and pore size. Additionally, it was found that the filtration performance of the electrospun web was much greater than that of a commercial high efficiency air filter media made of glass fibers.

Key words: Electrospinning, Electrospun Fiber, Filtration Performance, Aerosol Collection Efficiency, Pressure Drop

INTRODUCTION

Electrospinning, which is the electrostatic method generating ultrafine fibers, has been recently rediscovered for applications such as reinforced composites, filtration, protective clothing, and biomedical material [Frenot and Chronakis, 2003; Gibson et al., 2001; Buchko et al., 1999]. Electrospun fibers, in general, have a size range of 40-2,000 nm in diameter. Since the nano size fibers and porous structure of electrospun fiber webs result in a very high specific surface area, the electrospun filter media has very high collection efficiency [Tsai et al., 2003]. Most of the studies on electrospinning have been focused on the morphological characteristics of the electrospun materials [Deitzel et al., 2001a, b; Doshi and Reneker, 1995; Fong et al., 1999, 2002], while only a few studies on applications have been addressed [Gibson et al., 2001; Tsai et al., 2003]. Furthermore, there have been few systematic studies on the filtration properties of electrospun fiber webs, even though the electrospun ultrafine fiber nonwovens have been commercially used for air filtration applications.

In this study, consequently, the filtration characteristics of electrospun fiber webs were systematically examined. Ultrafine fiber webs were manufactured with various spinning conditions by a lab-scale electrospinning apparatus. The effect of processing variables of electrospinning on fiber size distribution and pore size distribution of the webs was analyzed. Finally, the filtration properties of the electrospun fiber webs were measured and compared with a commercial air filter media composed of glass fibers.

EXPERIMENTAL

1. Electrospinning

Nylon 6,6 was used, in this work, as a polymer material, and dissolved in formic acid. The concentration of Nylon 6,6 was 25% in weight. The Nylon 6,6 solution was spun by an electrospinning apparatus depicted in Fig. 1. The applied voltage was ranged from 20 to 50 kV, and the distance between the nozzle tip and collector was fixed as 110 mm. A drum type collector was used to investigate the effect of rotation speed of the collector on the morphology and characteristics of electrospun fibers. The collector speed was controlled by a frequency inverter and varied in the range of 30 to 100 RPM. Electrospun fibers were collected on the rotating drum for a few minutes, so that nonwovens were formed. In order to ensure perfect evaporation of solvent, the electrospun fiber webs were dried in the atmospheric condition for over 24 hours.

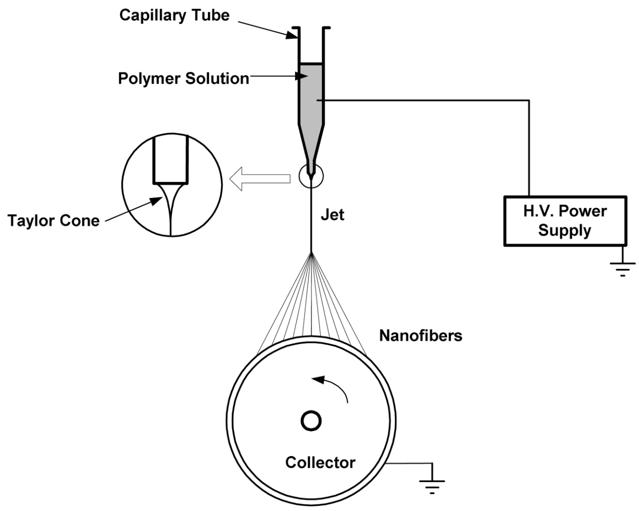

Fig. 1. Schematic diagram of electrospinning apparatus.

2. Characterization of Electrospun Webs

The morphology of the electrospun fibers was analyzed by a scanning electron microscope (SEM). Based on the SEM images, the size distribution of electrospun fibers was obtained. Since the pore size of a filter media plays a very significant role in aerosol filtration, pore size distributions of the electrospun webs were measured with a capillary flow porometer (CFP-1200 AEL, PMI Inc.). The porometer measures the distribution of pore size as well as the mean pore diameter.

