CHARACTERIZATION OF DEPTH FILTRATION MEDIA

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ABSTRACT
A depth filtration medium was characterized using liquid extrusion porosimetry and capillary flow porometry. Through pore characteristics including pore volume, pore diameter, the largest throat diameter, the mean flow throat diameter, pore volume distribution and flow distribution were measured. Analysis of results suggested the presence of three groups of wide mouth pores of diameter of about 5.2, 3.6 and 2.2 μm and one group of pores with diameter < 2 μm.

INTRODUCTION

Modern depth filtration media are designed to optimize filtration performance. Such media are usually very complex. Important through pore characteristics of depth filtration media are pore volume, pore throat diameter, the largest pore throat diameter, mean flow pore throat diameter, pore volume distribution, gas permeability and liquid permeability. No single technique can measure all these pore structure characteristics. Adequate characterization is likely to require application of multiple techniques. The application of the two extrusion techniques for this purpose has considerable potential. In this study, the use of the two extrusion techniques; Liquid Extrusion Porosimetry and Capillary Flow Porometry for characterization of a depth filtration medium has been investigated.

LIQUID EXTRUSION TECHNIQUES

In liquid extrusion techniques, a wetting liquid is allowed to spontaneously fill the pores of the sample. The pores are filled spontaneously because the sample/liquid interfacial free energy ($\gamma_{s/l}$) is less than the sample/gas interfacial free energy ($\gamma_{s/g}$). The liquid cannot spontaneously come out of the pores. An inert gas under pressure can remove the liquid from pores. Consideration of energy balance shows that differential gas pressure required to remove liquid from a pore is given by the following relation [1].

$$p = \gamma \cos \theta \, (dS/dV) \qquad (1)$$

where
dV = increase in volume of gas displacing liquid in the pore
dS = increase in surface area associated with dV
p = differential gas pressure on the sample

γ = surface tension of liquid (liquid/gas interfacial free energy)
θ = contact angle of liquid

Pores normally have irregular cross-sections and undefined pore sizes. Pore diameter, D, of a pore at any location along its path is defined such that (dS/dV) at that location is equal to the (dS/dV) of a cylindrical opening of diameter, D.

$$(dS/dV)_{pore} = (dS/dV)_{cylindrical\ opening\ of\ diameter, D} \quad (2)$$

From Equations 1 and 2;

$$p = 4 \gamma \cos \theta / D \quad (3)$$

Liquid displaced from pores flows out and can be measured. Similarly, gas can flow through pores emptied by the gas and the gas flow rate can also be measured [2](Figure 1).

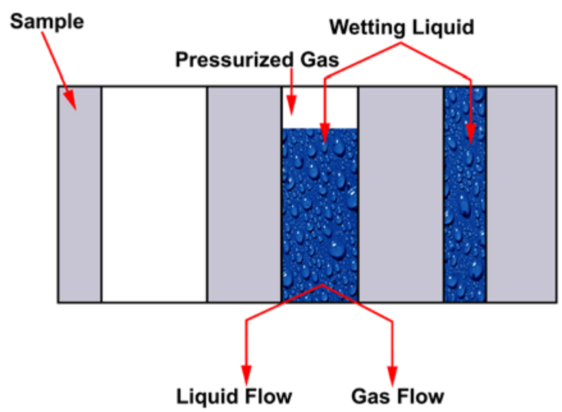


Figure 1. Principle of extrusion techniques

Liquid Extrusion Porosimetry

In this extrusion technique [3], the sample is placed on a membrane whose largest pore diameter is smaller than the smallest pore diameter of interest in the sample. The wetting liquid is allowed to fill pores of both the sample and the membrane. When gas under pressure displaces liquid from pores of the sample, the liquid flows through the liquid filled pores of the membrane. Because the gas pressure employed to displace liquid from pores of the sample is less than the gas pressure required to displace liquid from pores of the membrane, the membrane pores always remain filled with liquid and prevent flow of gas through the membrane pores. Thus, gas does not flow out through the membrane, but the extruded liquid flows through the membrane and either the volume or the weight of the displaced liquid is measured (Figure 2). For liquid permeability, the membrane is removed, gas pressure on excess liquid maintained on the sample is increased and liquid flow rate is measured (Figure 2). Thus, through pore volume, volume distribution, through pore diameter, through pore surface area and liquid permeability are measurable by this technique.

