Electrospun Ultrathin Nylon Fibers for Protective Applications

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ABSTRACT: Electrospun nylon 6 fiber mats were deposited on woven 50/50 nylon/cotton fabric with the motive of making them into protective material against submicron-level aerosol chemical and biological threats. Polymer solution concentration, electrospinning voltage, and deposition areal densities were varied to establish the relationships of processing-structure-filtration efficiency of electrospun fiber mats. A high barrier efficiency of greater than 99.5% was achieved on electrospun fiber mats without sacrificing air permeability and pressure drop. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 116: 2181–2187, 2010

Key words: electrospinning; nylon 6; ultrathin fiber; protection; aerosol

INTRODUCTION

Electrospinning technique is being widely used to produce ultrathin fibrous mat structures from many different materials and the resultant fiber mats have found various applications including tissue engineering, catalytic reaction materials, electrochemical electrodes, affinity membranes, and nano-composites.[1–11]

Electrospun fiber mats are promising barrier material for aerosol filtration.[12] Unlike traditional filtration media that cannot simultaneously achieve high filtration efficiency and low pressure drop or high air permeability, electrospun fiber mats have small fiber diameters and large surface areas, and hence they can significantly increase the filtration efficiency without sacrificing the air permeability. Usually the electrospun fiber mats are deposited on fabric substrates to combine advantages of both materials.[12,13] In this work, electrospun nylon 6 fiber mats were deposited onto 50/50 nylon/cotton fabric to prepare a protective clothing material against chemical and biological warfare agents. Nylon 6 was chosen for the electrospinning of ultrathin fibers because it has good toughness, high abrasion resistance, and easy process ability. The effects of solution concentration, electrospinning voltage and deposition areal density on the structure, and performance of electrospun fiber mats were investigated to establish processing-structure-filtration efficiency relationships for these new materials. The focus of this article is on the protective performance of electrospun fiber-deposited fabrics.

EXPERIMENTAL

Electrospinning of nylon 6 fibers

Nylon 6 and 2,2,2-tri-fluoro ethanol were purchased from Aldrich and used without further purification. Nylon 6 solutions with different concentration were prepared under constant stirring at room temperature. Figure 1 shows the electrospinning setup used in experiments. A 10 mL plastic syringe fitted with a needle tip (inner diameter = 0.4 mm) was used for electrospinning. For all experiments, the feed rate was fixed at 0.3 mL/h, electrospinning voltage ranged from 8 to 20 kV, and the collector distance from the syringe tip was about 15 cm. Electrospun nylon 6 fiber mats (Fig. 2) were collected on 50/50 nylon/cotton woven fabric placed on a rotating drum type collector. The areal density of electrospun fiber mats were controlled by varying the deposition time.

Fiber structure characterization

The morphology of electrospun fiber mats was examined using scanning electron microscopy JEOL JSM-6400F at an accelerated voltage of 5 kV. The

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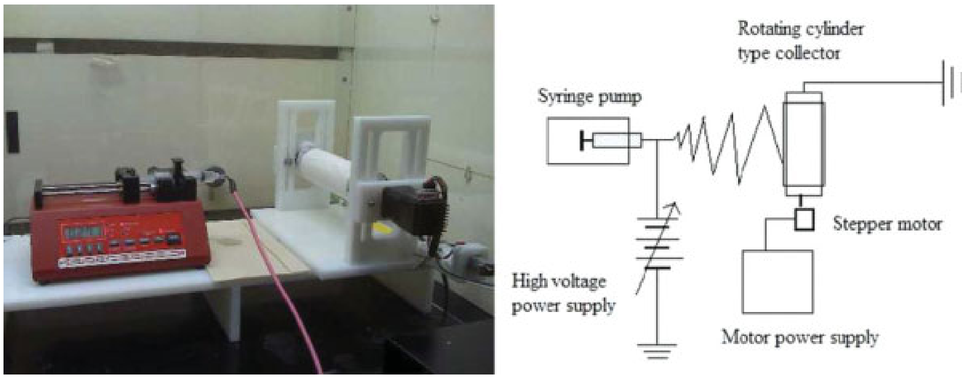

Figure 1 Photograph and schematic diagram of electrospinning setup. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

images were acquired and fiber diameters were obtained by measuring 100 electrospun fibers using the Revolution software provided by 4pi Analysis.

