Quality variables of meltblown submicron filter materials

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Meltblown submicron fibre filter media has been produced at a production rate of a few orders of magnitude higher than that of conventional single syringe electrospinning. Fibres are produced while varying the air pressure and die-to-collector distance (DCD). The submicron fibres are about 240 nm in diameter. The average fibre diameter of the submicron fibre nonwoven filters media decreases with increasing DCD and air pressure. The average fibre diameter of each sample is indirectly related to the hydrohead and filtration efficiency, but is directly related to the pore size. The quality factors improve slightly for samples produced at a greater DCD, whereas the quality factors for the media decrease slightly for samples produced at a higher air pressure. The average fibre diameter is related to the hydrohead, filtration efficiency, and pore size. The sample MSFM6 is found to have the smallest average fibre diameter and the highest filtration efficiency. The filtration efficiency of different submicron fibre nonwoven media samples improve with increasing DCD or air pressure. The use of nanofibrous media has proved to be a good way to filter out submicron-sized particles.

Keywords: Die-to-collector distance, Electrospinning, Filter media, Quality factor, Submicron fibre membrane

1 Introduction

Nonwoven filter media are being used since a very long time for water filtration, water desalination, effluent treatment, and the purification of air, water, coolant, cabin air, engine oil, and transmission oil1-4. Typically, nonwoven filter media are made up of microfibres and are being used for the removal of micro-particles, such as coarse dust particles. Microfibres do not effectively remove small particles, especially those in the range of diameter 100 - 1000 nm, because of their large pore sizes, that will not shrink, until particles are collected on the filter surface5,6. Moreover, particles trapped within the pores of nonwoven filter media cannot easily be removed. Furthermore, the structure of media causes non-uniform particle2. The filtration efficiency and pressure drop of nonwoven filter media are indirectly related to fibre diameter and pore size. As the fibre diameter decreases, the interception, inertial impaction efficiency, and consequently filtration efficiency increase. Submicron fibres have very high specific surface areas ranging from 10 m²/g (for 500 nm submicron fibres) to 100 m²/g (for 50 nm submicron fibres).

As compared to microfibre filters, submicron fibre filters hold great promise because they have less drag, higher permeability, high porosity, and smaller pressure drop. The higher specific surface area of submicron fibres ensures a greater probability of capturing particles, resulting in high filtration efficiency. Nonwoven submicron fibre media have low basis weights, smaller pore sizes, higher particle capture efficiencies, and therefore, higher filtration efficiency and longer filter life. The mean pore size and the pore size distribution are the key parameters that determine filtration performance of a medium. The pore size of the filter media determines the size of the particles that can be trapped. Submicron fibre membranes are effective in filtering particles smaller than 100 nm. The study recognizes that the filtering efficiency is significantly affected by the distribution of fibre diameters rather than their average size. In addition, air permeability is directly related to fibre diameter and pore size. Adding judicious quantities of submicron fibres to filter media leads to higher filtration efficiencies and minimal increase in pressure drop. The filtration efficiency of the air filter media coated with submicron fibres at 0.1 g/m² is very high7.

Submicron fibres reduce the most penetrating particle size (MPPS) when compared with conventional microfibre filters. Packing density rather than layer thickness influences submicron fibre MPPS. Multilayered submicron fibre filters have been found to have much higher capture efficiencies for submicron particles with the same pressure drop than a single-layer submicron fibrefilter. There are more than 25 companies worldwide that use submicron fibres.
fibres in their filters including the Donaldson Company, Hollingsworth & Vose, Ahlstrom, Amsoil Inc. and Exceed. Submicron fibres can be produced through electrospinning, multi-component fibre spinning, or modular melt blowing. Among the three methods, electrospinning is the most extensively investigated. However, a key drawback of electrospinning is a low production rate because of vapor generation. Some polymers are also difficult to dissolve and electrospinning can leave defects like beads and pores.