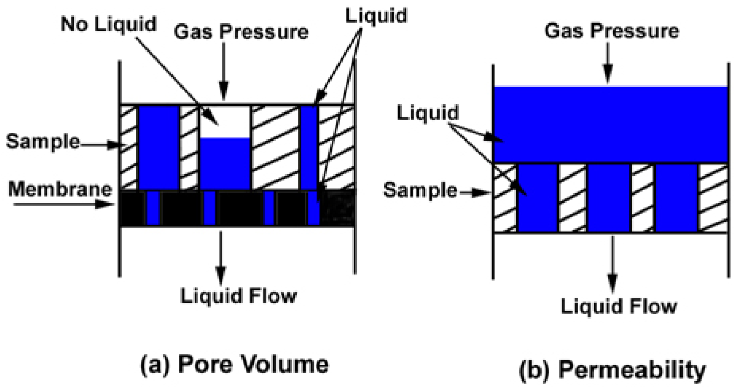
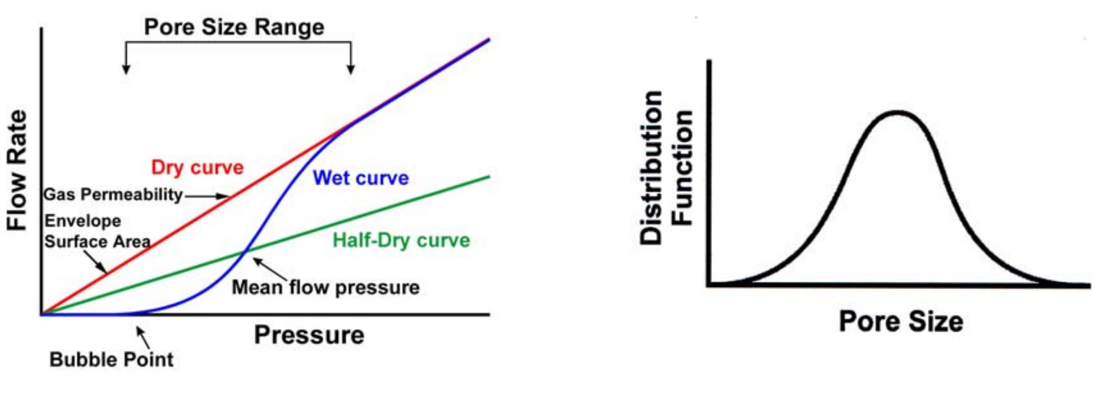


Figure 2. Principle of liquid extrusion porosimetry.

Capillary Flow Porometry

Capillary flow porometry is liquid extrusion flow porometry. In this technique the pressurized gas that flows out of the sample after emptying the pores is measured [4]. The differential pressure and the gas flow rates through the dry samples as well as through the empty pores of the wet sample are measured. The flow rate in the dry sample increases with increase in pressure. In case of the wet sample, initially there is no flow because all the pores are filled with the liquid. At a certain pressure the gas empties the largest pore (Equation 1) and gas flow starts through the wet sample. With further increase in pressure smaller pores are emptied and the flow rate increases until all the pores are empty and the flow rate through the wet sample is the same as that through the dry sample. This is schematically illustrated in Figure 3. The half-dry curve in this figure is computed from the dry curve to yield fifty-percent of flow through dry sample at the same pressure. The dry and wet curves yield the bubble point, the mean flow pore diameter, flow distribution and pore fraction distribution of through pores [5]. The dry curve yields gas permeability [6] and envelope (through pore) surface area [7] (Figure 3).



(a) Pore diameters, permeability & through pore surface area

(b) Flow distribution and pore fraction distribution

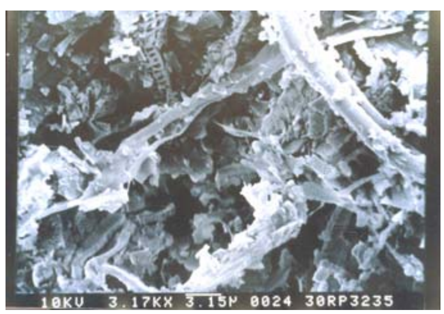
Figure 3. Through pore characteristics measurable by capillary flow porometry.