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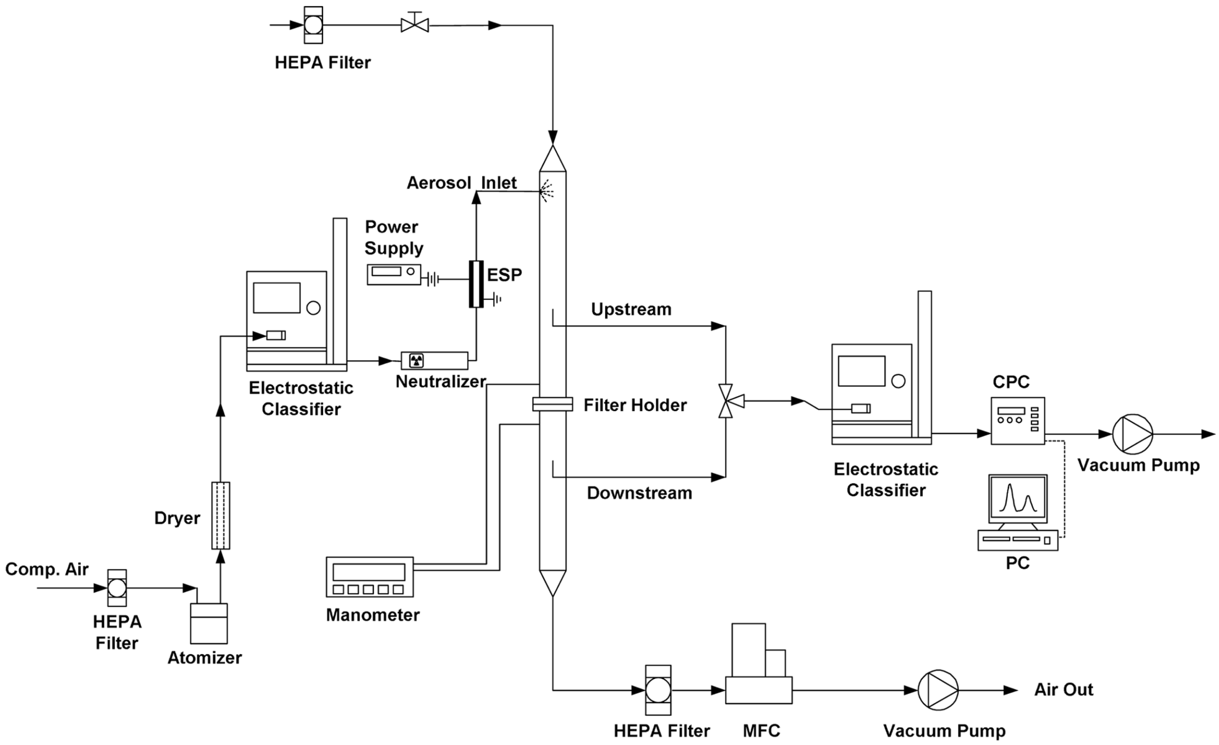


Fig. 2. Experimental setup for evaluating the filtration properties of electrospun webs.

3. Filtration Properties

Fig. 2 shows the experimental set up for measuring filtration properties of electrospun fiber webs. Air permeability and aerosol collection efficiency are the most important factors to evaluate the filter performance. Air permeability is defined as the rate of air flow through a porous material under a specified differential pressure across the material, usually 0.5 inches of water. In the present study, the pressure drop across the tested electrospun webs was measured with a micro-manometer (FCO510, Furness Controls Ltd.) for various face velocities. Face velocity is defined as the flow rate through a filter per unit filtration area. Electrospun webs were cut into circular sheets of 52 mm in diameter, and installed in a filter holder. The flow through the electrospun webs was derived by a vacuum pump, and controlled by an electronic mass flow controller.

Aerosol collection efficiency of the tested webs was calculated by measuring the aerosol concentration before and after passing through the webs. NaCl particles were used as a test aerosol. Polydisperse NaCl aerosol was generated by a collision atomizer (Model 3079, TSI Inc.) and dried by a diffusion dryer packed with silica gel. The dried particles entered an electrostatic classifier (Model 3080, TSI Inc.) extracting a known size fraction of submicrometer particles from the incoming polydisperse aerosol by using an electrical mobility detection technique. That is, the electrostatic classifier produces monodisperse aerosol of known size. In this study, aerosols of 50, 70, 100, 150, 200, 300, and 400 nm in diameter were used.

Most of the classified particles are singly charged, and the electrospun fibers are also highly charged. Even the single charge of particles can affect the collection efficiency of the electrospun webs due to the coulombic force. For this reason, the classified particles were electrically neutralized by a Kr-85 aerosol neutralizer (Model 3077, TSI Inc.). The neutralized particles are in a charging state of Boltzmann equilibrium [Reist, 1993], in which most particles have zero charge, but some still have multiple charges. The charged particles were finally removed by an electrostatic precipitator, so that only electrically neutral NaCl particles entered the test section.

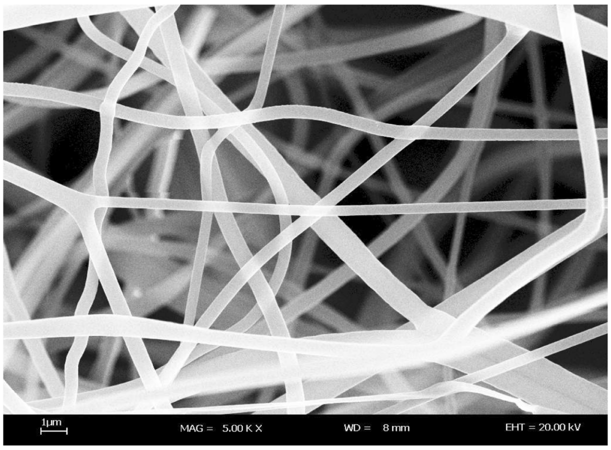
The concentration of NaCl particles was measured with a Scanning Mobility Particle Sizer (SMPS, Model 3936, TSI Inc.), which is composed of an electrostatic classifier (Model 3080, TSI Inc.) and a condensation particle counter (CPC, Model 3010, TSI Inc.). SMPS measures the size distribution of submicrometer aerosol, and is automated with a personal computer. Using the SMPS, we measured the upstream and downstream concentrations of NaCl particles, and then calculated the aerosol collection efficiency of the electrospun fiber webs.

