

CHARACTERIZATION OF PORE STRUCTURE OF FILTRATION MEDIA CONTAINING HYDROPHOBIC AND HYDROPHILIC PORES

Akshaya Jena and Krishna Gupta
Porous Materials Inc.
20 Dutch Mill Road, Ithaca, NY 14850

ABSTRACT

There is no unique technique that can measure the pore structure characteristics of mixtures of hydrophobic and hydrophilic pores. In this investigation water intrusion porosimetry and mercury intrusion porosimetry have been used to explore measurement of the pore structure of filter material containing mixtures of hydrophobic and hydrophilic pores. It is possible to obtain information on the pore structures of both kinds of pores within the limitations of the technique.

INTRODUCTION

Filtration media containing hydrophobic and hydrophilic pores are finding applications in a wide variety of industries including biotech, health care, pharmaceutical and power sources. Surface treated filtration media often contain hydrophobic and hydrophilic pores. During use of filtration media in biochemical applications, some of the pores appear to change their hydrophobicity. Suitability of such products depends upon their pore structure characteristics. However, determination of pore structure characteristics of such filtration media is complicated. There is no technique that can be used for characterization of such structure. In this investigation, water intrusion porosimetry and mercury intrusion porosimetry were used to obtain information on the pore structure characteristics of hydrophobic and hydrophilic pores in a mixture.

TECHNIQUE

Water intrusion porosimetry is used for characterization of a material that has hydrophobic pores. Water cannot flow spontaneously in to such pores. When pressure is applied on water

surrounding the sample water intrudes into the pores. The pressure and intrusion volume are measured. The pressure gives pore diameter through the following equation and the intrusion volume gives pore volume.

$$D = - 4 \gamma \cos \theta / p \quad (1)$$

where D is pore diameter, γ and θ are surface tension and contact angle respectively of the liquid and p is differential pressure (Jena & Gupta 2002).

Mercury is nonwetting for most materials. It does not flow spontaneously into pores. Pressure is applied on mercury for it to intrude into the pores. Pore diameter is obtained from pressure using Equation 1 and pore volume is computed from the intrusion volume (Jena & Gupta 2002)

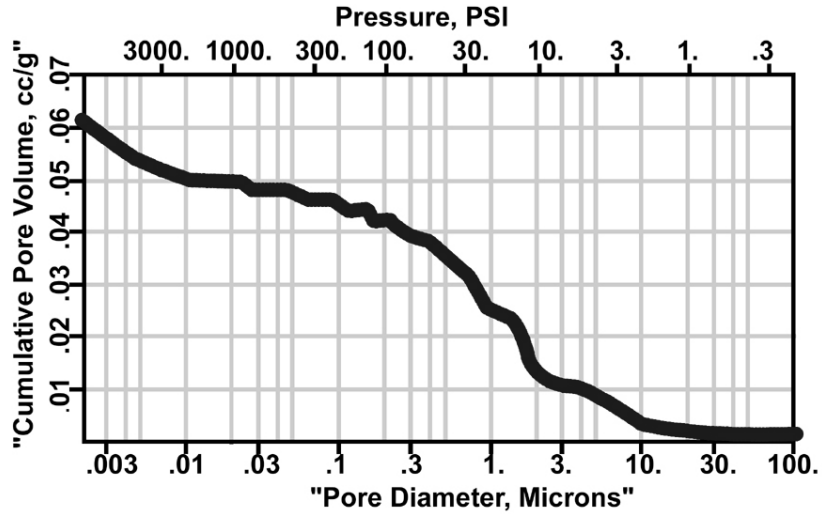
For capillary flow porometry a wetting liquid is used to fill all the pores of the sample and a non-reacting gas is used to remove the liquid from pores and permit gas flow. The differential pressures on the sample and gas flow rates through wet and dry samples are measured. The differential pressure is substituted in Equation 2 to obtain the pore throat diameter of through pores (Jena & Gupta 2002, Jena & Gupta 2000).