The low production rate makes electrospun submicron fibres prohibitively expensive. For this reason, other innovative approaches such as melt blowing, which produces media from thermoplastic polymers, must be employed. Melt blowing is a one-step nonwoven process in which a modular die extrudes thermoplastic fibre-forming polymer into converging air streams to rapidly form fine fibres. High velocity hot air helps to produce microfibres in this method. These fibres are deposited on a moving collector as a self-bonded web. Generally, melt blowing produces microfibres of 2–5 μm. However, submicron fibres as fine as 75 nm in diameter are currently meltblown to produce nonwoven filter media by using modular dies. Modular melt blowing can spin submicron fibres from thermoplastic polymers at a production rate of several orders of magnitude higher than conventional single syringe electrospinning. The production costs of melt blown submicron fibres could be $10/kg or below. In this study, nonwoven submicron fibre media has been produced through modular melt blowing, and the submicron fibres are characterized and evaluated.

2 Materials and Methods

2.1 Materials and Processing

Melt blown nonwoven submicron fibre media samples (carded webs) were produced from commercial grade polypropylene (PP, Aldrich Chemicals with no additives and processing aids, ρ ~ 0.900 g/cm³, Mw 224000, and Mw/Mn 4.2). A 15 cm melt blowing pilot line was used with a nanofibre modular die. Temperatures in the extrusion zone were between 180°C and 200°C, temperature in the die zone was 240°C, and the air temperature was 255°C. Webs were produced using a mass throughput of 0.02 gram/hole/min (ghm), and the collector speed was 0.86 m/min (mpm). A schematic diagram of the 5 inch wide melt blowing port is shown in Fig. 1. A thermoplastic fibre-forming PP polymer is extruded through small orifices into convergent streams of hot air that rapidly attenuate the extrudate into small diameter fibres. The air streams also transport fibres to a collector where they are bonded at fibre-fibre contact points to produce a cohesive nonwoven web. The fibres were collected as a mat on a rotating, smooth stainless steel drum. Mats from the drum were wrapped onto a floating take-up roll. The choice of a smooth collector was made to facilitate future modeling work on process behavior near the collector.

2.2 Experimental Procedure

Air pressure and die-to-collector distance (DCD) were each studied at three discrete levels by performing one-factor-at-a-time (OFAT) experiments. This experimental design allowed the effect of each variable to be studied individually by maintaining the other factor constant. The sample codes and the conditions are shown in Table 1.

Table 1—Factors and discrete levels for samples MSFM1-MSFM6

<table>
<thead>
<tr>
<th>Sample</th>
<th>DCD, cm</th>
<th>Air pressure, kPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>MSFM 1</td>
<td>Level 1</td>
<td>25</td>
</tr>
<tr>
<td>MSFM 2</td>
<td>Level 2</td>
<td>30</td>
</tr>
<tr>
<td>MSFM 3</td>
<td>Level 3</td>
<td>35</td>
</tr>
<tr>
<td>MSFM 4</td>
<td>25</td>
<td>30</td>
</tr>
<tr>
<td>MSFM 5</td>
<td>25</td>
<td>30</td>
</tr>
<tr>
<td>MSFM 6</td>
<td>30</td>
<td>30</td>
</tr>
</tbody>
</table>

Fig. 1—Schematic diagram of 5 inch wide melt-blowing used in the study
2.3 Characterization

The melt blown nonwoven submicron fibre media was conditioned as per the ASTM standards. Fibre diameters were measured by scanning electron microscopy (SEM). The average fibre diameter and SD were based on the measurement of 50 fibres per sample. Additionally, each sample’s air permeability and hydrostatic head were measured to evaluate its permeability. Air permeability is defined as “the rate of airflow passing perpendicularly through an area of $38 \text{ cm}^2$ under an air pressure differential of 125 Pa across the submicron fibre nonwoven media [ASTM D 737].” Air permeability was measured on a TexTest FX 3300 and hydrostatic head was measured using a TexTest FX3000. Hydrostatic head is a measure of the liquid resistance of a fabric before three drops of water appear on the surface of the nonwoven fabric.