RESULTS AND DISCUSSION

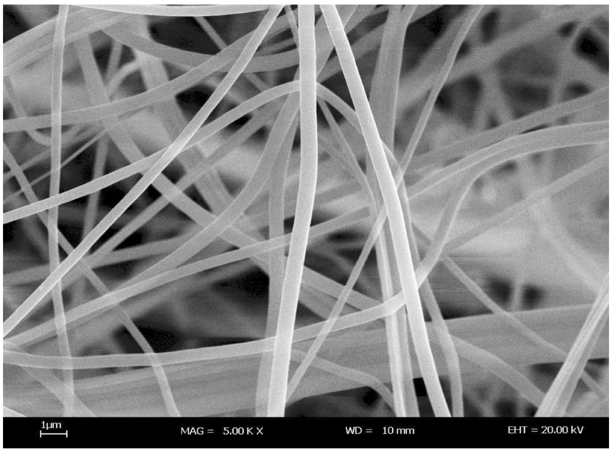
1. Morphology of Electrospun Ultrafine Fibers

Two electrospinning process variables were considered in this study: applied voltage and rotation speed of collector. Other variables such as the solution concentration, tip-to-collector distance, and feed rate of polymer solution were fixed.

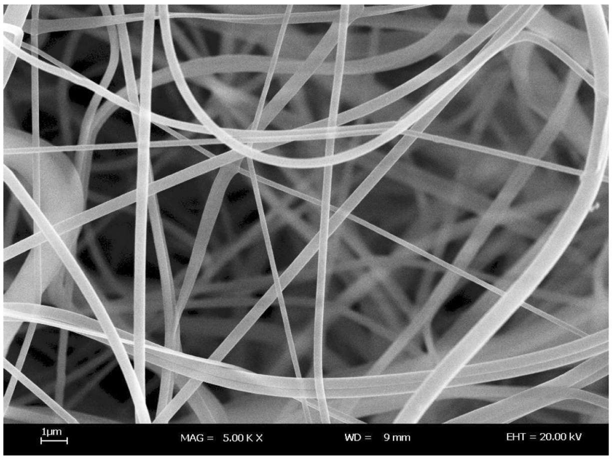
Fig. 3 shows SEM images of electrospun fibers as a function of applied voltage. The scale bar indicates 1 μm, and the magnification is 5,000×. 25 wt%. Nylon 6,6 solution was used as a polymer liquid, and the collector speed was 50 RPM for all samples of Fig. 3. As can be seen, the fiber diameter decreased with increasing applied voltage. In order to obtain more accurate information on the size distribution of electrospun fibers for the cases of Fig. 3, the diameter of each fiber was measured by using the SEM images. The number of fibers shown in an SEM photograph was so small that the size distribution could not be accurately represented. Therefore,