FILTRATION MEDIA

The dept filtration medium examined in this investigation consists of a matrix of highly fibrillated cellulose ribbon fibers and deposits of diatomaceous earth. Fibrillation of the cellulose opens up the fibers creating sub-micron, hair-like, filaments that form an integral, high surface area network which holds the diatomaceous earth securely in place (Figure 4). Diatomaceous earth is a non-metallic mineral composed of the skeletal remains of microscopic, single-celled, aquatic, microorganisms called diatoms. Diatoms are rigid, intricately shaped, highly porous and highly permeable mineral particles that when combined with fibrillated cellulose result in a medium with high dirt holding capacity, high flow rate, and low particulate release or shedding (Figure 4).

(a)Unrefined cellulose

(b) Fibrillated cellulose



(c) Fibrillated cellulose matrix containing deposits of diatomaceous earth

Figure 4. SEM micrographs of filtration medium and its components.

RESULTS

Variation of cumulative pore volume with pore diameter measured in the PMI Liquid Extrusion Porosimeter is presented in Figure 5. Galwick with surface tension, 16 dynes/cm was used as the wetting liquid. The contact angle was taken as zero [8]. The pore diameter is in the range of about 18 to 2 μm. The total pore volume is 0.68 cm^3/g.

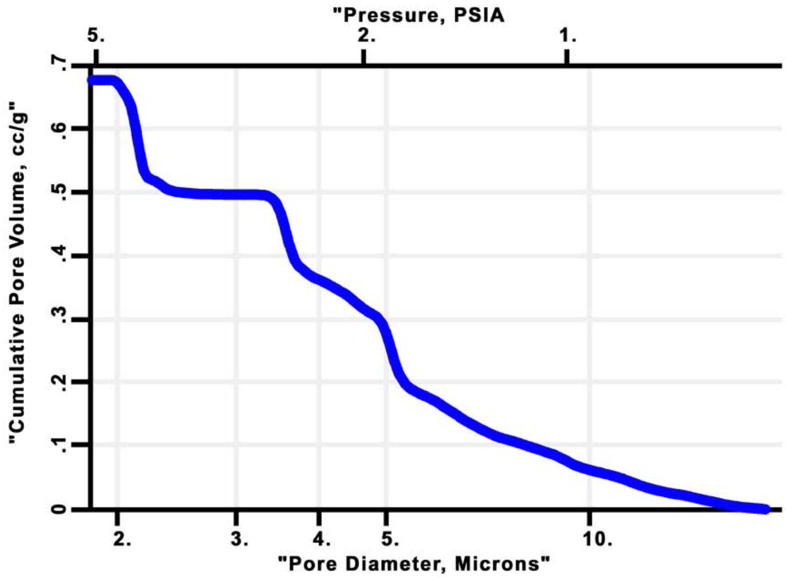


Figure 5. Cumulative pore volume and pore diameter of depth filtration medium measured by PMI Liquid Extrusion Porosimeter.

The variation of gas flow rate with differential pressure for depth filtration medium measured in the PMI Capillary Flow Porometer is presented in Figure 6. Galwick with surface tension, 16 dynes/cm was also used in this technique as the wetting liquid. The contact angle was taken as zero [8]. The dry curve, the wet curve and the half-dry curves are leveled. The pressure at which gas flow starts through the wet sample is indicated as bubble point pressure. The mean flow pressure in also indicated. Using these data various pore structure characteristics are computed.

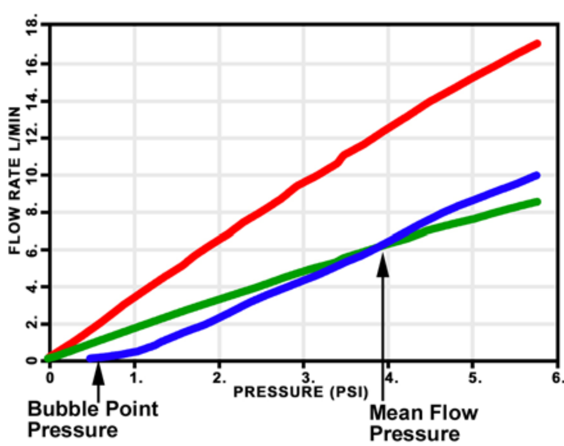


Figure 6. Variation of gas flow rate with differential pressure for dry and wet samples of the filtration medium measured in the PMI Capillary Flow Porometer.

