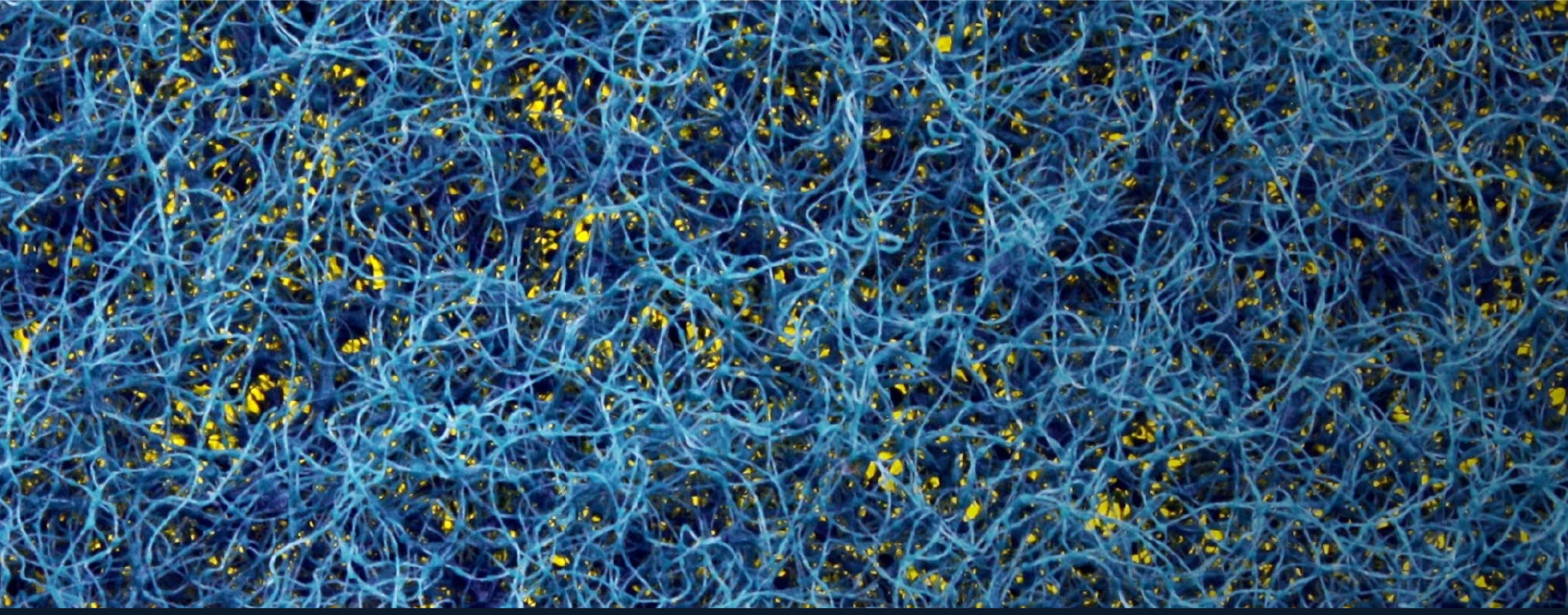


Covalent Academy Episode 25 Q&A

Porometry, Porosimetry, and Pycnometry: the 3 P's You Need for Porous Materials Characterization

Presented By: ***Nanette Jarenwattananon, PhD***

Senior Manager, Material Property Analysis and Analytical Chemistry
Covalent Metrology

01

Q: Does it matter what gas is used for Porometry?

For capillary flow porometry, an inert gas is typically utilized. ASTM F316 ("Pore Size Characteristics of Membrane Filters by Bubble Point and Mean Flow Pore Test") only requires a clean gas such as filtered air or nitrogen.

02

Q: Can Covalent analyze hollow fibers using its Porometer? If not, can a hollow fiber be analyzed in a sealed cell?

There is an attachment available for our porometer to do his type of work, but the hollow core fiber needs to be sealed. Please see the following applications note:
Measuring Pore Size in the Walls of Hollow Fiber Membranes :: Anton-Paar.com

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Q: What wetting fluid is typically used to wet samples for Capillary Flow Porometry?