(a) 20 kV

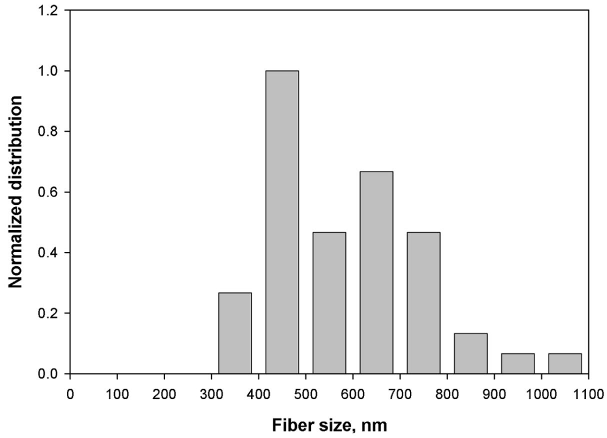


(b) 30 kV

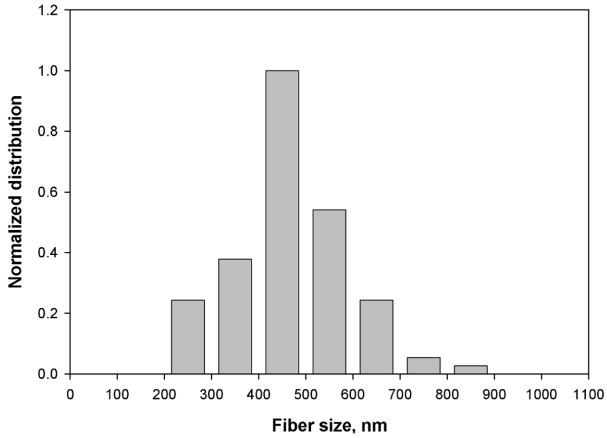


(c) 50 kV

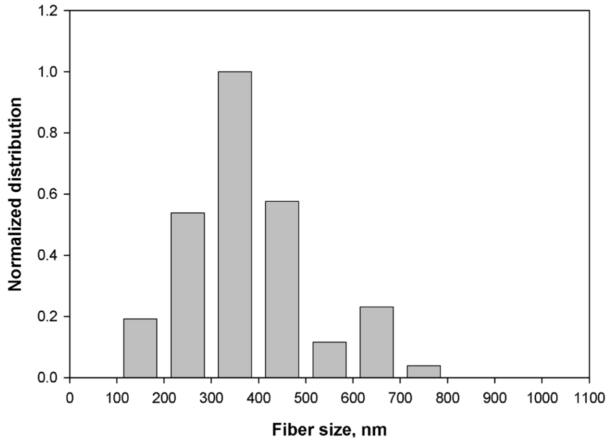
Fig. 3. SEM images of electrospun Nylon 6,6 fibers manufactured at various applied voltages.



(a) 20 kV



(b) 30 kV

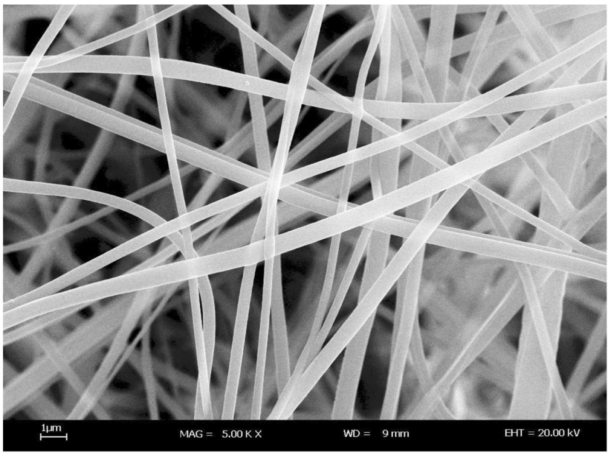


(c) 50 kV

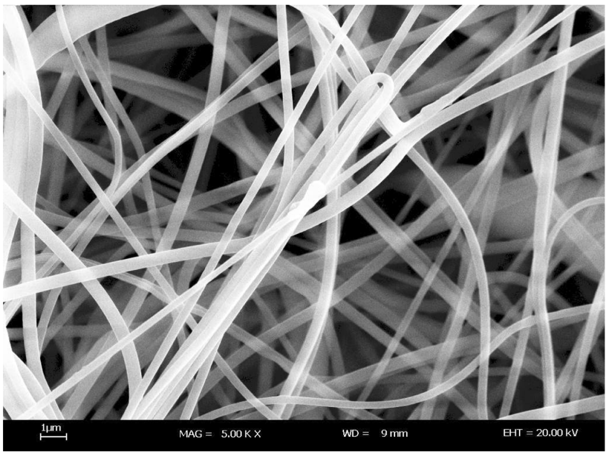
Fig. 4. Size distribution of electrospun fibers produced at various applied voltages.

additional two SEM photos for each case were used to increase the total fiber count, and ultimately to improve accuracy.

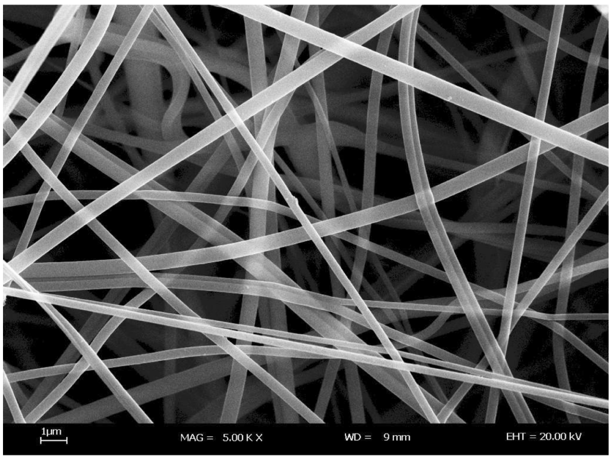
The fiber size distributions for each case of Fig. 3 were plotted in Fig. 4. The ordinate denotes the normalized distribution with respect to the maximum value. The difference of fiber size distribution among the three cases becomes clear. In the case of 20 kV, the peak existed in 400 nm regime, and most of the other fibers were larger than 500 nm. On the other hand, for the case of 30 kV, the number of fibers smaller than the peak size was increased even though the peak was located at the same size range with that of the case of 20 kV. When the applied voltage was 50 kV, much finer fibers were generated. The peak fiber diameter was in 300 nm, and the distri-



