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# **Modification Of Air Filter Media With Nylon-6 Nanofibers**

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## **Abstract**

Nylon-6 fibers with average diameters below 500 nm were electrospun onto conventional air filter media at varying weight coverage levels using a multi-nozzle bank. The initial filtration efficiency of the air filter media was improved significantly with increasing coverage level and decreasing size of nylon-6 fibers. Nylon-6 fibers were very durable on the air filter media at the coverage level of 0.1 g/m<sup>2</sup> due to the good adhesion with the air filter fibers. The production efficiency of the coated air filter media could be increased by using more nozzle banks for electrospinning without affecting the initial filtration efficiency of the air filter media significantly.

## **Introduction**

Electrospinning applies high voltages to droplets of polymer solutions or melts to overcome the liquid surface tension and enable the formation of ultra-fine fibers with the average diameter in the sub-micron range [1,2]. Fiber formation by electrospinning of polymer solutions has been extensively studied in terms of solution concentration [3-5], viscosity [4,6], conductivity [4,7,8], humidity [9], and voltage [6]. Although electrospinning has been widely utilized as a cheap and straightforward method to produce polymer nanofibers on the laboratory scale, the production efficiency is very low due to the low feeding rate of electrospinning process [4]. In order to improve the fiber production rate and in some cases to make multi-component fibers, multi-jet electrospinning has been applied [10] and modeled mathematically [11].

One of the earliest and most important applications of electrospinning is to improve the filtration efficiency of the conventional filter media. Due to the small fiber and inter-fiber pore sizes and large surface areas, electrospun polymer nanofibers have been used in a number of commercial filtration applications, such as coalescence filtration [12,13] and air filtration [14-16], for over more than 20 years. Electrospun polymer nanofibers were added to the slurry of glass fibers with an acrylic binder solution at pH around 2 followed by vacuum filtration and high temperature drying to prepare coalescence filter media [13]. Shin et al. has shown that glass-fiber filter media with small amount of electrospun polymer nanofibers exhibited much higher separation efficiency with increased pressure drop [13,17,18]. Wang et al. reported the preparation of a filtration medium for oil/water emulsion separation with high filtration flux by using a crosslinked electrospun poly(vinyl alcohol) fibrous substrate as a mid-layer [19]. To modify air filter media, electrospun polymer nanofibers were usually deposited on the air filter media directly. Filtration theory generally assumes a continuous air flow around the air filter fibers, which is less applicable when the fiber sizes become comparable to the mean free path of air molecules [16,20]. The importance of the molecular movement of air molecules at the fiber surface to the overall flow field can be described by the Knudsen number,  $K_n = \lambda/r_f$ , where  $\lambda$  and  $r_f$  are the gas mean free path and the fiber radius, respectively [16]. Graham et al. reported an empirical value of  $K_n$ , 0.25, above which the slip flow at the fiber surface must be considered [16]. In other words, when the fiber diameter is smaller than 520 nm the air velocity at the fiber surface is considered to be non-zero based on the mean free path of air molecules, 0.066 micrometers, under the standard conditions. This makes the portion of air flowing near the fiber surface larger than that in the case of non-slip flow and more particles travel near the fiber resulting in higher diffusion, interception and inertial impaction efficiencies [16]. Hajra et al. reported that the filtration performance of glass-fiber filter media was not improved by the addition of conventional polymer fibers with average diameter of 7  $\mu\text{m}$  but significantly enhanced by the similar masses of nanofibers with average diameters of 150 nm [12]. Polyamides such as nylon 6 [13,18] and Nomex [12,14] are among the most widely used polymers that have been electrospun into ultra-fine fibers and applied for conventional filtration purposes due to their good mechanical properties. In addition to the applications in traditional filtration, electrospun polymer nanofibers with immobilized specific ligands have also been applied as affinity membrane to permit the purification of molecules based on their physical/chemical properties or biological functions [21].

The main aim of this research is to investigate the filtration performance of commercial air filter media coated with electrospun nylon 6 nanofibers. The goal is to experimentally study the effects of average diameter and mass coverage level of nylon 6 fibers on the initial filtration efficiency, pressure drop and durability of air filter media. In order to improve the production efficiency of nylon 6 electrospun nanofibers a multi-nozzle electrospinning set-up was used. The effects of electric field interactions on the deposition of nylon 6 nanofibers and the resulting filtration efficiency of air filter media were experimentally explored.

## Experimental

### Materials.

Nylon-6 and 88% formic acid were purchased from Aldrich and VWR, respectively. Cellulose-based air filter media was provided by Clark Filter Inc. The thickness of air filter media is about 0.3 mm and the test area in the air filtration efficiency tester is 25.4 by 25.4 cm.

### Solution Preparation and Electrospinning.

Homogeneous nylon-6 solutions were prepared in 88% formic acid at room temperature by gently stirring for 8 hours. The electrospinning was performed using a nozzle-bank, which allowed the nylon-6 solution to be delivered into thirteen nozzles at the same time (Figure 1). Each nozzle had a 0.4mm inner diameter and nozzles spacing was one inch on center. The nylon solution was fed at a rate of 0.1 ml/hr-nozzle using a syringe pump (Model 100 kdScientific) and the tip-to-collector distance was 14 cm unless stated differently. A high voltage power supply (Gamma High Voltage Supply, ES 30-0.1P) was connected to the nozzle bank. The electrospinning voltage was about 29 kV. A piece of aluminum foil, black paper or moving air filter media were used as the collectors. A grounded aluminum plate was used to support the collectors and attract nylon fibers to the non-conductive collectors (black paper and air filter media). The rate of motion of the air filter media was controlled by a computer to achieve the desired coverage level of nylon-6 fibers.

### Measurement and Characterization.

The zero-shear-rate viscosity of nylon-6 solutions was measured using a Rheometrics AR 2000 rheometer. The solution conductivity and surface tension were determined with a conductivity meter (VWR International) and a microbalance, respectively. The morphology of electrospun nylon-6 fibers was observed with a scanning electron microscope (SEM, LEICA 440). The fibers were coated with gold and observed under 25 kV accelerating voltage. About 100 electrospun nylon-6 fibers and 40 air filter cellulose fibers were measured using an image analysis software (Scion Image, NIH Image software) to obtain the fiber size distribution. The initial filtration efficiency of the coated air filter media was investigated using a flat-sheet tester. Dust particles with average sizes of 0.3  $\mu\text{m}$ , 0.5  $\mu\text{m}$ , 0.7  $\mu\text{m}$ , 1  $\mu\text{m}$ , 2  $\mu\text{m}$ , and 5  $\mu\text{m}$  were pulled into the filtration efficiency tester at the air face velocity of 0.3 meter/min and counted at both upstream and downstream of the air filter media using a laser particle counter.

The volume,  $V$  (ml), of nylon-6 solution needed to achieve a desired coverage level on the moving air filter media was calculated by equation 1:

$$V = \frac{CA \times CL}{C \times d} \quad (1)$$

where CA and CL are the coverage area (m<sup>2</sup>) and coverage level (g/m<sup>2</sup>), respectively. C and d are the concentration and density of nylon solution, which are 0.2 and 1.20 g/ml, respectively. The motor speed (MS m/min) could be then calculated by equation 2.

$$MS = \frac{L}{V \times 60(\text{min/hr})} \quad (2)$$

where L and r are the length of air filter media (m) and solution feeding rate (ml/hr), respectively. L was calculated by dividing the coverage area with the width of air filter media, 0.3 m.