When considering a wetting fluid, there cannot be any chemical or physical interactions with the sample under test. The sample cannot dissolve, swell or reaction with the wetting fluid. Chemically inert solutions such as fluorocarbons (FC) with complete wetting behavior (contact angle of zero with all solids) are commonly utilized. Different FC's have different vapor pressures. Higher vapor pressure FC's are useful when running a "wet before dry" porometry analysis because the reduces the change of through-pores being re-wetting by blind pores. However, FC's with high vapor pressure can prematurely evaporate from still-full pores due to a high gas flow rate. In this case, low vapor pressures FC's can be used for a "dry before wet" run. Other liquids include mineral oil, isopropanol, ethanol, silicon oil, and water.

FLUID	BENEFIT
Porofil (Fluorocarbon)	Good universal wetting fluid
Galdfil (Fluorocarbon)	Similar to Porofil, but with a lower vapor pressure
Ethanol	Good for bubble point only because it has a high vapor pressure
Isopropanol	Good for bubble point only because it has a high vapor pressure
Mineral Oil	Good, but with limited wetting capability
Silicon Oil	Good, with very little vapor pressure
Water	Good for hydrophilic materials

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Q: What are the required properties of wetting fluids selected; or, do properties of wetting fluids affect gas displacement?

The wetting fluid chosen determines the attraction between the fluid and the pore wall and can affect the overall measurement. Please see the answer to Q3 for more information.

05

Q: What is the advantage of Mercury Porosimetry over Gas Porosimetry?

Gas porosimetry covers a pore range of 0.35 nm to >100 nm. Mercury porosimetry can capture a larger range of pores, from 3.2 nm to larger than 500 microns.

06

Q: How are the pore properties of thin films characterized?

Krypton gas adsorption at 87K is often utilized for surface area, micropore and mesopore (up to 10 nm) size distribution of thin film materials. Other methodologies include radiation-based techniques such as small angle X-ray scattering (SAXS), grazing-incidence small-angle scattering (GISAXS), ellipsometry, or X-ray/neutron porosimetry. Quantification of porosity in thin films can also be done via elemental analysis techniques such as energy dispersive X-ray spectrometry (EDX) or Rutherford backscattering spectroscopy (RBS). Positron annihilation lifetime spectroscopy (PALS) has also been used to determine pore size distribution and degree of pore filling in closed pore systems.

07

Q: Does through-pore length impact Porometry? (i.e., for two materials with the same limiting pore size / diameter, but differing pore lengths, will a Capillary Flow Porometer instrument measure the same mean pore size?)

Yes unless the pinch point is very close to the average pore diameter.

08

Q: For Capillary Flow Porometry, does the sample need to fully seal the 25 mm disc?

If the sample does not fully seal the chamber, the pressure/flow characteristics are not correct as gas is shunted around the sample. At Covalent, have Universal sample holder for samples with the following diameters: 10 mm, 18 mm, 25 mm, 37 mm, 47 mm, and 50 mm. For some samples, it is possible to seal the area surrounding the sample with a non-porous adhesive. There are also special use sample holders for hollow fibers, tubular samples, and in-plane pores (transverse pores within a material).

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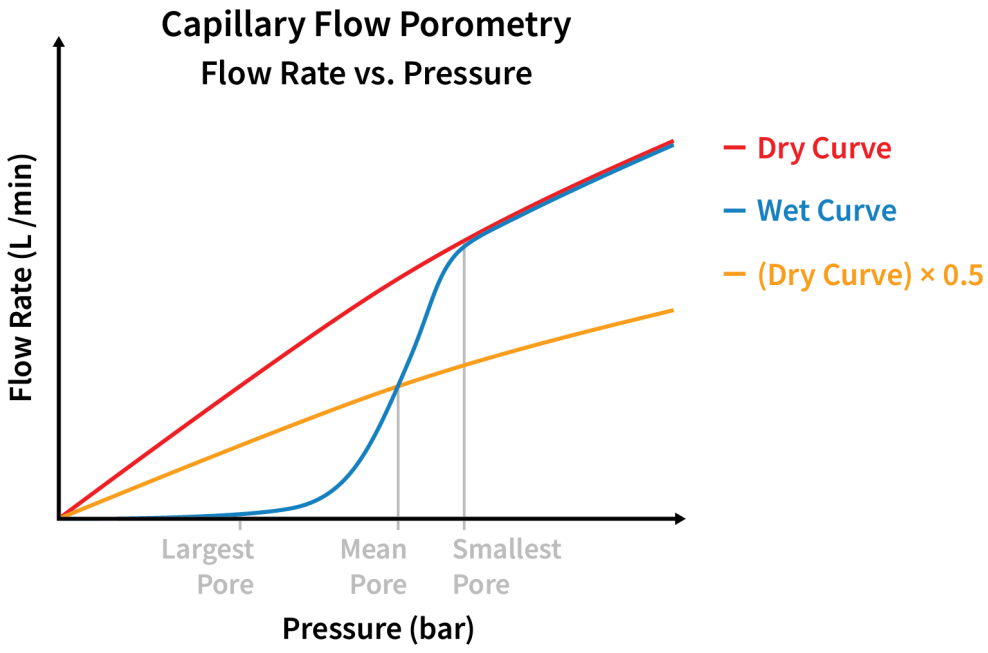
Q: Can granular samples be analyzed with Covalent's Capillary Flow Porometer? If not, what technique should be used?