$$D = 4 \gamma \cos \theta / p \quad (2)$$

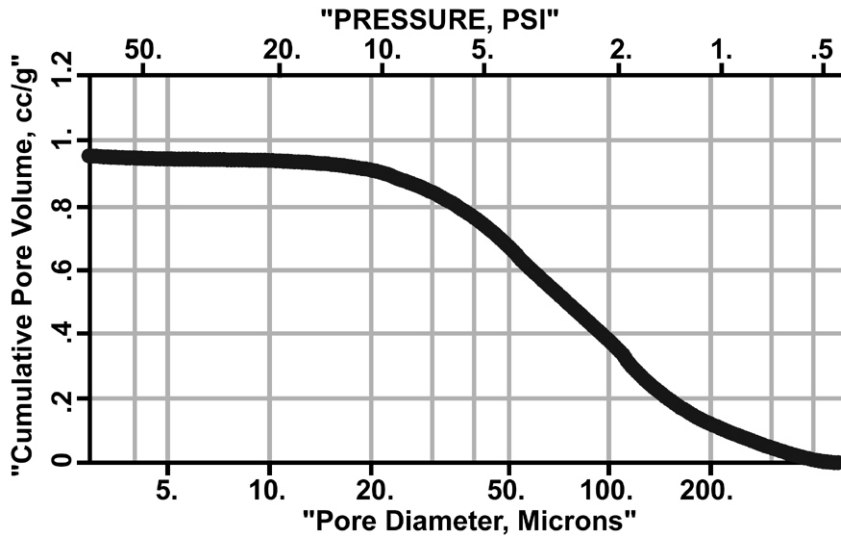
These measurements are used to compute the largest, the mean and the range of through pore throat diameters. Pore distribution, gas permeability and surface area are also determinable.

RESULTS AND DISCUSSION

The cumulative pore volume measured as a function of pore diameter in the water intrusion porosimeter is shown in Figure 1. This technique measures all the diameters associated with the pores along their lengths. The measured volumes and diameters are those of hydrophobic through and blind pores. The results are listed in Table 1. The pore volume and pore diameters of such hydrophobic pores are small.



a)



(b)

Figure 1. Cumulative through and blind pore volume measured as a function of pore diameter measured by (a) water intrusion porosimetry and (b) mercury intrusion porosimetry.

The change of pore volume with pore diameter measured by mercury intrusion porosimetry is shown in Figure 1. In this technique all diameters of through and blind pores along their length are measured. Both hydrophilic and hydrophobic pore volumes and diameters are measured. The results are listed in Table 1.

Table 1. Pore Structure Characteristics

Water Intrusion Porosimetry	Mercury Intrusion Porosimetry	Capillary Flow Porometry
Hydrophobic :Through & blind pores	Hydrophobic & Hydrophilic Through & blind pores	Hydrophilic & Hydrophobic: Through pores
Volume: 0.06 cm ³ /g	Volume: 0.97 cm ³ /g	
Diameter range: ~0.1–10 μm :	Diameter range ~30–300 μm	Throat diameter range: 1.85–73.81 μm The Largest: 73.81 μm
Median: 0.78 μm (Based on volume)	Median: 77.80 μm (Based on volume)	Mean: 19.74 μm (Based on flow):

The gas flow rates measured as a function of differential pressure in a capillary flow porometer are shown in Figure 2. A liquid that wetted both hydrophobic and hydrophilic pores was used. The wet and dry curves are generated using wet and dry samples. The half-dry curve is computed from dry curve to yield half of the flow rate through the sample at a given differential pressure. The pressure at which flow is initiated yields the largest through pore throat diameter, The pressure at which wet and half-dry curves intersect gives the mean flow pore diameter and the smallest pore detectable is obtained from the pressure at which wet curve meets the dry curve. The pore distribution is obtained from flow rates through wet and dry curves. Table 1 lists the data.

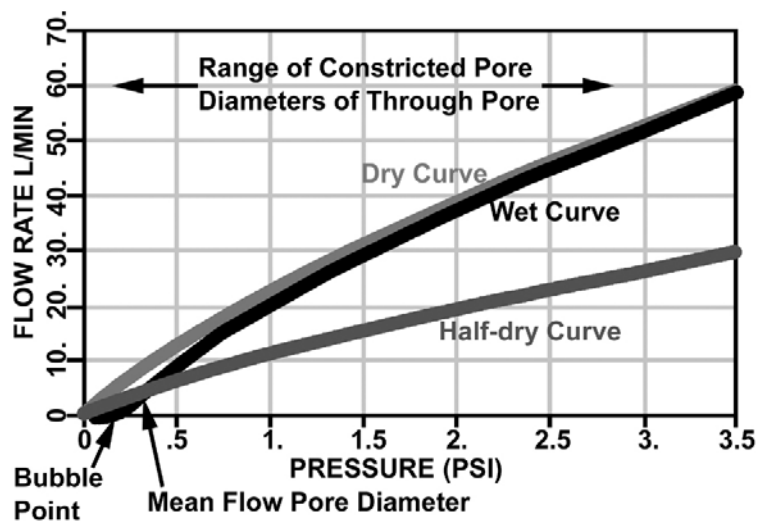


Figure 2. Flow rate measured as a function of differential pressure by capillary flow porometry.

