FAST EVALUATION OF AVERAGE FIBER DIAMETERS OF NONWOVENS

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ABSTRACT

Average fiber diameters of nonwovens can be very quickly determined by a number of simple techniques, which include the gas permeability technique, the envelope surface area technique and the gas adsorption surface area technique. The results obtained by these techniques and their limitations have been critically examined. Under conditions appropriate for each test method, the results are reliable.

INTRODUCTION

Performance of nonwovens is determined primarily by factors such as fiber diameter, packing density and electrostatic charge on the surfaces of fibers. For production control, evaluation and quality control, quick estimation of the average fiber diameter of nonwovens is essential. Techniques used for fiber diameter measurements are involved and time consuming. Often microscopic samples, which may not be representative of the bulk material are used. In this presentation, novel and completely automated techniques, which can measure average fiber diameter of bulk samples in less than ten minutes are described. The gas permeability technique permits average fiber diameter to be computed from measured pressure drop and flow rate through the sample. In the envelope surface area (external surface area) technique, the average fiber diameter is computed from the envelope surface area of the nonwoven calculated from the pressure drop and flow rate of gas through the sample of the nonwoven. Surface area measured accurately by the gas adsorption technique can also yield average fiber diameter. The three techniques and the results obtained using these techniques have been critically examined.

TECHNIQUES

The Gas Permeability Technique

According to Darcy’s law, flow of a fluid through porous media is given by [1]:

\[ F = \frac{kA}{\mu}(\Delta p / l) \]  

(1)
where:

- \( F \) = volume flow rate through the material at average pressure
- \( k \) = permeability having unit of \((\text{length})^2\)
- \( A \) = area of the sample
- \( \mu \) = viscosity of fluid
- \( \Delta p \) = differential pressure
- \( l \) = thickness of sample.

Consequently, \( k \) is given by:

\[
k = \frac{(F \mu l)}{(A \Delta p)} \tag{2}
\]

The principal parameters, which control permeability \((k)\) are fiber radius and packing density. In order to explore the nature of this relationship, let us model the porous material to consist of \( N \) pores of diameter, \( D \) per unit area of sample. Poiseuille’s equation gives the flow rate through such a sample [1].

\[
F = \frac{(ZA / \mu)}{(ND^4)(\Delta p / l)} \tag{3}
\]

Where, \( Z \) is a constant. In order to relate pore diameter and pore number to fiber radius, let us consider fibers of radius \( R \) and length \( L \) to form a square net. Estimating the number of fibers from the total volume of fibers and volume of each fiber, the term \((ND^4)\) becomes:

\[
ND^4 = \left[ \frac{R}{\pi c} \right]^2 \left[ \pi - 2c \right]^4 \tag{4}
\]

where, \( c \), the packing density is given by the volume of fibers to the volume of sample.

\[
c = (1-P) \tag{5}
\]

where \( P \), the porosity is the ratio of the volume of through pores to the total volume of sample. Substituting for \( ND^4 \) from Equation 4 in Equation 3 and equating expressions for flow rates from Equations 1 and 3:

\[
k = Z \left[ \frac{R}{\pi c} \right]^2 \left[ \pi - 2c \right]^4 \tag{6}
\]

Equating the expressions for \( k \) from Equations 2 and 6 and simplifying:

\[
\frac{(4 \Delta p A R^2) / (\mu F l)}{(4/z \pi^2) c^2 [1 + (8/\pi)c]} = (4/\pi) \tag{7}
\]

Although the model is simple, it brings out the fact that the dimensionless quantity on the left side of Equation 7 is likely to be a unique function of porosity.

Davies [2] used the measurements made on a wide variety of fibrous materials and their average fiber diameters, and plotted the function on the left side of Equation 7 against \( c \). In spite of the wide range of materials that he used, he did find a unique relationship (Equation 8). This
The experimental relationship is surprisingly close to the one predicted by the model, although, the model contained many approximations.

\[
\frac{4 \left( \Delta p A R^2 \right)}{\left( \mu F l \right)} = 64 c^{1.5} \left[ 1 + 52 c^3 \right], \quad P \approx 0.7 - 0.99
\] (8)

Equation 8 can be used to calculate average fiber diameter. Hinds has tested this relationship to calculate average fiber diameter \[3\]. The relationship works well in the range of packing density of 0.006 and 0.3 (porosity range of 0.994 and 0.7) \[3,4\]. The relation can be used in conjunction with measurement of flow rate to rapidly estimate the average fiber diameter within the domain of applicability of the relationship.