Filtration performance evaluation

The filtration performance and pressure drop value of the electrospun fiber mats deposited on nylon : cotton fabric was simultaneously evaluated using TSI 3160 advanced automated tester. During the evaluation, the TSI 3160 instrument used a bank of atomizers and an electrostatic classifier to challenge the electrospun fiber mats with 300 nm NaCl particles at a face velocity of 5.332 cm/s and an air flow rate of 32 L/min. Two condensation particle counters were used to simultaneously count the upstream and downstream particles and the penetration values were calculated.

Air permeability testing

The air permeability of electrospun fiber-deposited fabrics was measured separately using Frazier air permeability testing instrument. The measurement was carried out according to the ASTM D737-04 standard, with 1.4 mm orifice, 2.75 square inch test area, at 30 inch mercury pressure, 21°C, 65% RH.

RESULTS AND DISCUSSION

Filtration efficiency, pressure drop, and air permeability of electrospun nylon 6 fiber mats

One of the most important factors that affect the filtration efficiency of electrospun fiber-deposited fabrics is the areal density or basis weight of electrospun fiber mats. In this work, electrospun nylon 6 fibers were deposited onto 50 : 50 nylon/cotton woven fabrics from 12% wt/vol nylon solution at 10 kV spinning voltage. Deposited nylon 6 fibers have an average diameter of around 150 nm. The areal density of electrospun fiber mats was controlled by adjusting the fiber deposition time. Figure 3 shows the filtration efficiencies of nylon 6 fiber mat-deposited fabrics. Before fiber deposition (i.e., areal

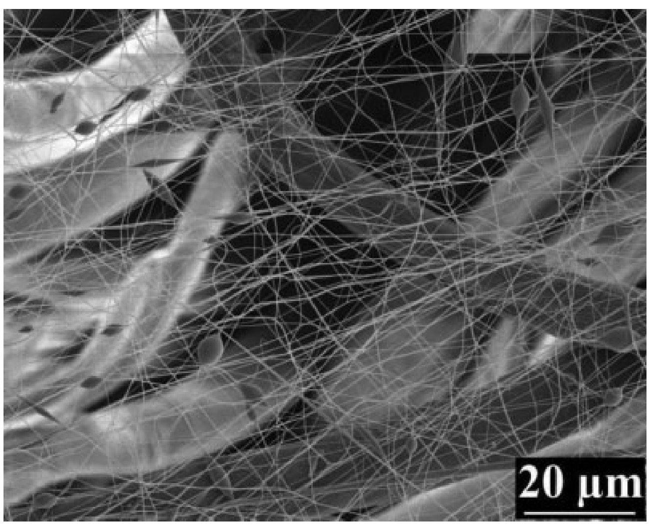

Figure 2 SEM image of a typical electrospun nylon 6 fiber-deposited nylon/cotton woven fabric.

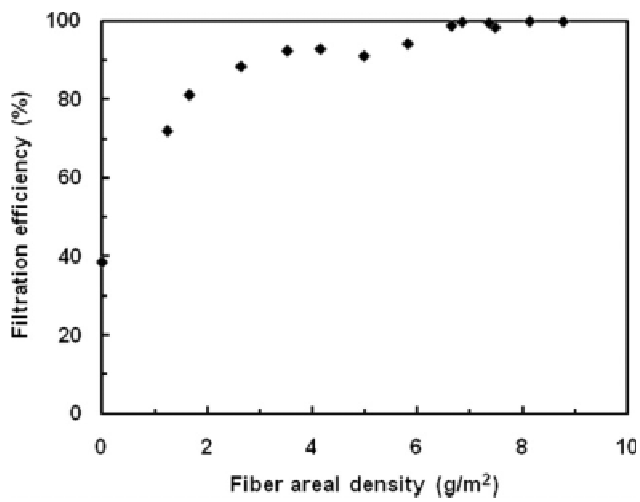

Figure 3 Filtration efficiency of electrospun nylon 6 fiber mats on 50/50 nylon/cotton fabric with different fiber areal density.