## Results and Discussion

### Nylon fiber size and morphology.

The properties of nylon-6 solutions with concentration ranging from 15wt% to 30wt% were summarized in Table 1. The solution viscosity increased with increasing solution concentration but solution conductivity decreased gradually. The solution concentration did not show obvious effect on solution surface tension. Since low solution viscosity and high conductivity favor thinner electrospun fibers [4,8], the average diameters of electrospun nylon 6 fibers decreased with decreasing solution concentration. However, a large number of bead structures were observed along the fibers electrospun from the 15% solution due to the less molecular entanglement (Figure 2) [4]. The average diameters of nylon-6 fibers increased from 120 nm to 300 nm and 700 nm as solution concentration increased from 20% to 25% to 30% (Figure 3).

At the feeding rate of 0.1 ml/hr-nozzle the electrospinning of nylon solutions with concentration of 20% or lower could be sustained for several hours but was interrupted frequently if the concentration was 25% or higher due to the high solution viscosity. The relatively broader fiber size distribution at high solution concentration might be caused by the instability of electrospinning. At a higher feeding rate, such as 0.3 ml/hr-nozzle, solution fed to the nozzle tips was more than that could be drawn into solution jets by the applied electric field. As a result, the solution started to drip from the nozzle tips.

Similar fiber sizes and morphology were observed when either conductive or non-conductive materials were used as collector. The aluminum plate placed behind the non-conductive collectors helped to attract the charged nylon fibers. Varying the needle tip-to-collector distance from 7 to 14 cm did not result in any observable effects on fiber size and morphology, which indicated that there was no further draw down of the fibers beyond 7 cm collector distance. Since fibers with diameters smaller than 500 nm are expected to favor higher filtration efficiency due to the slip-slow mechanism at the fiber surface [16], the 20% and 25% nylon solutions were used for the rest of study.

## **Adhesion between nylon-6 and air filter media fibers**

Adhesion between air filter media fibers and nylon-6 electrospun fibers was observed (Figure 4), which is desirable for good durability of nylon-6 fibers during the use of air filter media. The adhesion could be caused by the swelling effects of the residual aqueous formic acid solvent in the electrospun nylon-6 fibers on the air filter media fibers. Although the swelling of the air filter media fibers by the residual solvent of formic acid in the electrospun nylon 6 fibers might not be so obvious, it might be significant enough for nylon 6 nanofibers to bind with air filter media fibers after the deposition during the electrospinning since the nylon fibers are so small. The adhesion between the air filter fibers and electrospun nylon-6 fibers could be further improved by treating the air filter media with formic acid vapor for about 30 min after the electrospinning (Figure 5). However, the nylon-6 fibrous membrane was broken at some areas by the formic acid vapor. More detailed investigation is being conducted to improve the adhesion without destroying the nylon-6 electrospun fibers by pre- and/or post-treatment of air filter media with the formic acid and other solvents.

## **Filtration Performance of Modified Air Filter Media**

Typical air filter media used for coarse removal of dust particles from air have poor initial filtration efficiency. They are ineffective in removing small particles especially in the range from about 100 to 800 nm until a certain amount of particles are collected on the filter surface [14]. In order to improve the initial filtration efficiency, air filter media were coated with nylon-6 fibers with different average fiber diameters at several weight coverage levels. The difference of packing density of nylon-6 electrospun fibers at different weight coverage levels was shown in Figure 6. The initial filtration efficiency of the air filter media coated with nylon-6 nanofibers at the coverage level of 0.03, 0.06, 0.1 and 0.5 g/m<sup>2</sup> was improved (Figure 7) when compared with the original air filter media. The improvement was particularly significant for particles of less than 1 μm. The largest particle (5.0 μm) could be removed 100% even by the original air filter media. The durability of nylon-6 fibers at lower coverage levels was much better than that at higher coverage levels due to the better adhesion between nylon 6 fibers and air filter media fibers. For example, nylon-6 fibers could be easily peeled off from the air filter media at 0.5 g/m<sup>2</sup> coverage level but not at all at the coverage level of 0.1 g/m<sup>2</sup> or lower. The filtration efficiency of the air filter media coated with nylon-6 nanofibers at 0.1 g/m<sup>2</sup> was still very high even after the media was scratched heavily many times (Figure 8). The good durability of nylon-6 electrospun fibers at the weight coverage level of 0.1 g/m<sup>2</sup> or lower makes it possible for the modified air filter media to survive the handling and processing process in the use without losing the high filtration efficiency.

The diameter of the electrospun nylon-6 fibers showed a great effect on the filtration efficiency of the coated air filter media. Comparison between Figures 7 and 9 shows that an increase in average fiber diameter from 120 to 300 nm significantly reduces the filtration efficiency at all levels of mass coverage of nanofibers. The decrease in filtration efficiency with increasing nylon-6 fiber sizes is because there are fewer fibers electrospun from the same amount of polymer if the fibers are larger. If we assume that the nylon fibers are cylindrical, then increasing

the fiber diameter from 120 nm to 300 nm will cause the total fiber length to decrease by 6.25 times. As a result, the larger nylon fibers will result in fewer fibers per unit area at the same weight coverage level. This was consistent with the SEM observation (Figure 10 and Figure 6 c). The length of 120 nm nylon-6 fibers at the coverage level of  $0.08 \text{ g/m}^2$  should be similar as that of 300 nm nylon-6 fibers at the coverage level of  $0.5 \text{ g/m}^2$ . By plotting and interpolating the filtration efficiency for different particle sizes over the range of coverage levels using the data in Figure 7, the filtration efficiency of the air filter media coated with 120 nm nylon-6 fibers at the coverage level of  $0.08 \text{ g/m}^2$  could be estimated and was compared with measured filtration efficiency for air filter media coated with 300 nm nylon-6 fibers at the coverage level of  $0.5 \text{ g/m}^2$  (Figure 9). The similar filtration efficiency shown in Figure 11 indicated that the initial filtration efficiency of the air filter media coated with nylon-6 fibers with the average diameters of 120 and 300 nm was determined by the total length of nylon-6 fibers. Although at the same nylon 6 fiber length, dust particles have more chances to directly hit the 300nm nylon 6 fibers, the slip flow effects are more significant for 120nm nylon 6 fibers, which can improve the filtration efficiency for the dust particles.