(a) 30 RPM

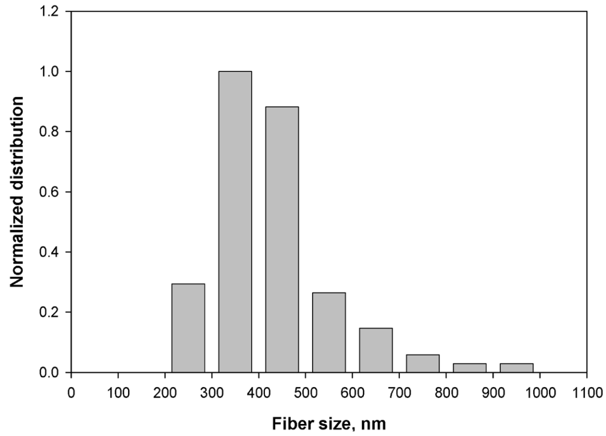


(b) 50 RPM

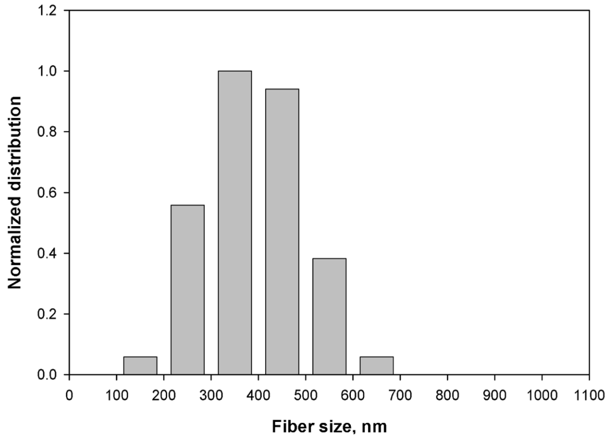


(c) 100 RPM

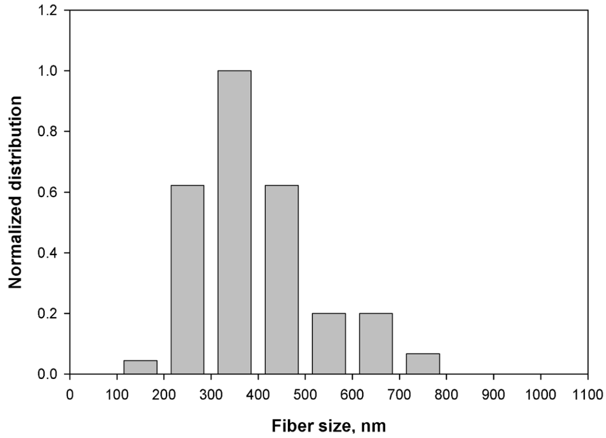
Fig. 5. SEM images of electrospun Nylon 6,6 fibers manufactured at various collector speeds.



(a) 30 RPM



(b) 50 RPM



(c) 100 RPM

Fig. 6. Size distribution of electrospun fibers produced at different collector speeds.

bution curve formed a kind of normal distribution in which the fiber counts on both sides of the peak were approximately balanced.

As a result, it was found that the diameter of electrospun fibers decreased with increasing electric field. This coincides with the results of existing works [Buchko et al., 1999; Fong et al., 1999]. The ultrathin fibers obtained in the electrospinning process are produced as a result of spiral motion and elongation of a polymer jet driven by a high electrical potential applied between the liquid polymer in a capillary and the collector, as shown in Fig. 1. Those events occur when the repulsive electrostatic forces in the polymer jet become large enough to overcome the adhesive force within the jet. It is supposed that the spiral motion and elongation of a polymer jet in an electrospinning process with higher electric field strength become more active, and consequently much finer fibers are obtained.

Fig. 5 shows the morphology of electrospun Nylon 6,6 fibers manufactured at different collector speeds. For all cases of Fig. 5, the potential applied to polymer solution was 40 kV, and the solution concentration was fixed as 25 wt%. Unlike the flat type collector, the drum collector used in the present work can give an additional variable to make an effect on the fiber shape. The variable is the rotation speed of collector. The rotating collector drives a strong centrifugal force in the vicinity of collector surface. This force elongates the fibers, which are about to be collected, along the rotational direction.

From Fig. 5, the difference of fiber size between the three samples is hard to be distinguished, so the fiber size distributions were graphed in Fig. 6 as the same manner with Fig. 4. For all cases, the peak sizes were almost same as 300 nm, but the distribution curve was slightly shifted to the left side indicating smaller fiber diameter as the collector speed was increased. Accordingly, through this study, it was validated that the size of electrospun fibers could be controlled by the rotation speed of a cylindrical type collector.