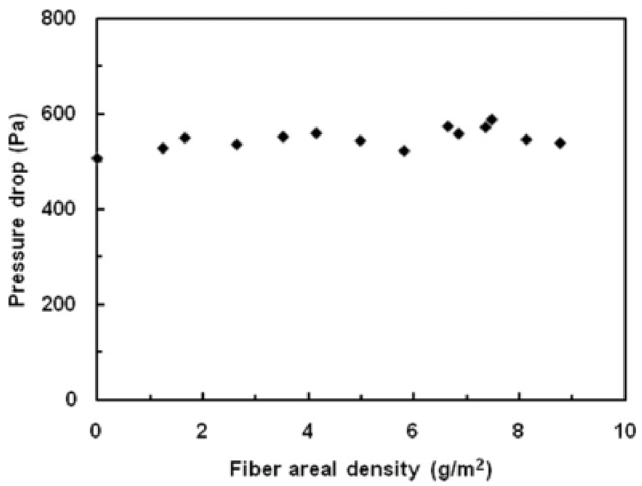

Figure 4 Pressure drop of electrospun nylon 6 fiber mats on 50/50 nylon/cotton fabric with different fiber areal density.

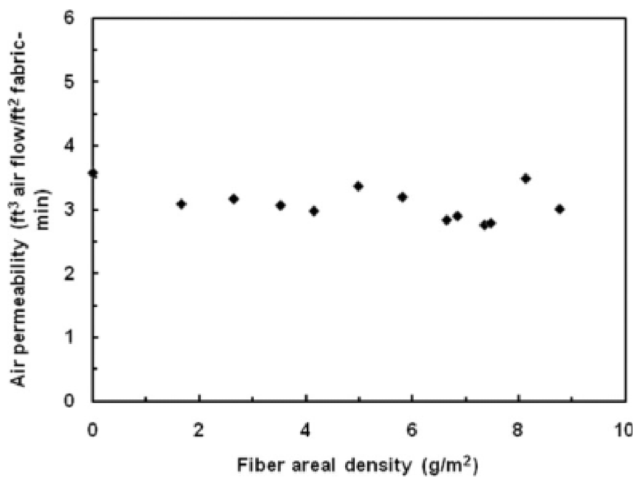

Figure 5 Air permeability of electrospun nylon 6 fiber mats on 50/50 nylon/cotton fabric with different fiber areal density.