The PMI Average Fiber Diameter Analyzer (Figure 1) was used to measure average fiber diameters of a number of fibrous samples having porosity in the range of 0.74 to 0.79 (c: 0.26 – 0.21). This instrument yields highly reliable, accurate and reproducible data.

![Figure 1 PMI Average fiber diameter analyzer](image)

**The Envelope Surface Area Technique**

The envelope surface area is the external surface area that sees flow of gas through the sample. Assuming the fibers to have the same length, the average fiber diameter can be calculated using the envelope surface area and the volume of fibers from their true density and mass.

\[
D = \frac{4V}{S} = \frac{4}{S \rho}
\] (9)

Where:
- \(D\) = average fiber diameter
- \(V\) = volume of fibers per unit mass
- \(S\) = envelope surface area of fibers per unit mass
- \(\rho\) = true density of fibers

Envelope surface area can be computed from measured flow rates of gas through porous media using the Kozeny-Carman relationship modified to include the contribution from Knudsen flow \[5\].

\[
\frac{F}{\Delta p A} = \left\{ \frac{P^3}{b(1-P)^2(S\rho)^2 \mu} \right\} + \left[ \frac{z P^2 \pi}{(1-P)(S\rho)\sqrt{2\pi\rho_g g}} \right]
\] (10)
where:
\[ F = \text{volume flow rate of gas at the average pressure} \]
\[ l = \text{thickness of sample} \]
\[ \Delta p = \text{pressure drop across the sample} \]
\[ A = \text{cross-sectional area of the sample} \]
\[ P = \text{porosity (pore volume / total volume), obtained from the bulk and true densities of the porous medium.} \]
\[ b = \text{a constant determined by the geometry of the pore structure of the porous medium. It has a value close to 5 for random pored media} \]
\[ S = \text{envelope surface area per unit mass of the solid} \]
\[ \rho = \text{true density of fibers} \]
\[ \mu = \text{viscosity of gas} \]
\[ z = \text{a constant. It has been shown to be (48 / 13\pi) [4]} \]
\[ \rho_g = \text{density of gas at average pressure} \]
\[ p = \text{mean pressure in the porous medium} \]

The PMI Envelope Surface Area analyzer (Figure 2) was used in this investigation. This is a completely automated instrument capable of giving envelope surface area accurately, reproducibly and rapidly from measured flow rates and pressure drops. The average fiber diameter is computed from the envelope surface area using Equation 9.

![Figure 2 The PMI Envelope Surface Area Analyzer](image)

**The Gas Adsorption Technique**

The gas adsorption technique is capable of measuring surface area accurately over a wide range particularly when the surface area is high. The measured surface area includes the surface area of blind pores and through pores (Figure 3). If the fibers are free from pores, the gas adsorption surface area is an appropriate and accurate measure of envelope surface area. Therefore, the gas
adsorption surface area can be used to compute average fiber diameter using Equation 9. For fast evaluation of average fiber diameter, surface area needs to be determined quickly. Normally, gas adsorption equipment takes a long time to measure surface area. However, PMI’s QBET series of gas adsorption equipment is capable of measuring surface area within a few minutes. The instrument shown in Figure 4 was used to measure surface area quickly.

Figure 3  Pores in fibers

Figure 4  Gas adsorption equipment for fast evaluation of surface area
RESULTS AND DISCUSSION

Fiber diameter by gas permeability

The average fiber diameters of five non-wovens, whose average fiber diameters were known, were used. The porosity of nonwovens was close to around 76%. The average fiber diameter was measured using the PMI Average Fiber Diameter Analyzer. The reproducibility of the result was better than ±3%. The measured values are plotted in Figure 5 against the actual fiber diameters. Good agreement between the measured values and the actual values may be noted from Figure 5. The measured values are within twenty percent of the actual values. Table 1 lists the porosity and the fiber diameters of the nonwovens.

The differences between the actual and measured values could be attributed to some of the limitations of the technique.