DISCUSSION

Pore Diameters

Diameter of a pore generally changes along its path. Therefore, each pore is associated with many diameters (Figure 7). Consider displacement of liquid in the pore shown in Figure 7 due to gas pressure applied from the top in the liquid extrusion porosimeter. At low differential pressures, the gas will first displace liquid from the wider part of the pore at its top and the diameter and volume of this part of the pore will be detected. On increase of pressure, gas will displace liquid from the narrower part, and diameters and associated volumes of this part will be measured. The gas pressure that displaces the liquid at pore throat, will remove liquid from the rest of the pore, which is wider than the throat. Thus, the diameters of the pore from the top to the throat will be measured and the diameters of the pore below the throat will not be measured (Figure 7).

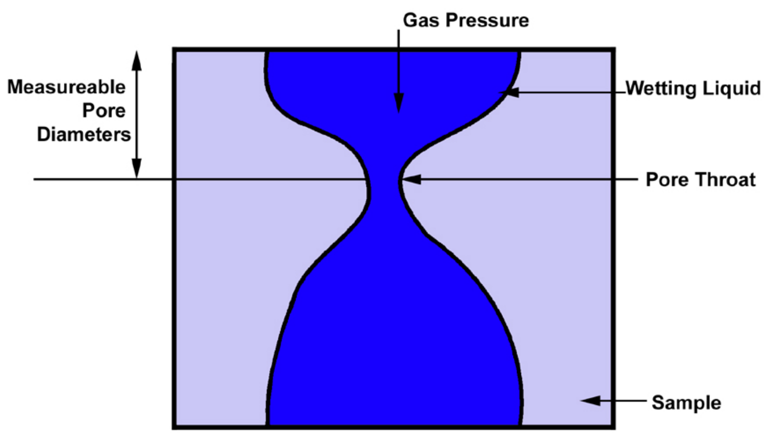


Figure 7. Pore diameters measurable by liquid extrusion porosimetry.

The pore diameters are along x-axis in Figure 5. The largest pore diameter of about 18.5 microns represents diameter of the widest mouth of pore and the smallest pore diameter of about 2 microns represents the narrowest detectable pore throat.

Pore Throat Diameters

Flow porometry senses the presence of a pore at a particular applied differential pressure by detecting increase in gas flow due to flow through the pore. All the liquid from a pore is emptied and gas starts flowing through the pore, when the differential pressure is sufficient to displace liquid from the most constricted part of the pore. Therefore, the pore diameter computed from the differential pressure is the diameter of the pore at its most constricted part or the pore throat (Figure 7).

Flow porometry measures only the throat diameter of each through pore (Figure 7). None of the other diameters of the pore is measured. The largest through pore throat diameter (bubble point pore diameter) and the mean flow through pore throat diameter are listed in Table 1.

Table 1. Throat diameters

	Bubble point	Mean flow	Range	Appreciable flow
Diameter, µm	18.59	2.37	18.59 - <1.7	<8

Pores present in appreciable numbers will result in appreciable flow. The flow distribution over diameter is presented in terms of the function, f.

$$f = - \ d [(Fw/Fd)\times 100] / d D \tag{4}$$

Where Fw and Fd are flow through wet and dry samples respectively at the same differential pressure. Figure 8 is a plot of the distribution function against pore diameter. The function is such that in the plot of distribution function against diameter, the area under the function in any pore diameter range is the percentage flow through that range.