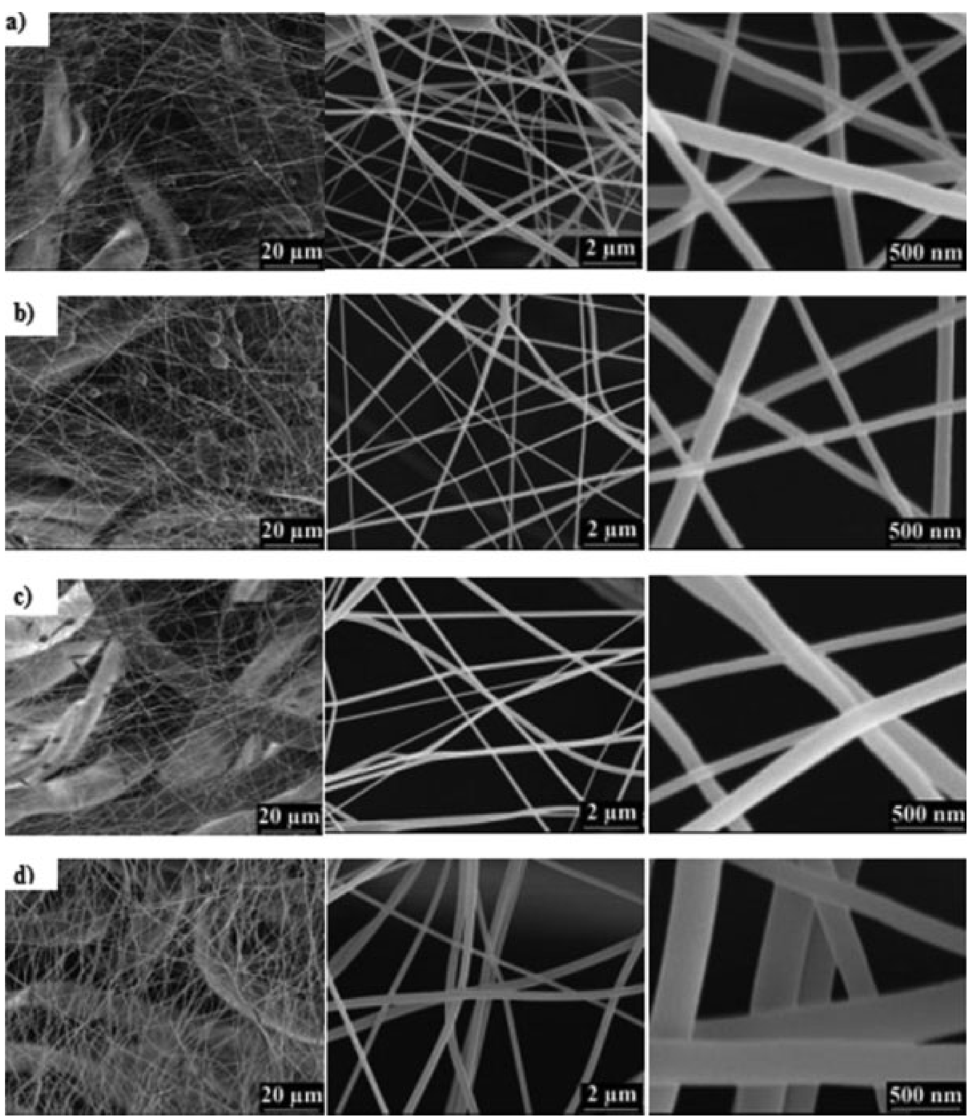

Figure 6 SEM images of electrospun nylon 6 fiber mats produced with different solution concentration: (a) 10%, (b) 11%, (c) 12%, and (d) 13% wt/vol.