1. Very high porosity and high fiber diameter result in large pores, high Reynolds number and turbulent flow. Hence, Darcy’s law is invalid and the technique is not applicable. At low porosity and high fiber density, the right side of Equation 9 may contain other terms besides c. For example the path may become more tortuous and contributions from slip flow may become appreciably. However, samples used in the present investigation had porosity in the range 0.74 to 0.79.

2. If appreciable portion of fiber axes deviates strongly from the position normal to the flow direction, packing density remains unchanged and the flow rate is higher. Hence, the measured average fiber diameter comes out larger. Orientation factor may be responsible for part of the error.

3. Clumping of fibers will also lead to a higher value of the average fiber diameter. This is expected to be particularly serious in case of low porosity products.

4. Fiber volume is normally calculated from the mass and true density of fibers. However, fiber volume calculated in this manner ignores the volume of blind pores and close pores, which do not permit flow. If volume of such pores is appreciable, fiber diameter measured by this technique will be smaller than the true value.

Fiber diameter from envelope surface area

The envelope surface area of nonwoven #1 was measured in the PMI Envelope Surface Area Analyzer and the average fiber diameter was computed. The values are listed in Table 2. The average fiber diameter measured by envelope surface area method is much lower than that measured by gas permeability method. The computed fiber diameter is expected to be accurate for fibers with relatively smooth surfaces. The result implies that the fibers have a very rough surface and channels for gas flow so that the measured surface area is high and the average fiber diameter is low.
Figure 5 Measured fiber diameters plotted against the actual fiber diameters

Table 1. Average fiber diameter by permeability technique.

<table>
<thead>
<tr>
<th>Nonwoven</th>
<th>Porosity, P</th>
<th>Actual fiber diameter, microns</th>
<th>Measured fiber diameter, microns</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>0.79</td>
<td>4.0</td>
<td>4.7</td>
</tr>
<tr>
<td>#2</td>
<td>0.79</td>
<td>6.5</td>
<td>7.8</td>
</tr>
<tr>
<td>#3</td>
<td>-</td>
<td>8.0</td>
<td>9.3</td>
</tr>
<tr>
<td>#4</td>
<td>0.77</td>
<td>12</td>
<td>14.0</td>
</tr>
<tr>
<td>#5</td>
<td>0.74</td>
<td>22</td>
<td>21.3</td>
</tr>
</tbody>
</table>
Glass fibers are expected to have smoother surfaces. The average fiber diameters of glass fiber nonwovens #6, #7 & #8 were determined from envelope surface area measurements. The results are included in Table 2. The results are in good agreement with the gas permeability results. The envelope surface area results are only about nineteen percent lower. However, identical trends in the behavior of the nonwovens is detected by both techniques.

Table 2: Average fiber diameter computed from envelope surface area and gas adsorption surface area

<table>
<thead>
<tr>
<th>Nonwoven</th>
<th>Proximity, P</th>
<th>Permeability fiber diameter, μm</th>
<th>Envelope surface area Area, m²/g</th>
<th>Fiber diameter, μm</th>
<th>Gas adsorption Area, m²/g</th>
<th>Fiber diameter, μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>0.79</td>
<td>4.7</td>
<td>0.202</td>
<td>2.6</td>
<td>4.526</td>
<td>0.11</td>
</tr>
<tr>
<td>#6</td>
<td>0.91</td>
<td>6.4</td>
<td>0.306</td>
<td>5.2</td>
<td>1.365</td>
<td>1.2</td>
</tr>
<tr>
<td>#7</td>
<td>0.74</td>
<td>4.7</td>
<td>0.412</td>
<td>3.9</td>
<td>1.360</td>
<td>1.2</td>
</tr>
<tr>
<td>#8</td>
<td>0.75</td>
<td>4.3</td>
<td>0.45</td>
<td>3.5</td>
<td>1.898</td>
<td>0.82</td>
</tr>
</tbody>
</table>

Even if the fibers have smooth surface, the average fiber diameter measured by the two techniques will not be equal, because the gas permeability technique measures the arithmetic average while the envelope surface area method measures a different quantity. Assuming equal length fibers, one gets from Equation 9:

\[ D = \frac{4V/S}{D^2/D} \quad (11) \]

where \( D^2 \) is the average of \( D^2 \) and \( D \) is the average of \( D \). Expressing \( (D^2/D) \) in terms of standard deviation \( \sigma \):

\[ D = D \left[ 1 + \left( \frac{\sigma}{D} \right)^2 \right] \quad (12) \]

Equation 12 suggests that average fiber diameter measured by envelope surface area method is larger than that measured by gas permeability method and the extent of the difference is determined by the magnitude of standard deviation.