In order to investigate how the dust particles interact with the nylon-6 electrospun fibers coated on the air filter media, the porosity of the original air filter media and the one coated with nylon-6 fibers at the coverage level of  $0.1 \text{ g/m}^2$  was measured using a capillary flow porometer (CFP-1100-AEHXL, PMI Inc.). During the porosity measurement, the air filter media was completely saturated with a liquid with very low surface tension to make all the pores of the air filter media to be covered by the liquid. When the applied air pressure exceeds the capillary attraction of the liquid in the pores, air will pass through the sample. Smaller pores have a higher capillary attraction than larger pores and thus smaller pores open up at higher pressures. Bubble point is the point when the largest pore is opened up by the air. So the bubble point pore diameter is the largest pore diameter of the sample. The porosity of air filter media was summarized in Table 2. The original air filter media exhibited larger mean pore size,  $11.1 \mu\text{m}$ , which decreased to about  $8.3 \mu\text{m}$  for the air filter media coated with electrospun nylon-6 nanofibers at the coverage level of  $0.1 \text{ g/m}^2$ . The coated air filter media also showed significantly larger amount of small pores, i.e. about 13.9% of pores are around  $2.1 \mu\text{m}$  in diameters, which is much smaller than those of the original air filter media. On the other hand, the bubble point pore diameter or the largest pore diameter was very similar, around  $31 \mu\text{m}$ , for both types of air filter media, which indicated that the air filter media was only coated very lightly at the weight coverage level of  $0.1 \text{ g/m}^2$ . The typical pore size distribution for original and coated air filter media was shown in Figure 12. The pore size was evenly distributed from about 4 to  $18 \mu\text{m}$  for the original air filter media with the smallest pores around  $2 \mu\text{m}$ . The coated air filter media showed a bimodal pore size distribution with many more small pores around  $2 \mu\text{m}$ . Since most of the particles tested in this research were smaller than the inter-fiber pores of the air filter media, the single-fiber mechanism should be the dominant capture mechanism for the improved initial filtration efficiency of the coated air filter media [14], which is consistent with the result that the initial filtration efficiency was proportional to the length of electrospun nylon-6 fibers.

The filtration performance of air filter media is evaluated not only by the initial filtration efficiency but also the pressure drop. Low pressure drop is desired to minimize the operating cost for the air filter media. The initial pressure drop over the air filter media coated with electrospun nylon-6 fibers was not increased obviously especially at the low weight coverage levels or small

fiber length when compared with the initial pressure over the original air filter media, 0.31 inch H<sub>2</sub>O (Figure 13). It suggests that coating the conventional air filter media with small amount of ultra-fine electrospun nylon-6 fibers is a very effective way to improve the filtration performance of the air filter media.

In order to improve the production efficiency of the coated air filter media, instead of one nozzle bank as shown in Figure 1, two nozzle banks separated at about 8 cm and 24 cm were utilized to electrospin nylon-6 fibers. As a result, the moving speed of the original air filter media during the electrospinning process could be doubled to achieve the same weight coverage level. The deposition of electrospun nylon-6 fibers were studied by using a piece of black paper as the collector at an extended electrospinning time of 15 min. At the smaller separation distance of 8 cm between the two nozzle banks, the interactions between the similarly charged nylon-6 solution jets caused instable electrospinning especially for those nozzles located in the middle of each nozzle bank probably due to the stronger electric field interactions at those locations. The electric field interactions caused lighter coverage of nylon-6 fibers electrospun from those middle nozzles and a larger separation distance of 13 cm between the nylon fiber deposition spots from each nozzle bank than the distance between the two nozzle banks of 8 cm (Figure 14 a). When the two nozzle banks were separated at 24 cm, the distance between the nylon fiber deposition spots from each nozzle bank was also about 24 cm, which indicated that the interactions between the similarly charged nylon solution jets from each nozzle bank was minimized at this nozzle bank separation distance (Figure 14 b). At the same weight coverage level of 0.1 g/m<sup>2</sup>, the filtration efficiency of the air filter media coated with nylon-6 fibers electrospun from the two nozzle-bank set-up was slightly lower when compared with the case in which only one nozzle bank was used (Figure 15), which might be due to the increased instability of electrospinning caused by the electric field interactions when more nozzle banks were used. More investigation will be conducted to mathematically simulate the electric field interactions between the similarly charged nylon solution jets under current multiple-nozzle-bank set-up and to study the effects of such electric field interactions on the deposition of nylon-6 fibers and filtration efficiency of the coated air filter media.

## Conclusions

Nylon-6 fibers with average diameters ranging from 120 to about 700 nm could be electrospun from nylon-6 solutions in 88% formic acid. The fiber size and size distribution was mainly affected by solution concentration. The filtration efficiency of the air filter media could be improved significantly by coating with nylon fibers at the weight coverage level ranging from 0.03 to 0.5 g/m<sup>2</sup>. The filtration efficiency increased with increasing coverage level and decreasing nylon fiber sizes at the same weight coverage level. On the other hand, the filtration efficiency is similar for air filter media coated with 120nm and 300nm nylon 6 fibers at the similar nylon-6 fiber length. Nylon-6 fibers were very durable on the air filter media at weight coverage level of 0.1g/m<sup>2</sup> or lower due to the sufficient adhesion between the nylon-6 fibers and air filter media fibers.



Our results also showed that the interactions between the similarly charged nylon solution jets during electrospinning affected the deposition of nylon fibers. At the same weight coverage level, the filtration efficiency of the air filter media coated with nylo-6 fibers electrospun using a single nozzle bank was slightly higher when compared with the case in which two nozzle banks were used.

## Acknowledgement

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Table 1. Solution properties and electrospun nylon fiber diameter.

Concentration (wt%)	Viscosity (Pa*s)	Conductivity (mS/cm)	Surface tension (mN/m)	Fiber size (nm)
15	0.5	4.62	41.21	-
20	1.5	4.30	42.33	120
25	2.4	3.55	42.57	300
30	8.7	2.90	41.44	700

Table 2. Porosity of air filter media before and after coated with electrospun nylon-6 fibers

Sample	Mean pore diameter ( $\mu\text{m}$ )	Fraction of pores at mode size, (%)	Diameter of mode size ( $\mu\text{m}$ )	Bubble point pore diameter ( $\mu\text{m}$ )
Original air filter	11.1	4.6	9.3	31.4
Air filter media coated at $0.1\text{g/m}^2$	8.3	13.9	2.1	30.1

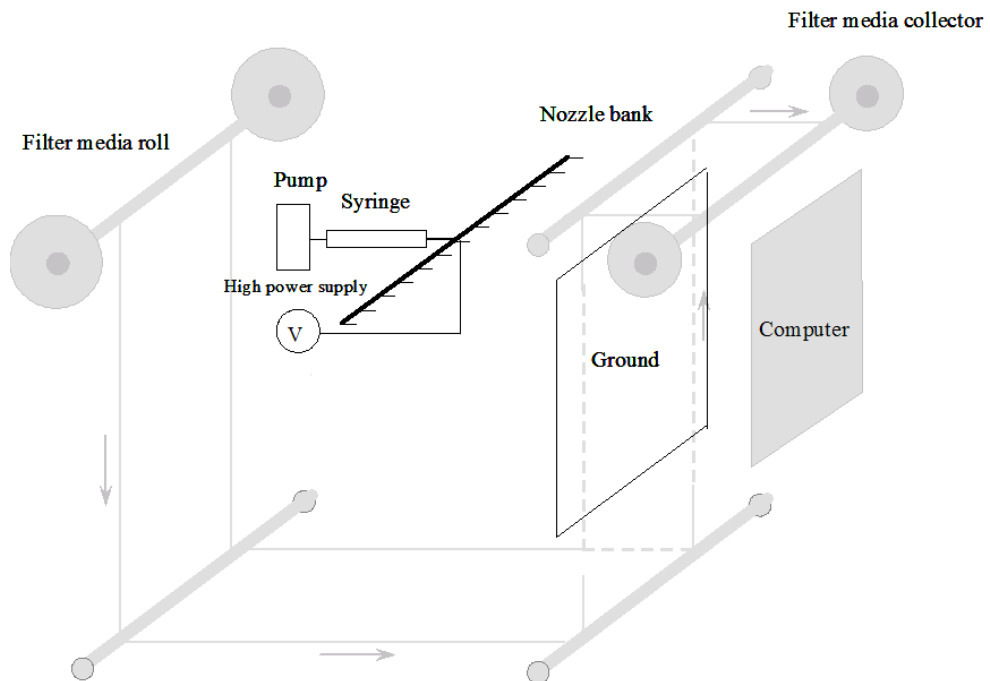


Figure 1. Electrospinning setup with two nozzle banks. The shaded part is the collecting system of coated air filter media and the arrows indicate the rolling direction of the media.