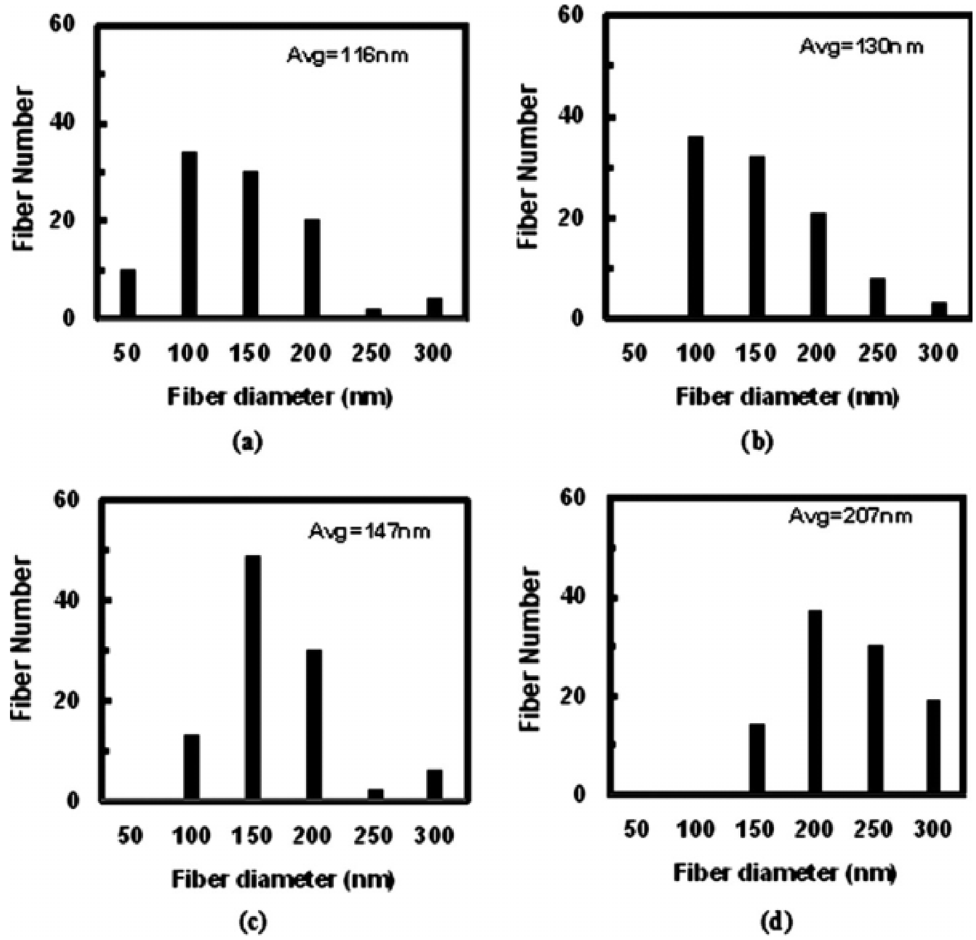

Figure 7 Fiber diameter distribution of electrospun nylon 6 fiber mats produced with different solution concentration: (a) 10%, (b) 11%, (c) 12%, and (d) 13% wt/vol.

density = 0), the filtration efficiency of the 50/50 nylon/cotton fabric is only 38%, indicating most of the challenging particles penetrate through the fabric. Depositing a small amount of electrospin nylon 6 fibers significantly increases the filtration efficiency. For example, when the fiber areal density is 1.2 g/m^2, the filtration efficiency increases to 72%, i.e., a 90% increase in efficiency. Considering that the areal density of the unmodified substrate nylon/cotton is 250 g/m^2, the deposition of 1.2 g/m^2 of electrospun fibers leads to a weight increase of only 0.5%.

With further increase in fiber areal density, the filtration efficiency continues to increase and an efficiency greater than 99.5% can be obtained when the areal density is between 6.5 g/m^2 and 8.5 g/m^2, corresponding to a weight increase of $<$3.4%. The filtration efficiency can be further improved by optimizing the structure of electrospun fiber mats, as discussed in the following sections.

One major challenge in developing protective materials is to maintain low pressure drop and high air permeability while increasing the filtration efficiency. Many filtration media provide high efficiency by sacrificing pressure drop or air permeability. Figures 4 and 5 show that the pressure drop and air permeability of electrospun nylon 6 fiber-deposited fabrics with different areal densities are not significantly decreased as compared with the unmodified substrate fabric. Therefore, electrospun fiber mats can provide high filtration efficiency without sacrificing pressure drop and air permeability.

In the following sections, the effects of solution concentration and electrospinning voltage on the structure and filtration efficiency of nylon 6-deposited fabrics are discussed using fabrics with a small areal density (such as 1.2 or 1.7 g/m^2) because the filtration efficiency is sensitive to electrospun fiber mat structure at this low deposition amount.

Effect of solution concentration on electrospun fiber mat morphology and filtration performance

Electrospun nylon 6 fiber mats were deposited on 50/50 nylon/cotton fabric from solutions with different concentrations under an electrospinning voltage of 10 kV. Figure 6 shows SEM images of these electrospun fiber mats, and their fiber distributions are shown in Figure 7. It is seen that with increase in

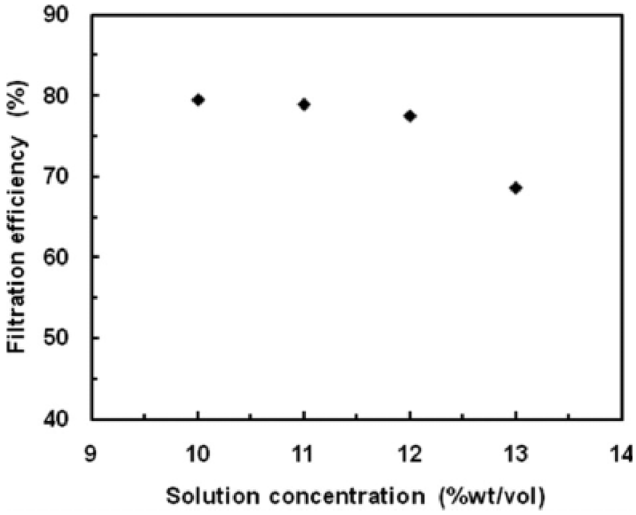

Figure 8 Filtration efficiency of electrospun nylon 6 fiber mats prepared using different concentration of solution.