Flow porometry measures the throat diameter of the through pore. One diameter for each through pore is measured (Figure 3). The other two porosimetry techniques measure all the pore diameters of through and blind pores (Figure 3). The through pores are important for barrier and fluid flow characteristics. .

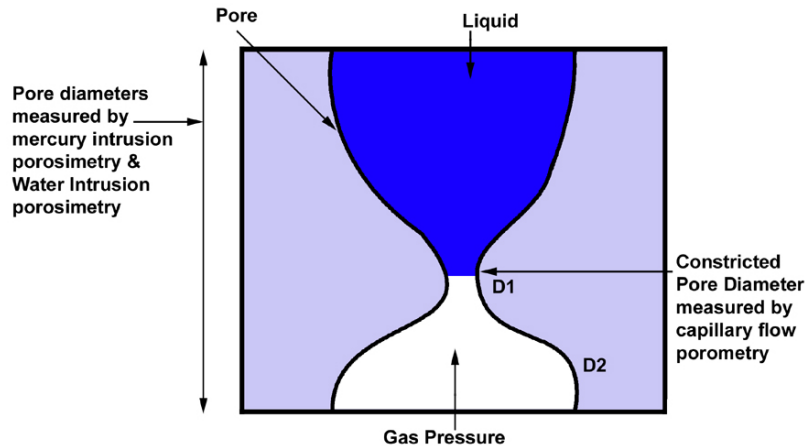


Figure 3. Measurable pore diameters.

The total pore volume measured by mercury porosimetry is $0.97 \text{ cm}^3/\text{g}$ compared with the pore volume of only $0.06 \text{ cm}^3/\text{g}$ measured by water porosimetry for the hydrophobic pores. The volume of hydrophobic pores is only 6 % and the volume of hydrophilic pores is 94 %. Thus, only a very small fraction of pores in this material is hydrophobic. The hydrophobic pores were not clearly detected by mercury porosimetry because of their very low volume in the material. Water extrusion porosimetry was sensitive enough to detect the small volume of hydrophobic pores.

The small pore diameters of hydrophobic pores measured by water porosimetry were in the range of $\sim 0.1\text{--}10 \text{ }\mu\text{m}$ with a mean of $0.78 \text{ }\mu\text{m}$: The diameters of large hydrophobic pores were in the range $\sim 30\text{--}300 \text{ }\mu\text{m}$ with a mean of $77.80 \text{ }\mu\text{m}$ The throat diameters of the hydrophobic and hydrophilic pores measured by flow porometry were in the range of only $1.85\text{--}73.81 \text{ }\mu\text{m}$. with a mean of $19.74 \text{ }\mu\text{m}$. This result implies that many of the large hydrophilic pores have narrow constrictions and wide mouths and the hydrophobic pores are mostly blind.

The material tested was fibrous. The hydrophilic pores in this material were through pores with large diameters and large pore volume. On the other hand the hydrophobic pores primarily small diameter blind pores with very small pore volume. It is postulated that the hydrophilic pores are present between the strands of fibers and the small hydrophobic blind pores are present within the strands of fibers. Suitable treatment and manufacturing process may result in such pore characteristics

CONCLUSIONS

A partially hydrophobic and partially hydrophilic fibrous material was characterized by water intrusion porosimetry, mercury intrusion porosimetry and capillary flow porometry. The analysis of results obtained using these techniques showed that the volume of hydrophobic pores was only 6 %. These pores had very small pore diameters compared with the hydrophilic pores. The hydrophobic pores are primarily blind. The hydrophilic pores were primarily through pores. It is suggested that the large hydrophilic through pores are between the strands of fibers and the tiny hydrophobic pores are inside the fiber strands. Although, the hydrophobic pores were very small and had very small pore volume, it was possible to detect these pores by water intrusion porosimetry. This investigation suggests the importance of water intrusion porosimetry for such investigations.

REFERENCES

- Jena, A. K. and Gupta, K. M., 2002, 'Characterization of *Pore* Structure of Filter Media, Fluid/Particle Separation Journal, Vol. 14, pp. 1.
- Jena, A, & Gupta, K., 2002, Analyse der Porendurchmesser von Mehrschichtfiltermitteln, *F & S Filtrien und Separieren*, Vol. 16, pp. 13.
- Jena, OA, & Gupta, K., 2000, 'In-Plane and Through Plane Porosity of Coated Textile Materials, *Journal of Industrial Textiles*, Vol. 29, pp. 317..
- .