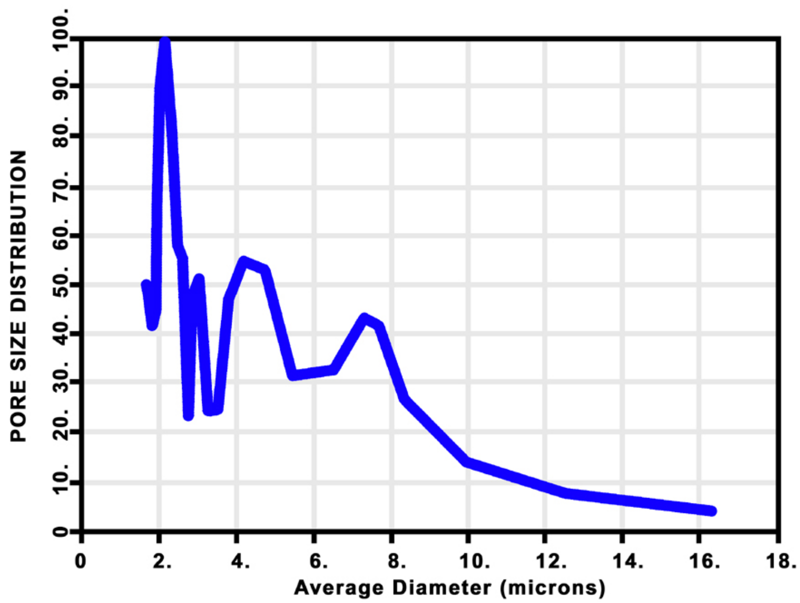


Figure 8. Flow distribution over pore throat diameter

The variation of the distribution function suggests that there are few pores with large throat diameters. Pores with throat diameter > ~8 µm are not appreciable (Table 1). However, there are many pores with throat diameter less than ~1.7 µm (Table 1). The bubble point pore diameter is close to the maximum pore diameter measured by liquid extrusion porosimetry.

Volume of Different Diameter Pores

The total pore volume is 0.68 cm^3/g and porosity is 19 %. The distribution of pore volume over diameter is expressed in terms of the distribution function, fv.

$$fv = - (dV/d \ln D) \tag{5}$$

The distribution function is shown in Figure 9. The function is such that area under the curve in any pore diameter range is equal to the volume of pores in that diameter range.

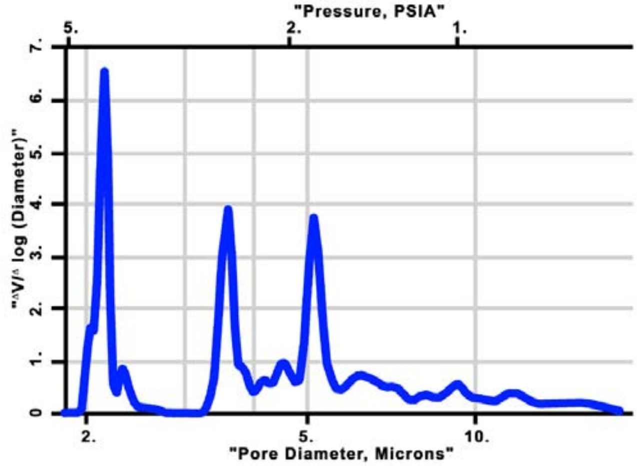


Figure 9. Pore volume distribution

The pore volume distribution over pore diameter expected for a pore like the one in Figure 7 is shown in Figure 10. The pore volume decreases as the pore diameter decreases and at the throat diameter the pore volume becomes large because at the pressure required to displace liquid from the throat, the entire liquid below the pore is emptied. Thus, pore throat diameter is associated with large volume for wide mouth pores. If the pore does not have a wide mouth the pore throat with its low volume may not be detectable.