If packed granules, gas or mercury porosimetry could be utilized.

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Q: When analyzing very small, closed micropores: can density measured with Pycnometry be compared to the density measured with Porosimetry to get an idea of pore volume?

Closed pores are isolated from the sample's external surface and cannot be accessed by external fluids; thus we cannot measure them directly with pycnometry or porosimetry. However, we can estimate the closed porosity (the volume of closed pores within a material) by comparing true density results (from pycnometry) with the expected density of the based material.



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Q: What technique would Covalent recommend for a sample with a highly asymmetric pore structure?

It depends on what material property is of interest, as well as the pore sizes in the material. Methods such as SEM, TEM, micro-CT, and confocal laser microscopy can be employed to image pore morphology and structure. Calculations in porometry and porosimetry make some assumptions about linear, cylindrical pores; however, they can still be employed for analysis of other materials. Ultimately it depends on the material: its pore sizes, and what the property of interest is.

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Q: What is "Mean Flow Pore"?

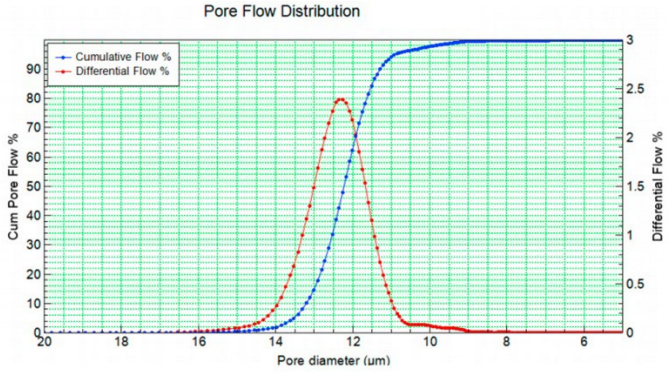
A dry curve is generated by plotting the airflow against the dry material vs. applied gas pressure. A second curve is plotted by dividing the dry material's measured air flow at each pressure by 2; it assumes that airflow is ½ that of the dry curve ("half dry curve"). Next, the material is saturated with wetting fluid and airflow through the material is measured as a function of gas pressure.

The largest pores empty first, defining the "Maximum Pore Size" (Max PS) and bubble point. The "Minimum Pore Size" (Min PS) is defined as the point where the wet curve meets the dry curve. The "Mean Pore Size" or "Mean Flow Pore Size" corresponds to the pore size calculated at the pressure where the wet curve and half-dry curve meet.

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Q: What is "Cumulative Pore Volume"? How does it relate to "Mean Flow Pore"?

Strictly speaking, "pore volume" and "cumulative pore volume" cannot be obtained from a porometry measurement (porosimetry needs to be performed). In a porometry experiment, we measure pressure P, from which we can calculate pore size, or radius r (from the Washburn equation). The "cumulative flow curve" is a percentage of the flow that has passed through pores of a certain size or larger. "Differential flow" is an increase in flow rate per unit increase in pore radius (first derivative of cumulative flow). The "mean flow pore (width)" is the pore width at which 50% of the flow is through pores larger than this width, and 50% of the flow is through pores smaller than this width.



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Q: How does material porosity affect adsorption?

Porous materials can adsorb more gas than non-porous/dense materials. The desorption temperature of the gas can also depend on the porosity of a material.

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