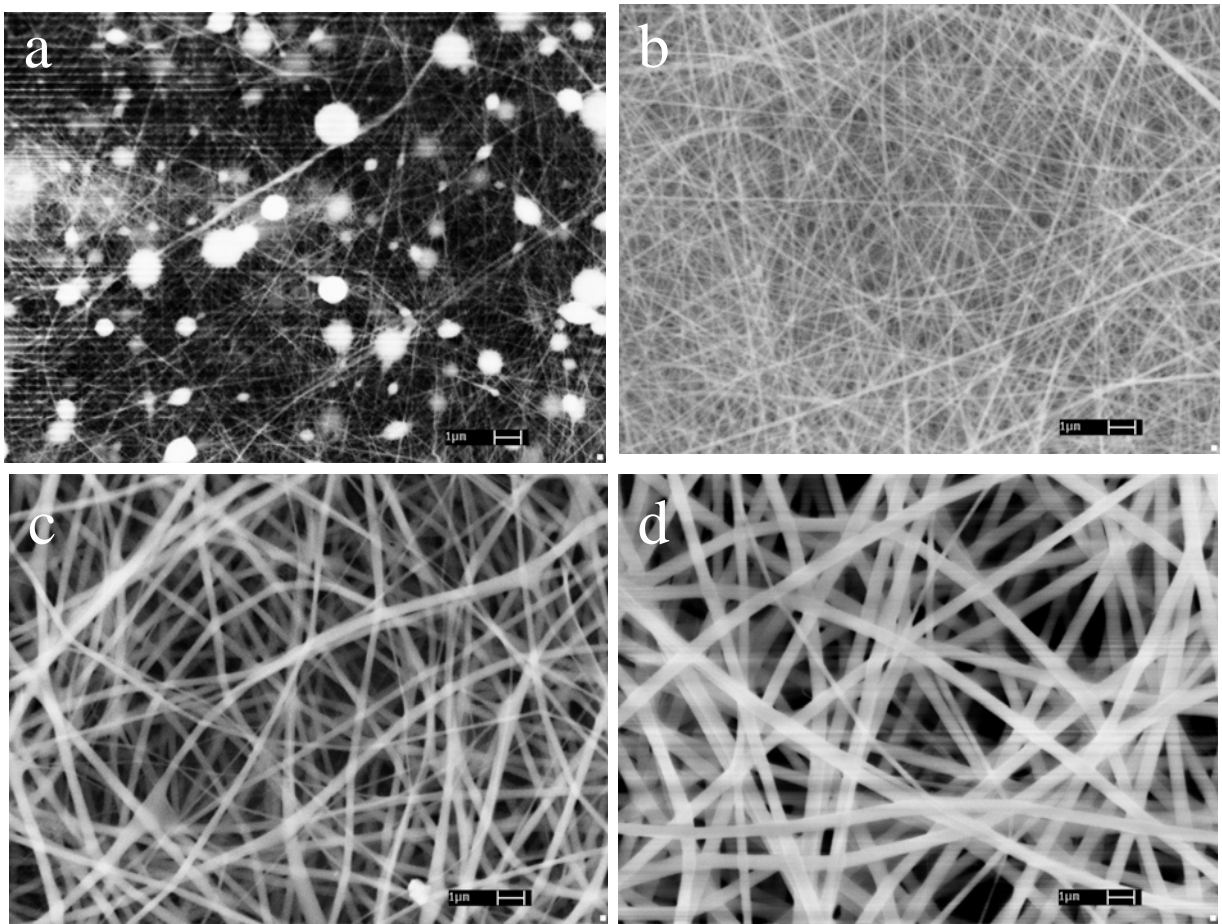


Figure 2. SEM of nylon-6 fibers electrospun from (a) 15%; (b) 20%; (c) 25%; and (d) 30% solution. The magnification bar is 1  $\mu\text{m}$  in the images.

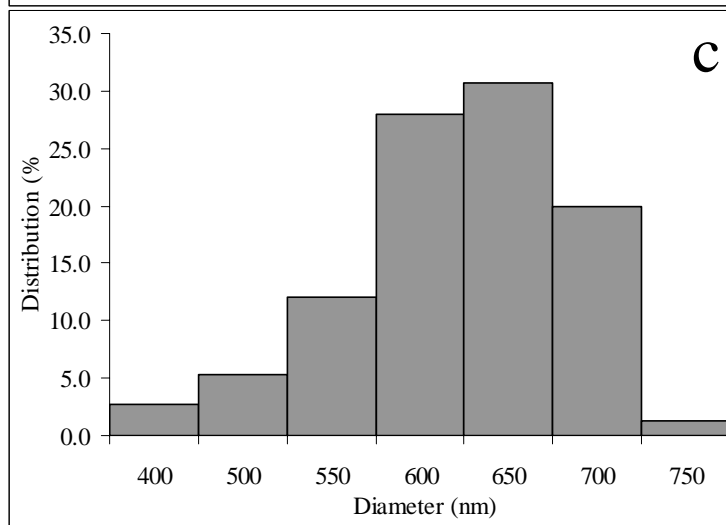
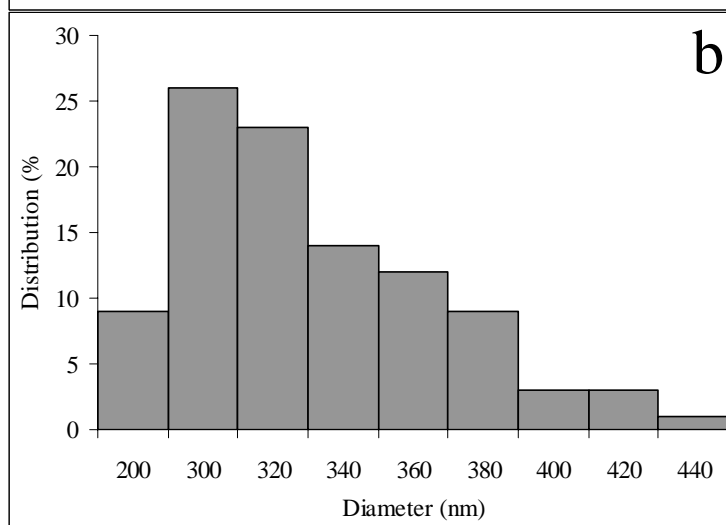
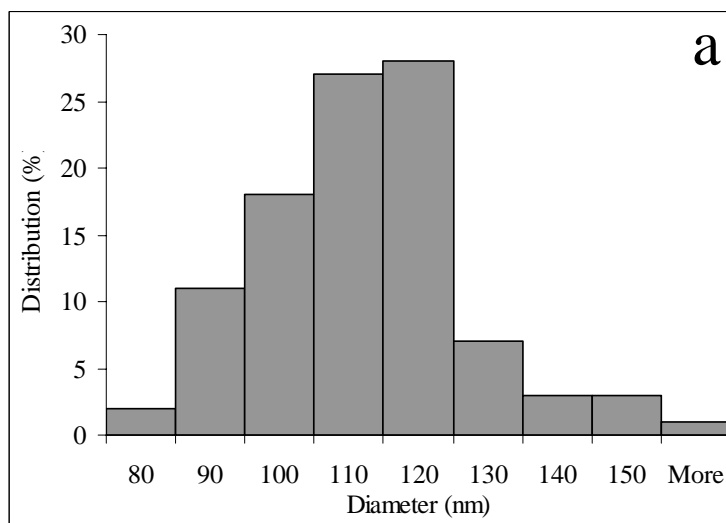