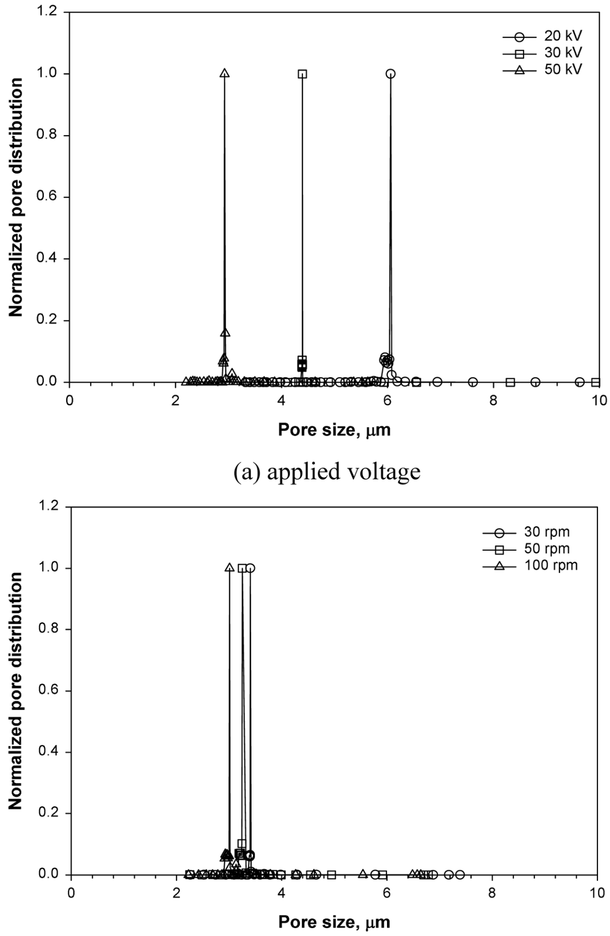
2. Pore Size Distribution

The pore size of a fibrous filter media is an absolutely important factor determining the filtration characteristics of the media. Generally smaller pore of a fibrous air filter leads to higher air resistance and higher aerosol collection efficiency simultaneously, both of which are the most critical properties of air filters. In this study, the pore size distributions of the electrospun fiber webs were measured with a capillary flow porometer. Table 1 lists the mean pore size of electrospun webs manufactured at different spinning conditions. The mean pore size ranged from 3 to 6 μm. As the applied voltage and collector speed increased, the pore size decreased. Fig. 7 shows the pore size distributions of the electrospun fiber webs described in Table 1. Basically, each of the webs had an extremely uniform pore distribution. That is, only the pores of equal size were formed in the electrospun webs. The uniform pore distribution property of electrospun fiber webs is very important in application as membrane materials requiring higher uniformity of pore size.

The peak pore size, which is nominally identical to the mean pore size for the webs considered here, was significantly decreased with increasing electric field. On the other hand, the peak size was decreased slightly with collector speed. Correlating these results with the fiber size distributions of the electrospun webs mentioned in Fig. 4 and Fig. 6, it can be seen that the fiber size is the crucial parameter to determining the pore size of an electrospun fiber web.

Table 1. Mean pore size of electrospun fiber webs prepared through this study

Electrospinning parameters		Mean pore size of electrospun webs, μm
Applied voltage, kV	Collector speed, RPM	
20	50	6.06
30	50	4.40
50	50	2.93
40	30	3.40
40	50	3.25
40	100	3.00



(a) applied voltage

(b) collector speed

Fig. 7. Pore size distribution of electrospun fiber webs.

3. Filtration Properties: Air Permeability

The primary aim of this study is to investigate the filtration characteristics of electrospun fiber webs and the effect of electrospinning parameters on filtration properties of the webs. The filtration properties considered here are the air permeability and aerosol collection efficiency. This section describes the air permeability of electrospun nanofiber webs.

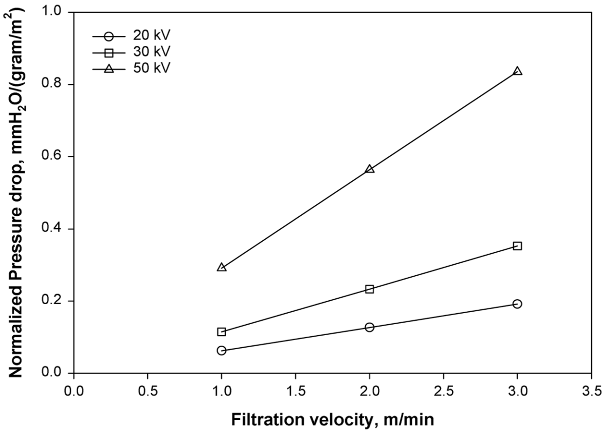
ASTM (American Society for Testing and Materials) defines the air permeability as the rate of air flow passing perpendicularly through a known area under a prescribed air pressure differential between the two surfaces of a material [ASTM D 737]. Darcy's law, which is the first law of filtration theory, states that the pressure drop across a porous material is proportional to the flow rate of fluid through it [Brown, 1993]. Equating this sentence, the differential pressure across a filter media is expressed as follows.