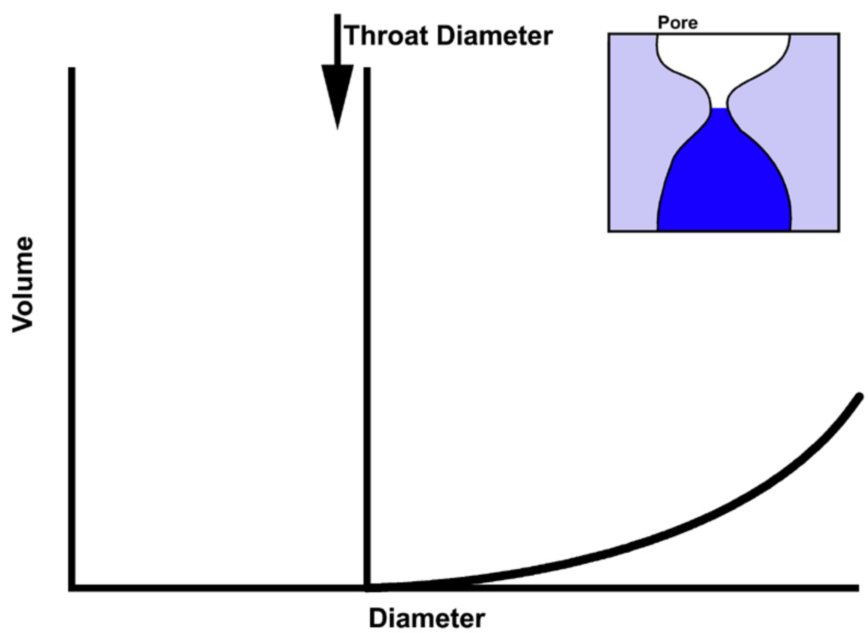


Figure 10. Expected Volume distribution over pore diameter of a wide mouth pore measured by liquid extrusion porosimetry

The measured pore volume distribution over pore diameter (Figure 9) is similar to the expected variation for wide mouth pores (Figure 10). The distribution suggests the presence of three sets of pore throats of diameter of about 5.2, 3.6 and 2.2 μm. Such three sets are also suggested by data obtained by flow porometry (Figure 8).

The presence of many narrow throat diameters suggested by flow porometry is not detected by liquid extrusion porosimetry because the pore volume associated with these pores is very small. Most likely these pores are not associated with wide mouths.

The bubble point measured by flow porometry is close to the maximum pore diameter measured by liquid extrusion porosimetry. This result implies that very few pores have mouths wider than the bubble point pore diameter so that their volume is very small to be detectable. Because wide parts are associated with pores with largest throat diameters, these are the first pores to be detected by extrusion flow porosimetry. The probable pore structure of depth filtration media is illustrated in Figure 11.

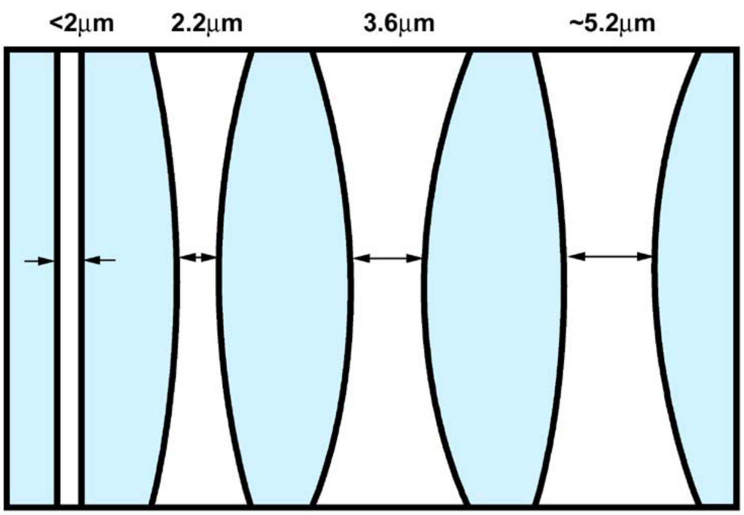


Figure 11. Probable pore structure of the depth filtration media

The pore characteristics of filtration media are consistent with the structure of the media. The three sets of detected large pores may be associated with the fibrillated fibers and the set of small pores may be due to the diatoms.

SUMMARY AND CONCLUSION

1. The pore structure of a depth filtration medium was characterized using liquid extrusion techniques;

    Liquid Extrusion Porosimetry

    Capillary Flow Porometry.

2. Pore volume, pore diameter and pore volume distributions of through pores were measured by Liquid Extrusion Porosimetry.
3. The largest throat diameter, the mean flow throat diameter and flow distribution through pores were measured by Capillary Flow Porometry.
4. Analysis of results suggests that the depth filtration medium contains three sets of wide mouth pores of diameter of about 5.2, 3.6 and 2.2 μm and another set of pores having diameter less than 2 μm.
5. The two techniques did not use any toxic material.

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