Figure 3. Distribution of fiber diameters electrospun from (a): 20%; (b): 25% and (c): 30% nylon-6 solutions

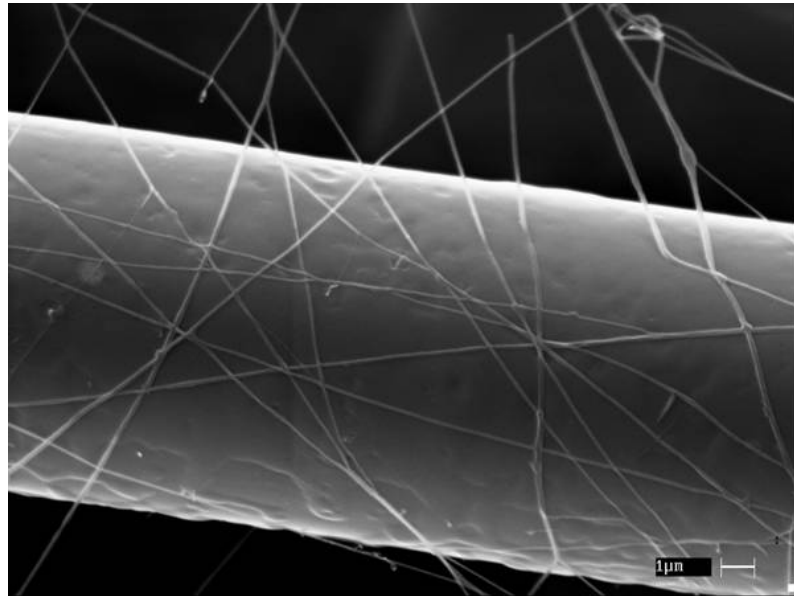


Figure 4. Adhesion between air filter media fiber and nylon-6 electrospun fibers.

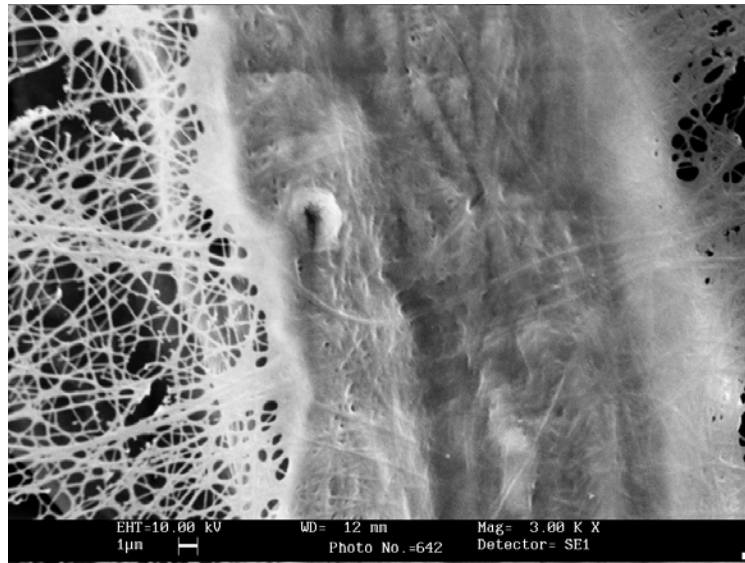


Figure 5. Adhesion between air filter fiber and electrospun nylon-6 fibers treated with formic acid vapor after electrospinning

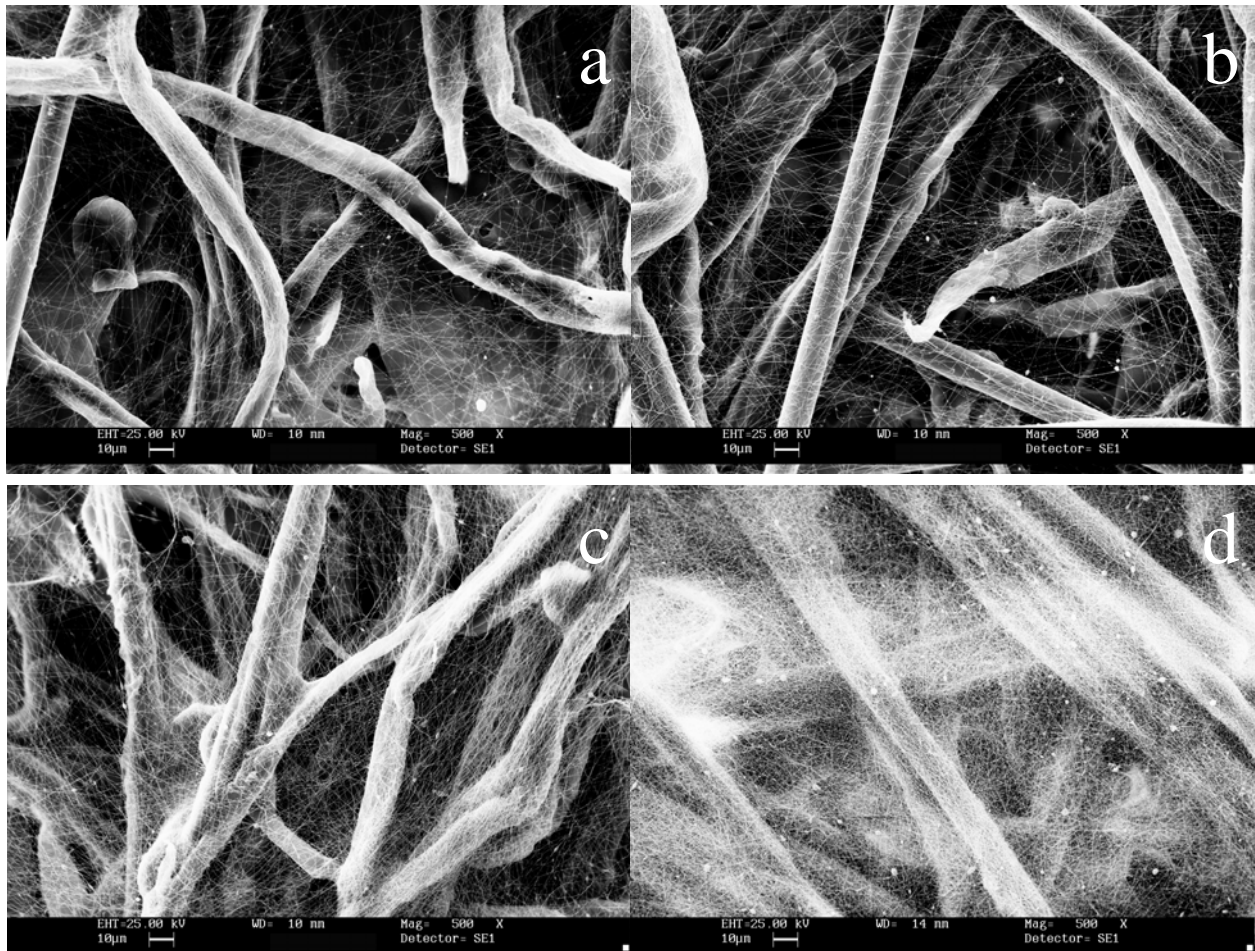


Figure 6. SEM images of air filter media coated with nylon-6 electrospun fibers (average diameter = 120 nm) at the weight coverage levels of (a): 0.03 g/m<sup>2</sup>; (b): 0.06 g/m<sup>2</sup>; (c): 0.1 g/m<sup>2</sup>; (d): 0.5 g/m<sup>2</sup>.