The accuracy of average fiber diameters determined through envelope surface area is governed by the limitations of the technique.

1. Surface area computation is based on contributions to flow from viscous flow and slip.

Consequently, low porosity samples would be more appropriate for measurement. Also high porosity results in high pore diameter, high flow rate and less accurate surface area. Although
relatively high porosity samples were used in this study, the agreement between the results of the two techniques are good.

(2) When the fibers have very rough surface and contain blind pores, through pores and closed pores, the fiber volume calculated using true density is less than the volume occupied by the fibers. Therefore, the computed average fiber diameter is smaller. Serious errors may arise due to the presence of appreciable blind pores, through pores and closed pores.

(3) If the fibers have rough surfaces, through pores and channels, the surface area would be large. The large surface area and the lower volume computed from the density would tend to give a lower value of the average fiber diameter.

**Fiber diameter from gas adsorption surface area**

Average fiber diameters were computed from gas adsorption surface areas determined rapidly by QBET. The results are listed in Table 1. The average fiber diameter measured by the gas adsorption technique is much lower than those obtained from the other techniques because the gas adsorption surface area is very high. The gas adsorption technique measures the external surface area as well as the surface areas of blind pores (Figure 3). Because the fibers have a lot of blind pores the gas adsorption technique gives very low values for average fiber diameter.

There are a number of limitations of this technique, which contribute to error.

(1) Appreciable blind pores, through pores and rough surface contribute to surface area and volume of fibers computed from measured density of fibers. This is the major source of error.

(2) Presence of appreciable amount of closed pores do not contribute to surface area, but contribute to errors in computation of volume of fibers.

The average fiber diameters measured using the gas adsorption technique is the same as that measured by envelope surface area technique, but is related to the average fiber diameter measured by gas permeability technique through Equation 12.

**Comparison of the techniques**

Comparison of the three techniques in terms of their merits and limitations are presented in Table 3.

**SUMMARY AND CONCLUSION**

1. Techniques for average fiber diameter determination are time consuming and involved. Therefore, techniques for rapid determination of fiber diameter are important.
2. Strengths and limitations of the three techniques, gas permeability technique, envelope surface area technique and gas adsorption technique, which may be used to estimate average fiber diameters speedily have been discussed.
3. Each technique works well within the domain of its applicability.
Table 3. Merits and limitations of techniques for fast evaluation of average fiber diameter

<table>
<thead>
<tr>
<th></th>
<th>Permeability technique</th>
<th>Envelope surface area technique</th>
<th>Gas adsorption technique using PMI QBET</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Test time</strong></td>
<td>A few minutes</td>
<td>A few minutes</td>
<td>A few minutes</td>
</tr>
<tr>
<td><strong>Cost</strong></td>
<td>Inexpensive</td>
<td>Inexpensive</td>
<td>Inexpensive</td>
</tr>
<tr>
<td><strong>Operator Involvement</strong></td>
<td>Very little</td>
<td>Very little</td>
<td>Very little</td>
</tr>
<tr>
<td><strong>Measured pore Diameter equals</strong></td>
<td>( D )</td>
<td>( D \left[ 1 + \left(\frac{\sigma}{D}\right)^2\right] )</td>
<td>( D \left[ 1 + \left(\frac{\sigma}{D}\right)^2\right] )</td>
</tr>
</tbody>
</table>

For Best Results:

<table>
<thead>
<tr>
<th></th>
<th></th>
<th>Low</th>
<th>Not important</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Porosity</strong></td>
<td>High</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Fiber Orientation</strong></td>
<td>Perpendicular to flow</td>
<td>Not important</td>
<td>Not important</td>
</tr>
<tr>
<td><strong>Blind, through &amp; closed pores and rough fiber surface</strong></td>
<td>Not important</td>
<td>Negligible</td>
<td>Negligible</td>
</tr>
</tbody>
</table>

References