$$\Delta P = \left(\frac{Q}{A_f}\right)\left(\frac{\mu}{\kappa}\right) t = K \mu V_f \quad (1)$$

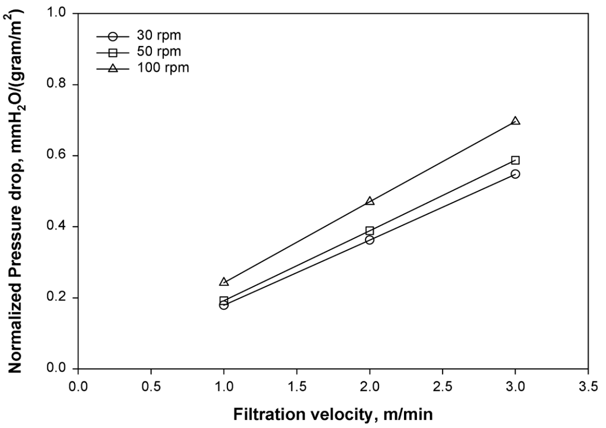
where ΔP is the differential pressure across a filter media, Q the flow rate, A_f the filtration area, μ the viscosity of fluid, κ the permeability of the filter media per unit media thickness per unit viscosity, K the constant of the filter media, defined as t/κ, and V_f the face velocity defined as Q/A_f.

Based on Eq. (1), air permeability of the electrospun fiber webs was evaluated by measuring the differential pressure across the webs at several distinct face velocities. Pressure drop data were plotted in Fig. 8. For the assessment of air permeability for each sample, all the measured pressure drop data should be normalized to the thickness of each sample because the pressure drop of a filter is directly proportional to its thickness. Due to difficulty in measuring the thickness of electrospun webs, however, pressure drop data were normalized to the basis weight of each sample. Table 2 shows the basis weight data of electrospun fiber webs used for evaluating filtration performance.

As seen in Fig. 8, the slope of pressure drop was increased with increasing applied voltage and collector speed. Higher slope means lower air permeability. Considering the fiber and pore size distributions of electrospun webs, the variation of air permeability of the webs manufactured at different spinning conditions is readily understood. Consequently, air permeability of the electrospun webs, which is deeply related to both of fiber and pore size distribution of the webs, decreased with decreasing fiber and pore size.



(a) applied voltage



(b) collector speed

Fig. 8. Air permeability of electrospun fiber webs.

Table 2. Basis weight of electrospun fiber webs used for evaluating filtration performance

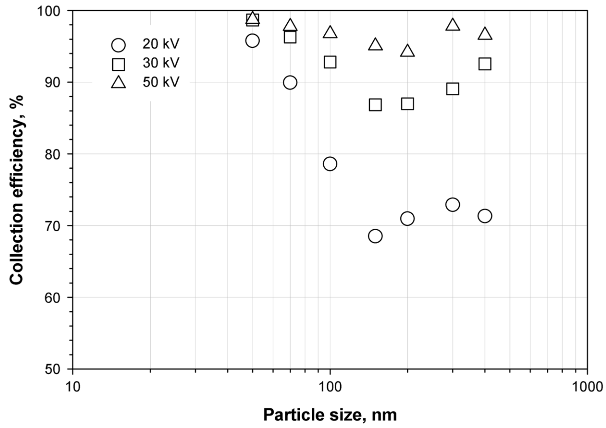
Electrospinning parameters		Basis weight of electrospun webs, g/m^2
Applied voltage, kV	Collector speed, RPM	
20	50	17.75
30	50	18.22
50	50	15.89
40	30	16.48
40	50	16.15
40	100	17.84

4. Filtration Properties: Aerosol Collection Efficiency

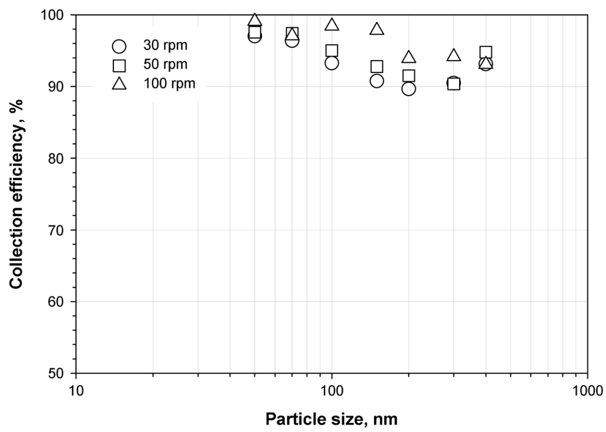
Collection efficiency of electrospun fiber webs for submicron aerosol particles was plotted in Fig. 9. Monodisperse NaCl particles were used as test aerosols. Collection efficiency of a fibrous filter media was calculated as follows.

$$\eta = \left[1 - \left(\frac{C_{downstream}}{C_{upstream}}\right)\right] \times 100,\ \% \tag{2}$$

where η is the collection efficiency, $C_{upstream}$ and $C_{downstream}$ the aerosol concentrations before and after passing through the media, respectively. In this study, the number concentration of aerosol particles was measured.