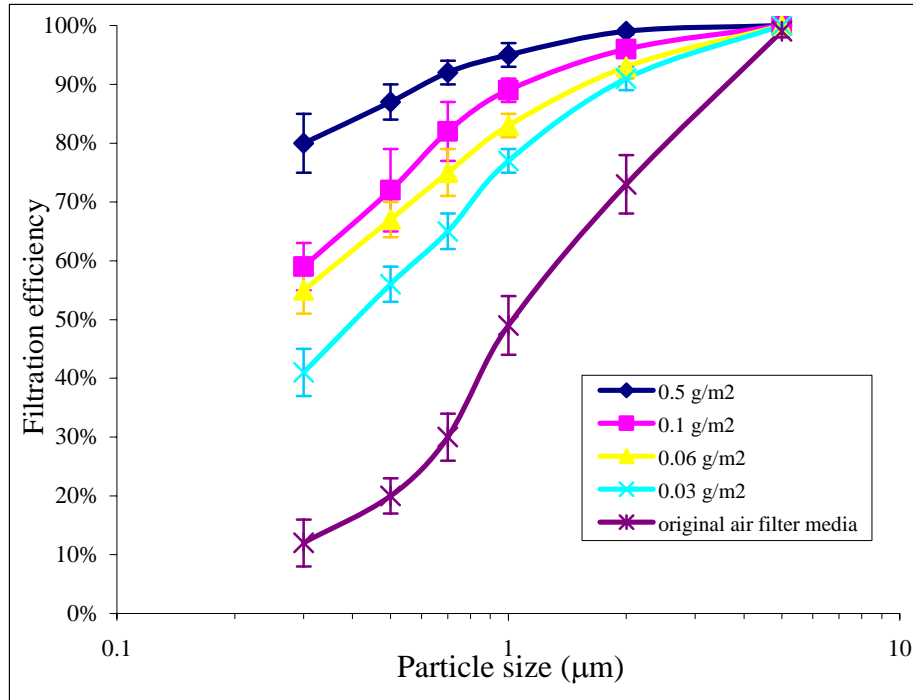


Figure 7. Filtration efficiency of the air filter media coated with nylon-6 fiber with average diameter of 120 nm at different coverage levels versus the particle size.



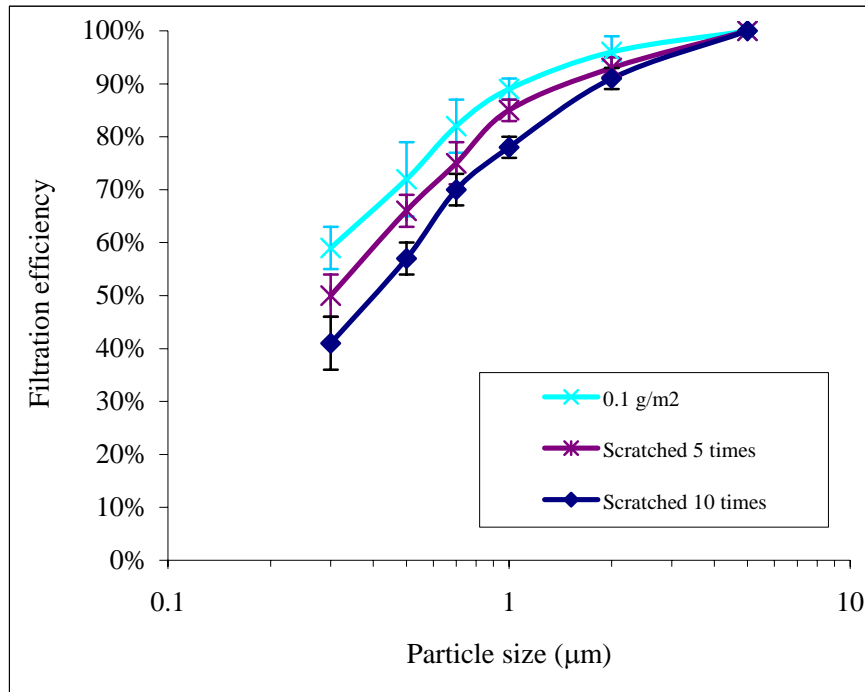


Figure 8. Durability of filtration efficiency of air filter media coated with nylon-6 nanofibers (average diameter = 120 nm) at coverage level of 0.1 g/m<sup>2</sup>.

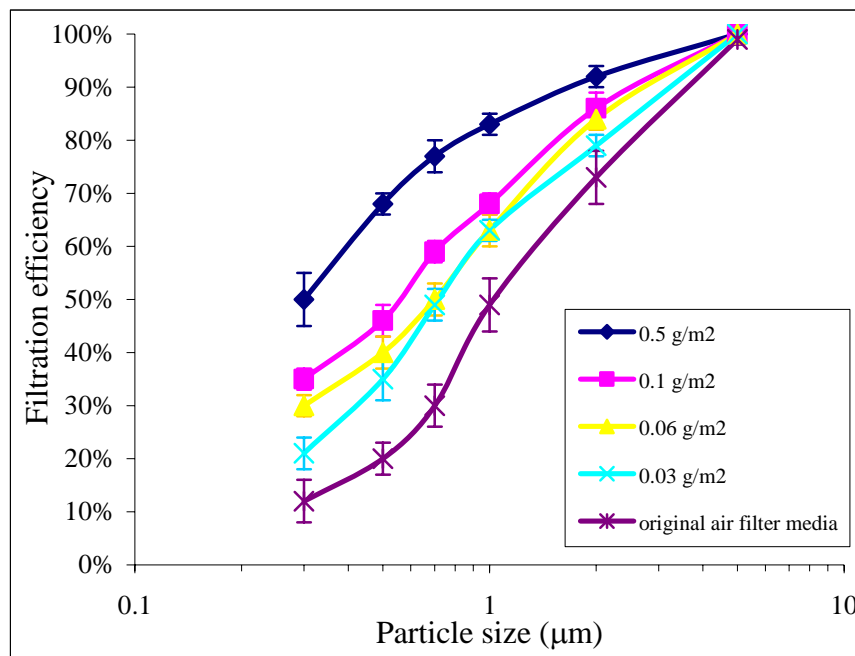


Figure 9. Filtration efficiency of the air filter media coated with nylon-6 fiber with average diameter of 300 nm at different weight coverage levels versus the particle size.