solution concentration, fiber diameter increases. This is a result of increased solution concentration, which in turn leads to increased solution viscosity and resistance to jet stretching during whipping, thereby resulting in thicker fiber diameter.[14–16] From Figure 6, it was seen that beads are formed at lower solution concentrations of 10% and 11% wt/vol, although these solutions produce electrospun fibers with smaller diameters. The formation of beads might be caused by insufficient chain entanglements at low concentrations.[17–19] When the solution concentration increases to 12% and 13% wt/vol, the solution viscosity and chain entanglement density increase and favor the formation of smooth electrospun fibers without beads.

Figure 8 shows the effect of solution concentration on the filtration efficiency of electrospun nylon 6 fiber mats deposited on nylon/cotton fabric. These mats have the same areal density, i.e., 1.2 g/m^2. In

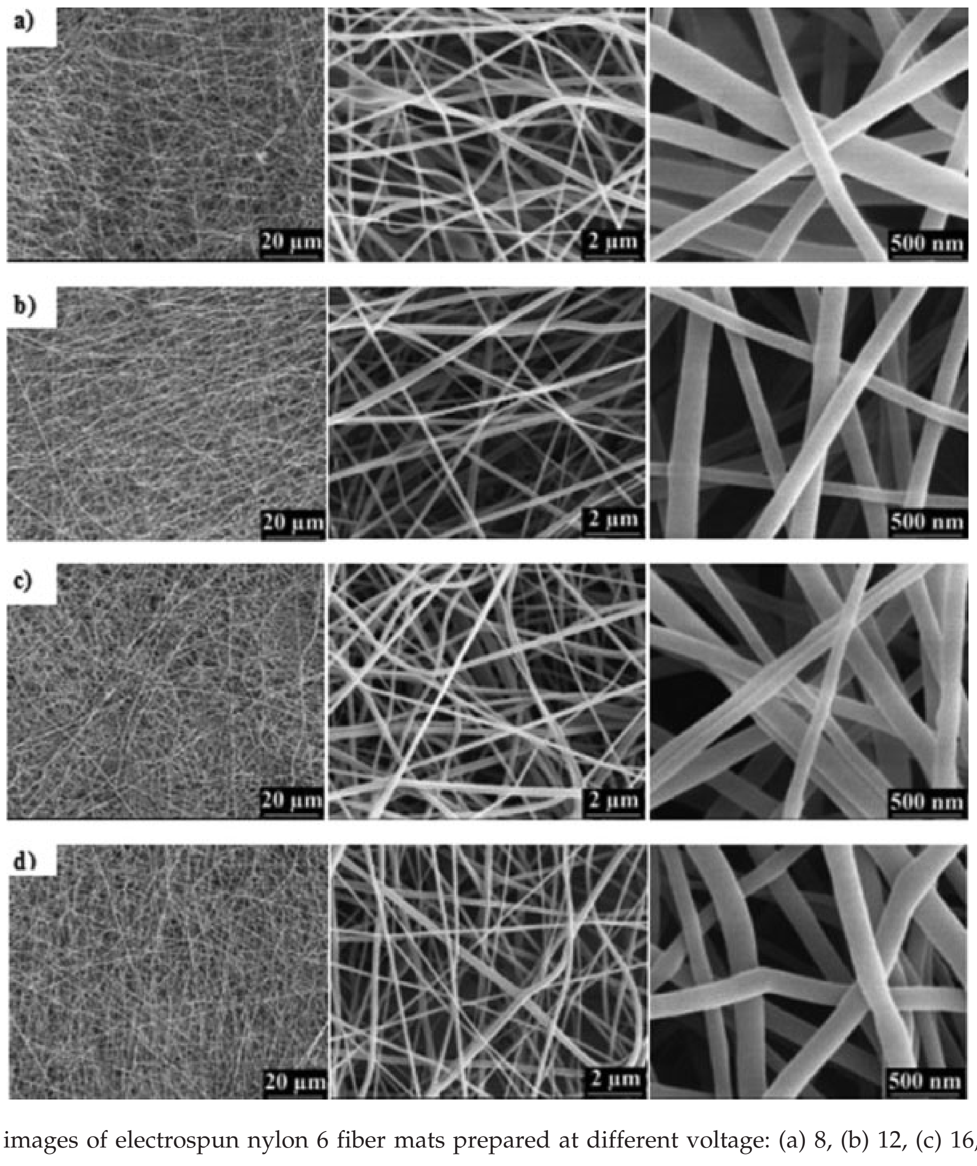

Figure 9 SEM images of electrospun nylon 6 fiber mats prepared at different voltage: (a) 8, (b) 12, (c) 16, and (d) 20 kV.

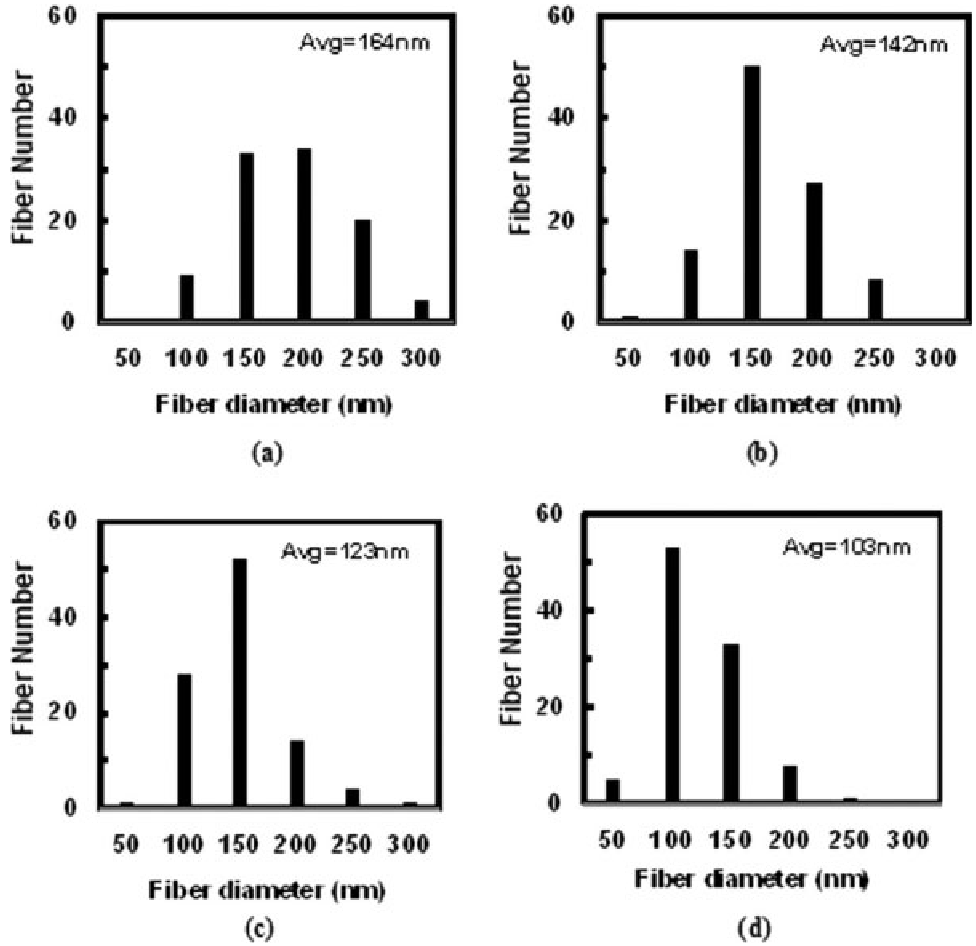

Figure 10 Fiber diameter distribution of electrospun nylon 6 fiber mats prepared at different voltage: (a) 8, (b) 12, (c) 16, and (d) 20 kV.

general, as fiber diameter increases, the filtration efficiency decreases.[13,20] The filtration efficiencies of electrospun mats produced from 10%, 11%, and 12% wt/vol solutions are greater than 75% due to their smaller diameters, but the mat efficiency is lower than 70% when the solution concentration is 13%.