(a) applied voltage



(b) collector speed

Fig. 9. Collection efficiency of electrospun filter media for submicron aerosol particles.

The measured efficiency data shows a typical collection efficiency curve, which has a minimum value in the particle size range of 100 to 300 nm [Lee and Liu, 1980; Park et al., 2001]. The variation of efficiency between the tested webs showed a similar trend with the case of air permeability. The electrical potential made a significant effect on the collection efficiency of electrospun webs, while the collector speed gave only a little influence on the efficiency. These results can be also explained by using the fiber and pore size distribution data. Reduced fiber size of a fibrous web induces increased specific surface area of the web, which enables the probability of particle deposition on fiber surface to be increased. Particle collection mechanisms concerned with this study are mainly Brownian diffusion, interception, and electrostatic attraction. The electrostatic attraction becomes more important when the fiber and particles are charged, but in this study the fiber charge was too weak to affect the collection efficiency [Park, 2004]. Therefore, the difference of collection efficiencies between the electrospun webs produced at different applied voltages is mainly due to fiber diameter.

5. Comparison with Commercial Air Filter Media

The collection efficiency of an electrospun fiber web was compared with a commercial high efficiency air filter media composed of glass fibers. The glass fiber air filter has a tentative collection efficiency of 98% for 0.3 μm spherical aerosol at face velocity of 3 m/min. The electrospun fiber web was manufactured at the applied voltage of 50 kV and collector speed of 50 RPM. The basis weight of the web is 16.48 g/m^2, while the commercial glass fiber filter media have 81.46 g/m^2. Pressure drop at 3 m/min of face velocity was measured as 13.27 mmH_2O for the electrospun web and 37.05 mmH_2O for the glass fiber filter media.

Fig. 10 shows the collection efficiency of the two samples. The efficiency of the electrospun web was slightly higher than that of commercial glass fiber filter media. This result indicates that the electrospun fiber web has much greater filtration performance relative to the high efficiency glass fiber filter media.

CONCLUSIONS

Through this work, filtration characteristics of electrospun ultrafine fiber webs were experimentally studied. The effect of processing parameters on the fiber morphology and pore size distribution of the electrospun webs was examined. The parameters considered were the applied voltage and rotation speed of collector. As a result, it was found that the fiber size of electrospun webs was largely influenced by the electrical potential applied to polymer solution, and also slightly increased with increasing collector speed due to the elongation of the fibers along the rotating direction. Higher applied voltage induces higher charge density of a polymer jet, ultimately resulting in finer fiber size. Thinner fibers of electrospun webs led to lower pore size, which is the key parameter determining the filtration performance of a filter media. Both of pressure drop across an electrospun web and aerosol collection efficiency of the web were increased with decreasing mean pore size. Consequently, the process variables of electrospinning, especially the applied voltage of them, gave a significant effect on filtration properties of the electrospun fiber webs. The filtration performance of the electrospun webs was much greater than that of a commercial high efficiency glass fiber filter media, which is widely used for air conditioning process. It means that the electrospun fiber webs have the possibility to be used as a main material of fibrous air filter media.

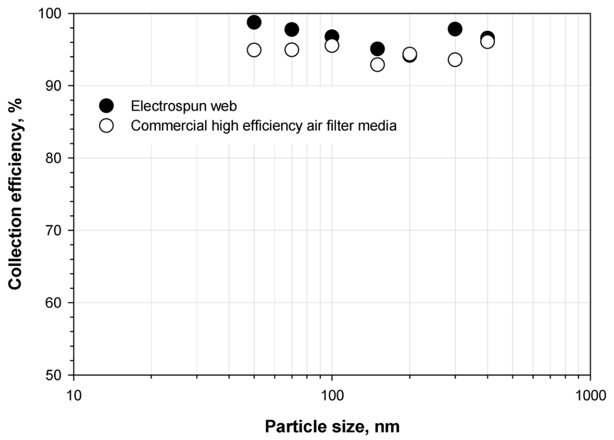


Fig. 10. Comparison of collection efficiency of an electrospun web manufactured at 50 kV and 50 RPM with a commercial high efficiency air filter media.

ACKNOWLEDGMENT

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NOMENCLATURE

A_f : filtration area [m^2]
$C_{upstream}$: upstream aerosol concentration [#/cc]
$C_{downstream}$: downstream aerosol concentration [#/cc]
K : constant of a filter media [m^{-1}]
ΔP : pressure drop across a filter media [Pa]
Q : flow rate [m^3/s]
t : filter thickness [m]

Greek Letters

η : collection efficiency [%]
κ : permeability of a filter media per unit media thickness per unit viscosity [m^2]
μ : viscosity of fluid [Pa·s]

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