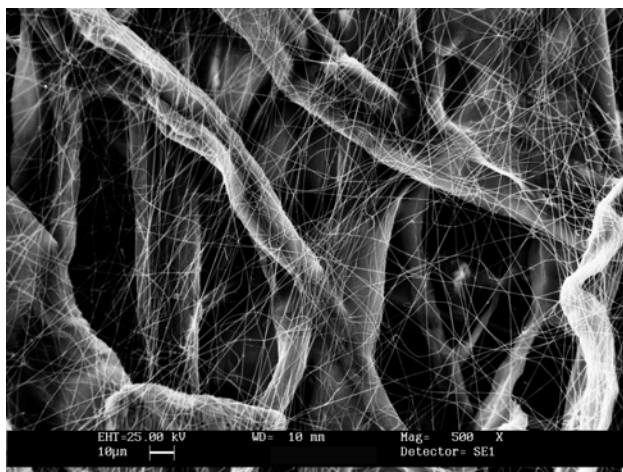


Figure 10. The packing of nylon-6 electrospun fibers with the average diameters of 300 nm on the air filter media at the weight coverage level of 0.1 g/m<sup>2</sup>.

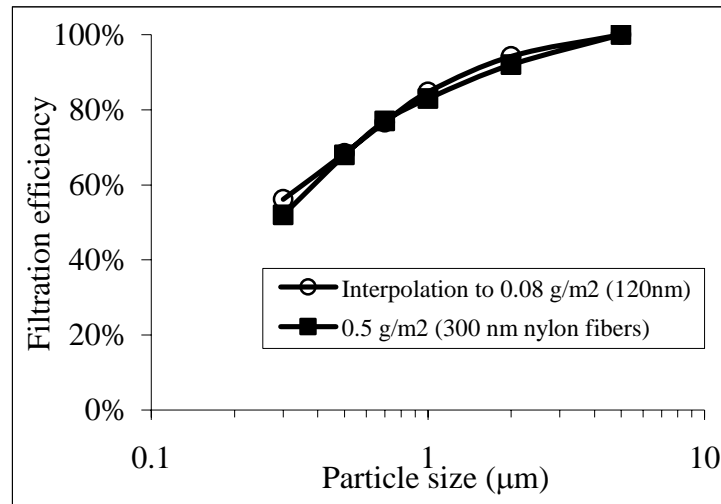


Figure 11. Filtration efficiency of air filter media coated with nylon-6 electrospun fibers with different fiber sizes at the similar fiber length.

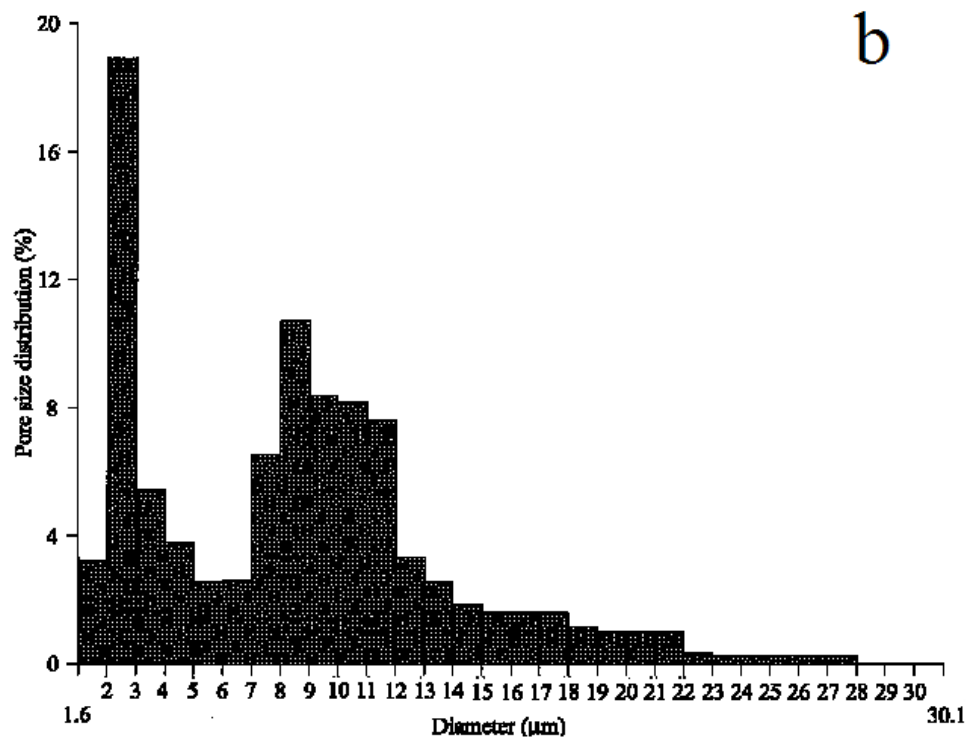
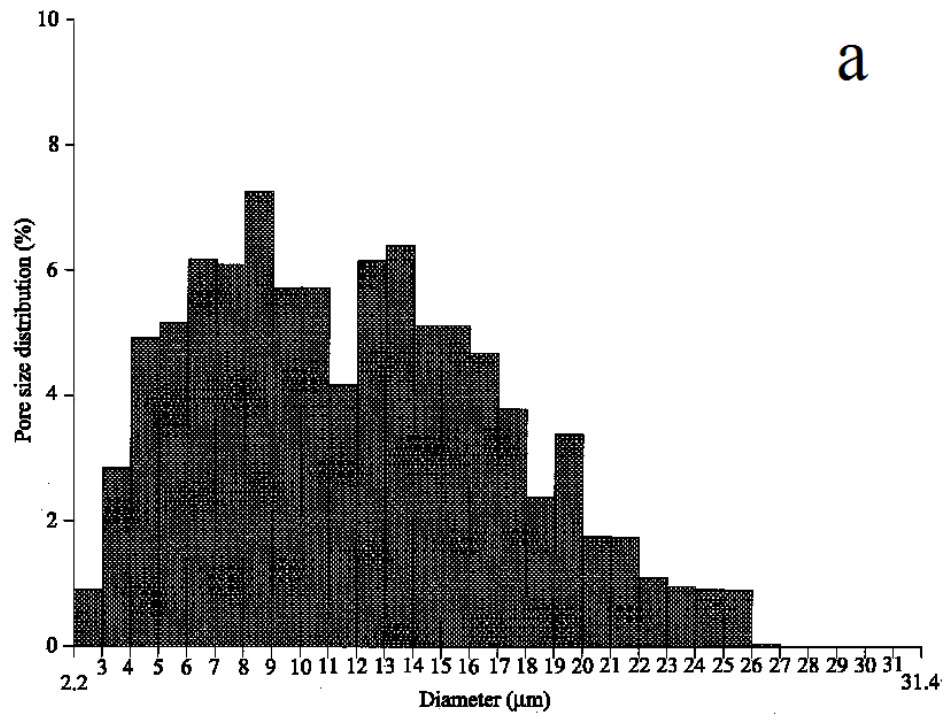


Figure 12. Pore size distribution of (a): original air filter media; and (b): air filter media coated with nylon-6 electrospun fibers at the weight coverage level of  $0.1 \text{ g/m}^2$ .