Effect of electrospinning voltage on fiber morphology and filtration performance

Another crucial parameter in fiber diameter control is the voltage between the spinneret and collector. In general, a voltage higher than 6 kV is able to cause the polymer solution drop at the tip of the needle to distort into the shape of a Taylor Cone during the initiation of electrospinning.[21] Previous studies of the effect of voltage on electrospun fiber morphology have reported conflicting results. Some researchers reported that a higher voltage leads to enhanced stretching of the solution due to the greater columbic forces in the jet as well as the stronger electric field.[22–26] As a result, the fiber diameter becomes smaller at higher voltages. In addition, when a solution of lower viscosity is used, a higher voltage favors the formation of secondary jets, which in turn also causes reduced diameter. However, some other reports show that larger fiber diameters are obtained at higher voltages.[27] This is probably because the flight time for the fibers to stretch and elongate is reduced at high voltages. There are also reports showing that fiber diameter does not change significantly when the electrospinning voltage changes.[28–30] Therefore, in order to obtain electrospun fiber mats with desired diameter and performance, electrospinning voltage must be carefully controlled to obtain a proper balance among electric field strength, jet flight time, and jet stability, etc.

Figures 9 and 10 shows SEM images and fiber diameter distributions of electrospun nylon 6 fiber mats deposited on nylon/cotton fabric from 12% wt/vol solution using different voltages. The deposition areal density is 1.7 g/m^2. It is seen that with increase in electrospinning voltage, the fiber diameter decreases gradually. Therefore, in this work, higher applied voltage enhances polymer jet extension, resulting in finer fiber diameter.

Figure 11 shows the effect of electrospinning voltage on the filtration efficiency of electrospun nylon 6

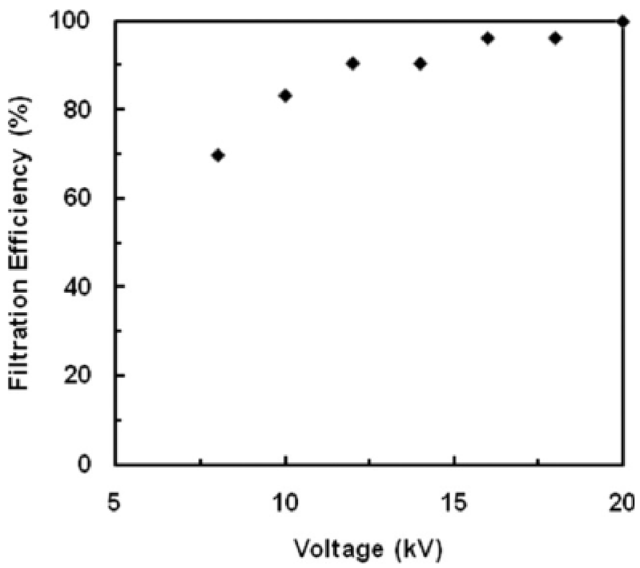

Figure 11 Filtration efficiency of electrospun nylon 6 fiber mats prepared at different voltage.

fibers on 50/50 nylon/cotton fabric. When the spinning voltage was increased from 8 to 20 kV, the filtration efficiency of electrospun fiber mats increased continuously. Hence, increasing spinning voltage yields a corresponding enhancement in filtration efficiency.

CONCLUSIONS

Electrospun nylon 6 fiber mats were deposited on 50 : 50 nylon : cotton woven fabrics with varying processing parameters such as solution concentration, electrospinning voltage, and deposition time (or areal density). It was found that depositing ultrathin nylon 6 fiber mats can significantly improve the filtration efficiencies without sacrificing pressure drop and air permeability. The effect of electrospinning conditions, such as applied voltage and solution concentration, on the filtration efficiency was studied. It was observed that the filtration efficiency increased with decreasing solution concentration and increasing applied voltage. Filtration efficiency of the fabric was increased by a maximum of more than 250% after the deposition of electrospun fiber mats. Electrospun fiber mats promise to be highly effective components of high performance barrier fabric materials.

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