A fabric with a higher hydrohead reading indicates that it presents a greater barrier to liquid penetration. The pore size of the nonwoven media was measured using a capillary flow porometer. When the applied air pressure exceeds the capillary attraction of the wetting liquid in the pores, air passes through the sample in order to determine the mean pore size and pore size distribution. Generally, smaller pores have more capillary power than larger pores; thus, smaller pores require higher pressure to open. The thickness of each nanofibre sample was measured according to ASTM D 5729-95 using digital thickness instrument TMI 49-70. The basis weights ($w$) of the five 5 × 5 inch rectangular submicron fibre nonwoven media samples were calculated according to ASTM D 3776 as per the following formula:

$$w(\text{g/m}^2)= m/A,$$  

where $m$ is the mass of each sample; and $A$, the area of each sample. Filtration efficiency was measured on a TSI automated filtration tester. The average particle size and weight average particle size were 75 nm and 260 nm, respectively. The filtration efficiency ($\eta$) was calculated based on the aerosol concentration upstream ($c_{up}$) and the aerosol concentration downstream ($c_{down}$), as shown below:

$$\eta = \frac{c_{up} - c_{down}}{c_{up}},$$  

The filter media should have high filtration efficiency ($\eta$) and low pressure drop ($\Delta P$). A small pressure drop is desired to keep the operating cost down. The quality factor (QF) was defined as a quantitative criterion to compare various fibrous filters. The QF was determined as follows:

$$QF = -\ln \left(1-\frac{\eta}{\Delta P}\right),$$  

Though the quality factor (QF) took into account the thickness ($t$), porosity ($\varepsilon$), and fibre diameter ($d_F$) of the submicron fibre nonwoven media, in our opinion another factor (the basis weights of the samples) also affected filtration efficiency. In order to take into consideration the variations in the basis weight, the filtration efficiency was normalized with respect to the basis weight, as given below:

$$\eta_w = \frac{\eta}{w},$$  

3 Results and Discussion

3.1 SEM Analysis

The filtration efficiency of the nonwoven filter media is significantly influenced by the fibre diameter of each sample. Each set of SEM images, taken at 10.9 mm and 7.5mm working distances and ×2000 magnification, shows that the fibres are in the submicron range (Fig. 2).
The bundled fibres were not taken into consideration for further analysis. Figure 2 shows SEM images of submicron fibre nonwoven media (MSFM). Variation in fibre diameter is observed in the images of the nonwoven filter media samples. The coarse submicron fibres having diameters greater than 100 nm show better filtration efficiency. Due to bundling, which is a problem with modular melt-blowing, some of the fibres appear to be coarser.

3.2 Effects of DCD on Properties of MSFM Samples
The average fibre diameters of samples MSFM1, MSFM2, and MSFM3 are 570, 560, and 550 nm (Table 2). A 10 cm increase in the DCD at a constant air pressure produces finer fibres. DCD is directly related to the time allowed for primary airflow-dependent fibre attenuation. Finer fibres, produced with a longer separation between the die and the collector, are a consequence of greater deformation and attenuation. These finer fibres are subjected to greater air drag while hot, and are not crystallized. The deformation of the hot, uncrystallized fibres occurs far from the die, which results in slightly finer fibres at higher DCDs.

Air permeability of the samples increases nominally from 25 cm to 35 cm with increasing DCD as a constant air pressure. Therefore, it is assumed that the increased in DCD shows a positive relationship on air permeability. The average pore size is an important factor of filtration efficiency and pressure drop of a nonwoven filter medium. The average pore sizes of the samples are found to be indirectly related to the DCD at a constant air pressure. The average pore size of the nonwoven filter media dropped from 10.38 microns to 6.45 microns as shown in Table 2. The porosity of a filter is primarily determined by the average fibre diameter and the way the fibres are consolidated. The pore size reduces due to a higher degree of fibre entanglement. Hence, there is greater consolidation when there are more or longer submicron fibres in each mass unit of polymer. Hydrohead, which signifies the liquid resistance of a nonwoven fibrous filter media, is increased with DCD (Table 2). The impact nonwoven fabric structure has lesser amounts of voids, which restricts the fabric to transmit more amount of air. High air permeability is equal to a good barrier property. Air permeability depends on the pressure. Considering the standard deviations in hydrohead values, the increase in hydrohead can be considered significant. As the average fibre diameter is decreased, the specific surface area of the finer fibres is increased. Concomitantly, the average pore size is decreased, and the media’s resistance to the liquid penetration increases. The filtration efficiency of the filter media shows a slight increase with DCD at a constant air pressure (Table 2). This is due to the smaller pore sizes and greater specific surface areas of the finer fibres produced at higher DCDs. The small pressure drop of the nonwoven submicron fibre filter media is an interesting observation. A small pressure drop normally results from the highly porous structure of extremely thin nonwoven submicron fibre filters. At first, the high QF values could be ascribed to the small pressure drops in the nonwoven filter media. The QF values for submicron fibre nonwoven filter media improve slightly at a greater DCDs and a constant air pressure (Table 2). The improvement in the QF values is also due to synergistic effects of greater filtration efficiencies and smaller pressure drops.