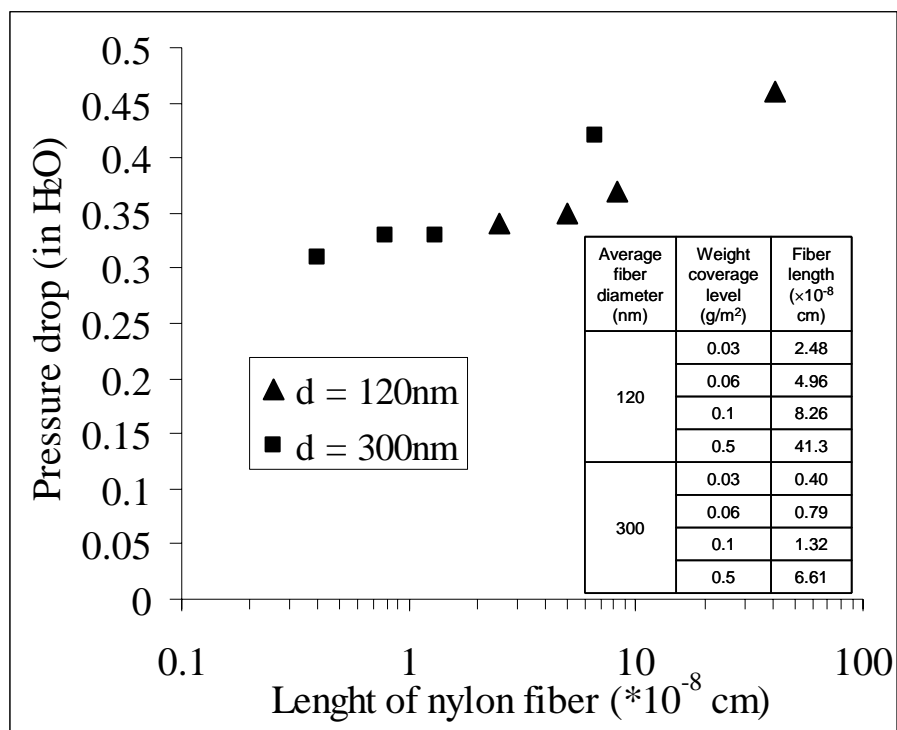


Figure 13. Initial pressure drop of the air filter media coated with nylon-6 fibers with different fiber length. The inserted table gives the calculated length of nylon fibers with different diameters at different weight coverage levels.

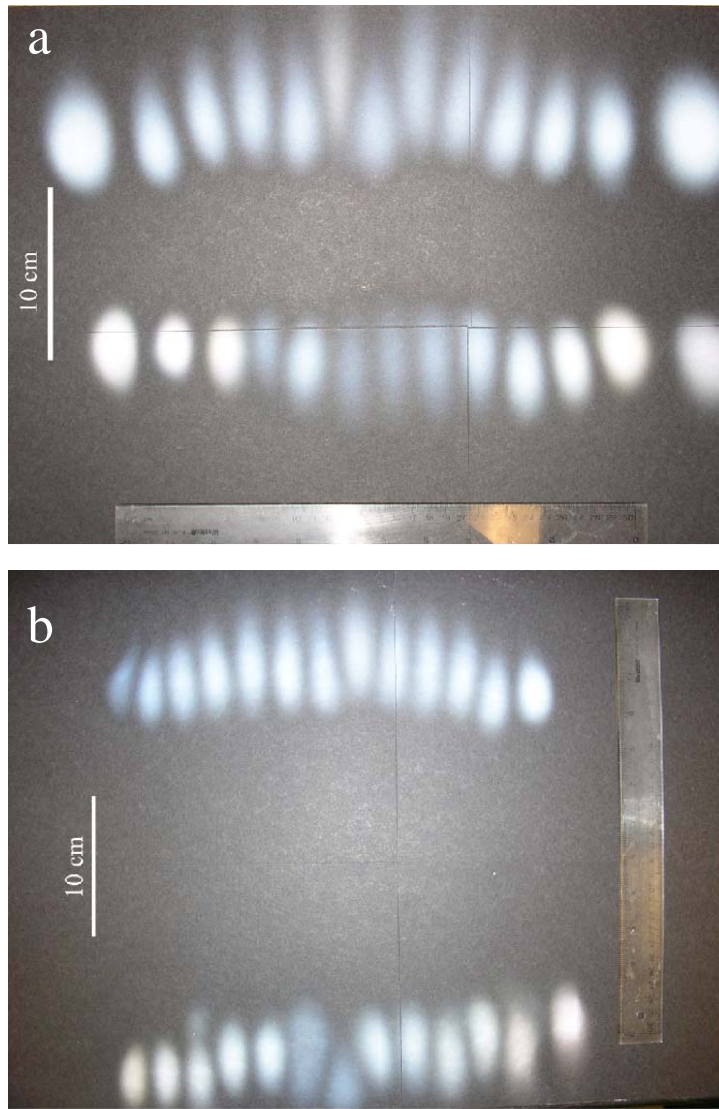


Figure 14. Deposition of nylon-6 fibers electrospun from two nozzle banks separated at (a): 8 cm and (b): 24 cm.

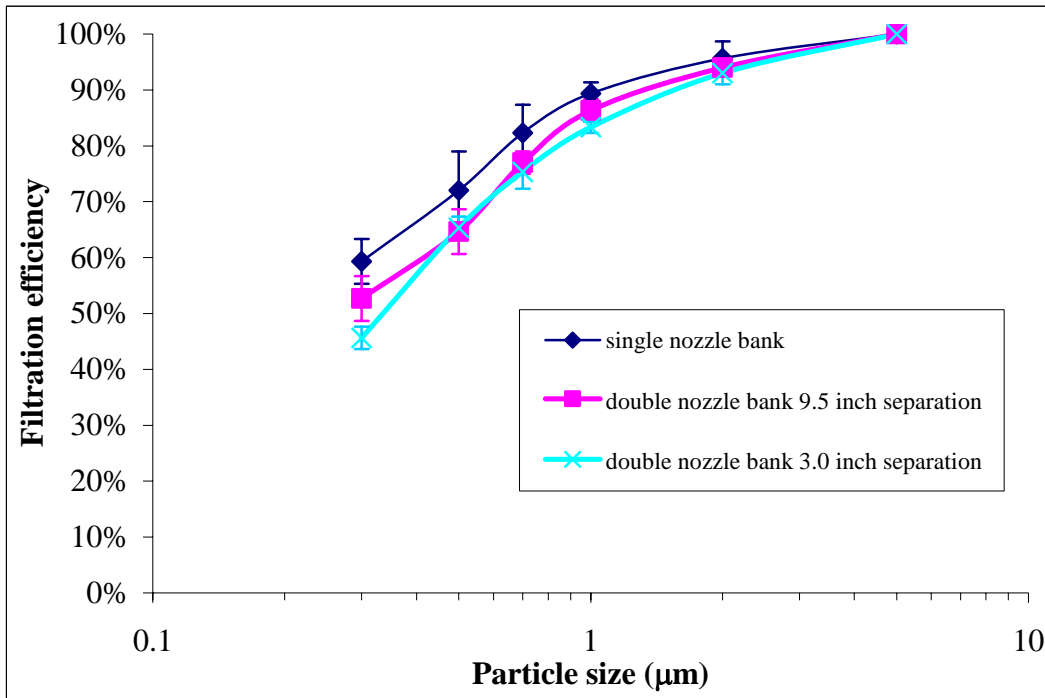


Figure 15. Filtration efficiency of air filter media coated with nylon-6 fibers electrospun from a single or two nozzle banks at the same weight coverage level,  $0.1 \text{ g/m}^2$ .