POROMETER
FILTER AND MEMBRANE TESTING TECHNOLOGY

APPLICATION NOTE
ASSESSMENT OF NONWOVEN MATERIALS BY GAS LIQUID POROMETRY

The term nonwoven refers to fiber-based products made by processes not including weaving nor knitting (ASTM D1117-80). The fibers in nonwoven materials are bonded together by chemical, mechanical, heat or solvent treatments. Nonwoven materials are used in wide variety of applications such as filtration, automotive, construction, aeronautical, food, medical and many others. In 2013 the US nonwoven market size was approximately $5.4 billion and was estimated to be nearly $7.5 billion in 2017.

As for every technical material, in-depth characterisation of structure and properties is essential to better understand and improve performance for given applications.

Gas Liquid Porometry (GLP) is one such widely used characterization tool for nonwovens This method measures pore size and pore size distribution of through pores in a fast, simple measurement. The technique is based on the displacement of an inert and nontoxic wetting liquid which is spontaneously embedded in the porous network of a material. The liquid is them removed by application of an inert pressurised gas (e.g. nitrogen). Following an inverse relationship between pressure and size, the largest pores in a material empty first and, as the applied pressure increases, smaller and smaller pores are emptied until the material is “dry” (i.e. the liquid is expelled of od the smallest pores). The measurement has two variables – applied differential gas pressure and flow rate through the material. The pressure required to empty pores of a certain diameter is used to calculate a pore size according to the Young–Laplace formula $P = 4*\cos(\theta)*\gamma/D$, where (P) is the pressure required to displace the liquid from the pore, (θ) is the contact angle of the wetting fluid with the material, (γ) the surface tension of the wetting liquid and (D) is the pore diameter. The flow, is used to determine the relative quantities of pores in a given size range, and to determine permeability and other flux characteristics.

APPLICATION TO NONWOVEN MATERIALS

“Gas Liquid Porometry” – hereafter referred to as “GLP” measurements were carried out on representative melt blown and spun bound materials, using a POROLUX™ 100 porometer from POROMETER nv (figure 1). The POROLUX™ 100 was specially designed for nonwoven characterisation taking into account the characteristic pressure and flow ranges of typical nonwovens, and it is based on the “pressure scan” method – a fast, yet reproducible method whereby pressure is continually increased and resulting flow rates are recorded simultaneously. This “scanning” method is preferred for environments where simplicity, speed and reproducibility are required. This makes the POROLUX™ 100 an ideal choice for quality control and assurance.

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The maximum operating pressure of the POROLUX™ 100 is 1.5 bars (22 psi), which permits measuring pore sizes between 0.427 µm and 500 µm. The determination of the first bubble point – hereafter referred to as “FBP”, or maximum pore size, is based on the test method described in the standard ASTM F 316-03.

The POROLUX™ 100 was designed with conformance to standards and manual methods in mind. It therefore has user selectable settings, which can be adapted to different criteria traditionally used for FBP calculations. Options include the calculation of the FBP at the pressure that corresponds to a flow rate of 30, 50, 100 ml/min, or taking the pressure at which the first measureable flow is detected by the equipment. This flexibility allows a user to find the settings optimal for a particular material, or for cross-correlation with other methods (e.g. visual determination).

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For bubble point determination of very porous samples, such as nonwoven it is recommended to choose “first flow” or “FBP” above a certain flow value”, because it is not always easy to calculate the FPB at one specific flow value (e.g. 30 ml/min). In each measurement, a total of 30 data points were taken in the pressure range from 0 to 100 mbar. Three replicates for each sample were measured. The FBP, mean flow pore (MFP) and smallest pore size (SP) of sample “A” (spunbond type material) and “B” (meltblown type material) are given in table 1:

Sample A	Average (µm)	SD (µm)	RSD (%)	Sample B	Average (µm)	SD (µm)	RSD (%)
FBP	375.8	2	0.6	FBP	70.5	0.1	0.2
MPF	82.8	6	7.5	MPF	19	1	6
SP	28.3	2	6	SP	7.3	0.3	3

For the calculation of the pore size distribution, it is also necessary to measure gas permeability – i.e. the flow rate across a dry material as a function of increasing pressure – a “dry curve”. Then, the “half-dry” curve is obtained by dividing the flow values of the dry curve by 2. The MFP (Mean Flow Pore) size is found at the pressure where the wet and the half-dry curves intersect. This point corresponds to the size at which 50 % of the total gas flow occurs through pores larger, and half the total gas flow through pores which are smaller (than the mean value). The SP – “smallest pore size” corresponds to the pressure where the wet and the dry curves meet – this is the highest pressure at which a pore is emptied. Due to the nature of the calculation of the MFP and the SP, the latter typically display a higher standard deviation than FBP values.

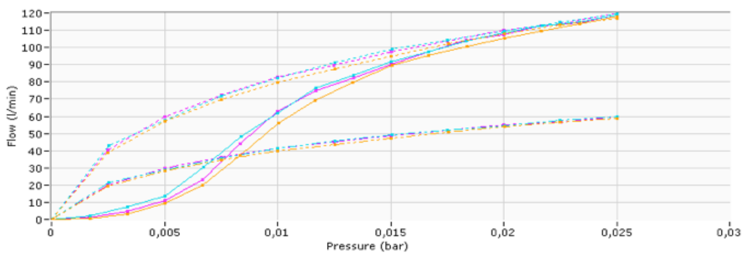

Figure 2: Wet, dry and half dry curves for material A

As an example, the wet, dry and half dry curves for sample “A” are shown in figure 2. The rounded shape of the dry curves (dotted lines) is typical of non-woven materials. It is readily apparent from below that small deviations in the “wet” flow curves yields large differences in intersection points of the half-dry curve (the MFP value)

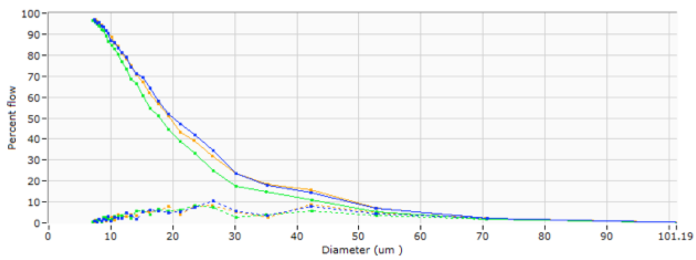

Figure 3: Cumulative and differential flow curves for material B

The gas flow through the sample is used to calculate the cumulative filter flow distribution against the pore size (percentage of the total flow which corresponds to pores of a certain size and larger) and the differential filter flow (increase in flow rate per unit increase in pore diameter, commonly defined as pore size distribution). The results for sample B are shown in figure 3.

Overall, GLP has proven to be a very useful tool for the characterisation of nonwovens. If you wish to know more about how the POROLUX™ 100 can help your nonwoven business, contact us at info@porometer.com.

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