3.3 Effects of Air Pressure on Properties of MSFM Samples
The average fibre diameters in the samples are indirectly related to the primary airflow pressures at a constant DCD (Table 2). Fibre diameters reduce from 590 nm to 520 nm with the increase in airflow pressure from 10 psi to 20 psi. Finer fibres, produced at higher air pressure, are the result of the greater deformation and attenuation due to higher air drag for only those fibres. Deformation of the hot uncrystallized fibres results in the production of finer fibres at higher air pressure. Hence, the variability in the average fibre diameter may be due to variations in the airflow pressure and air velocity along the face of the MB die as well as toward the collector.

<table>
<thead>
<tr>
<th>Property</th>
<th>MSFM1</th>
<th>MSFM2</th>
<th>MSFM3</th>
<th>MSFM4</th>
<th>MSFM5</th>
<th>MSFM6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number average fibre diameter, microns</td>
<td>0.57</td>
<td>0.56</td>
<td>0.55</td>
<td>0.59</td>
<td>0.56</td>
<td>0.52</td>
</tr>
<tr>
<td>Thickness, mm</td>
<td>0.44</td>
<td>0.51</td>
<td>0.53</td>
<td>0.52</td>
<td>0.51</td>
<td>0.51</td>
</tr>
<tr>
<td>Basis weight, g/m²</td>
<td>25</td>
<td>24.7</td>
<td>24.5</td>
<td>25</td>
<td>24.7</td>
<td>24.8</td>
</tr>
<tr>
<td>Air permeability, cm³/s/cm²</td>
<td>14.9</td>
<td>15.2</td>
<td>15.3</td>
<td>14.8</td>
<td>15.2</td>
<td>17.1</td>
</tr>
<tr>
<td>Average pore size, microns</td>
<td>10.4</td>
<td>6.7</td>
<td>6.5</td>
<td>6.7</td>
<td>6.7</td>
<td>6.4</td>
</tr>
<tr>
<td>Hydrohead, mbar</td>
<td>70.5</td>
<td>73.6</td>
<td>75.5</td>
<td>52.5</td>
<td>73.6</td>
<td>79.2</td>
</tr>
<tr>
<td>Filtration efficiency, %</td>
<td>80.2</td>
<td>81.5</td>
<td>81.9</td>
<td>78.6</td>
<td>81.5</td>
<td>82.5</td>
</tr>
<tr>
<td>Pressure drop, Pa</td>
<td>54.9</td>
<td>54.9</td>
<td>49</td>
<td>53</td>
<td>54.9</td>
<td>57.9</td>
</tr>
<tr>
<td>Quality factor (QF), Pa⁻¹</td>
<td>0.029</td>
<td>0.031</td>
<td>0.035</td>
<td>0.033</td>
<td>0.031</td>
<td>0.027</td>
</tr>
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</table>
The air permeability of the fibre samples is related to air pressure at a constant DCD. Average air permeability is increased from 14.8 cm³/s/cm² to 17.1 cm³/s/cm² at a constant DCD. The average pore sizes are indirectly related to air pressure. The average pore size of the nonwoven filter media drops from 6.7 µm to 6.34 µm with increasing air pressure. As explained above, this study recognizes that the web’s porosity relies considerably on the average fibre diameter and the consolidated fibres. Hence, as the average fibre diameter decreases, the pore size also should have decreased due to the increase in the specific surface area of the finer fibres. As explained above, the higher degree of fibre entanglement resulting from the greater number or longer fibres creates a denser medium with smaller pores. Hydrohead increases with the rise in air pressure at a constant DCD. A decrease in the mean pore size could have resulted from an increase in the specific surface area of the finer fibres and higher degree of fibre entanglement for the greater number or longer fibres produced for the same mass of the polymer at higher air pressure.

The filtration efficiency of the samples increases with air pressure at a constant DCD. The filtration efficiency greatly improves from 25 kPa to 30 kPa because the resulting pores are smaller, the finer fibres show greater specific surface area, and that the long, thin fibres show greater entanglement. The small pressure drops in the samples are encouraging because this is required to keep costs low. Furthermore, the study recognized that the highly porous structures of the media heavily influence the pressure drops. With increasing air pressure, the pressure drop increases due to the greater degree of fibre entanglement in the long, thin, and dense fibres produced at high air pressure. The filtration efficiency is directly related to the pore size distribution. With increase in the number of layers the pore size decreases and the filtration efficiency increases. Filtration fineness is one of the important concerns for the filter media performance. Because of the very high surface area of melt-blown nano webs to volume ratio and resulting high surface cohesion, tiny particles of the order of 0.4 µm can be easily trapped in the melt-blown nanofibrous structured webs and hence the filtration efficiency can be improved.

The basis weights of the submicron fibre nonwoven media samples are varied at a constant DCD or at a constant air pressure (Table 2). The variation in the basis weights of the samples certainly affects filtration efficiency. Therefore, in order to compare the filtration efficiency of different samples, the efficiency values are normalized [NFE (w)] to the basis weight of each sample (Tables 2 & 3). The normalized filtration efficiency (ηw) improves with increased DCD or air pressure (Fig. 3). Therefore, it

<table>
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<tr>
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<th>MSFM5</th>
<th>MSFM6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basis weight, g/m²</td>
<td>48.01</td>
<td>52.25</td>
<td>58.04</td>
<td>55.95</td>
<td>55.51</td>
<td>54.81</td>
</tr>
<tr>
<td>Filtration efficiency (η), %</td>
<td>80.2</td>
<td>81.5</td>
<td>81.9</td>
<td>78.6</td>
<td>81.5</td>
<td>82.5</td>
</tr>
<tr>
<td>Normalized filtration efficiency (ηw), m²/g</td>
<td>3.2</td>
<td>3.25</td>
<td>3.27</td>
<td>3.14</td>
<td>3.25</td>
<td>3.29</td>
</tr>
</tbody>
</table>

Fig. 3—Normalized filtration efficiency of submicron fibre nonwoven media (a) effects of DCD on NFE (w) and (b) effects of air pressure on NFE (w)
could be concluded that the filtration efficiency improves because fibres produced with greater DCD or air pressure has greater specific surface areas and smaller pore sizes, irrespective of the basis weight of the samples.

4 Conclusion
The melt blowing technique, which conventionally produces microfibres, has been modified by using modular dies to produce nonwoven submicron fibre filter media. Submicron fibres that are derived from thermoplastic polymers could be spun by using the modular melt blowing method. The productivity, achieved by using the modular melt, is higher than that of conventional single syringe electrospinning. Thus, this modular melt blowing method allows one to achieve an impressive reduction in manufacturing costs without using harmful solvents. Submicron fibres with diameters as small as 240 nm could be produced. These fibrous media are suitable for filtering out submicron-sized particles. This research also discovered inverse relationships among the average fibre diameter, DCD, and air pressure. The average fibre diameter is related to the hydrohead, filtration efficiency and pore size. MSFM6 is found to have the smallest average fibre diameter and the highest filtration efficiency ($\eta$). The most encouraging observation of the study is the high quality factors for nonwoven submicron fibre filter media. MSFM3 is found to have the highest quality factor. Increasing the DCD slightly improves the quality factors, whereas higher production air pressure reduces the quality factors. The normalized filtration efficiency ($\eta_n$) of the submicron fibre nonwoven media samples improve with an increase in the DCD or air pressure. MSFM6 (the optimum DCD 30 cm and air pressure 138 kpa) has the highest normalized filtration efficiency ($\eta_n$).

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References