Bio-Technology R&D:

Performance Enhancement & Innovative Product Development Through Pore Structure

Pore Structure Characteristics Governing Performance

Innovative bio-technical products are currently being increasingly and widely used in health care, medical, and pharmaceutical industries. Some of the examples include substrates for tissue growth, drug delivery systems, implants, wound dressings, arterial grafts, filters for separation of bacteria from body fluids, and substrates for organ culture. Performance of all such products is dependent on their pore structure characteristic, because pore structure governs the flow and the kinetics of the bio-chemical processes which occur in the bio-technical applications.
For example, implants must have certain critical pore size for the blood vessels to grow during tissue growth. Pores, smaller or larger than the critical pore, discourage blood vessel growth and hinder tissue culture.

Pore structure characteristics important and relevant for bio-technical applications are pore diameter, through pore throat diameter, pore distribution, pore volume, porosity, surface area, hydrophobic and hydrophilic nature of pores in a mixture, gas and liquid permeability, water vapor transmission rate, and diffusional flow. During applications, the chemical environment, the operating temperature, the humidity, and the stresses generated during the operation can considerably alter the pore structure characteristic. Therefore, not only the determination of pore structure characteristic is important, evaluation of the influence of stress, temperature, and chemical environments on structural characteristics is also relevant.

Techniques for Measurement of Pore Structure Characteristic

Following techniques can be used for evaluation of pore structure characteristics relevant for bio-technical applications.

Liquid Extrusion Porosimetry

Measures through pore volumes, diameters, and distribution over a range of temperatures, stresses, and chemical environments including variable humid atmospheres. Can measure hydrophobic through pores as well as hydrophilic through pores. In a mixture, the characteristics of hydrophobic and hydrophilic through pores can also be determined.

Capillary Flow Porometry

Measures a wide range of through pore throat diameters, pore distribution, and gas permeability over a range of temperatures, stresses, and chemical environments including variable humid atmospheres.

Permeametry
Measures gas, vapor, and liquid transmission rates of many chemical species over a wide range of temperatures, pressures, and concentrations.

Water Vapor Transmission Analysis
Measures water vapor transmission rate as functions of humidity gradient, temperature gradient, and pressure gradient.

Vacuapore
It is a water intrusion porosimeter and it measures through and blind pore hydrophobic pore volume, diameter and distribution. Characteristics of hydrophobic and hydrophilic pores in a mixture can be determined in combination with mercury intrusion porosimetry.

Mercury Intrusion Porosimetry
Measures through and blind pore volume, diameter and distribution.

BET Sorptometry
Measures surface area, very small through and blind pore volume, distribution, chemisorptions of many chemicals over a wide range of temperatures and pressures.

Pycnometry
Measures true and bulk density of materials.

How Can PMI Help?

Porous Materials, Inc. is a company that designs, manufactures, and sells instruments for pore structure characterization. The company has vast experience in patenting of novel technology for pore structure characterization and developing custom instruments for a wide variety of applications. We have published many papers and presented many oral and poster papers in conferences on pore structure characterization of components used in biotechnology. Some of these papers will be found in our website. We have supplied instruments to many biotechnology industries and research laboratories all over the globe.

Our **Liquid Extrusion Porosimeter** and **Advanced Capillary Flow Porometer** are specially designed for biotech industry. **All other instruments** needed for pore structure characterization of in biotechnology are also designed and manufactured by us. The products developed by biotechnology are often tiny. PMI instruments have the unique ability to test very small component that are less than a few tenths of a millimeter in size. Some of the components can retain their integrity in only certain specific environments. PMI instruments have the ability to operate over a wide range of temperature, pressure, and chemical environments.
In all our instruments, test execution, data acquisition, data storage, and data reduction are fully automated. Windows-based menu driven operation of the instrument is very simple to use. The results are objective, accurate, and reproducible. Report generation is also highly versatile.

We also have our in-house contract testing laboratory. We have tested biotech components of many customers from a wide variety of organizations including biotech companies, universities, and research organizations.

Example of Application of PMI Instruments:

Pore Structure Characterization of Substrates for Tissue Growth and Culture

The PMI pore structure characterization instruments measured the very interesting pore structure characteristics of substrates used for tissue growth and culture.

Through Pore Volume & Distribution

The instruments used for these measurements are summarized below.

(i) Volume of the through pores: Measured by **PMI Liquid Extrusion Porosimeter**

(ii) Volume of through and blind pores: Measured by **PMI Mercury Intrusion Porosimeter**

Pore volume (Figure 1) and pore distribution (Figure 2) demonstrate interesting characteristics.

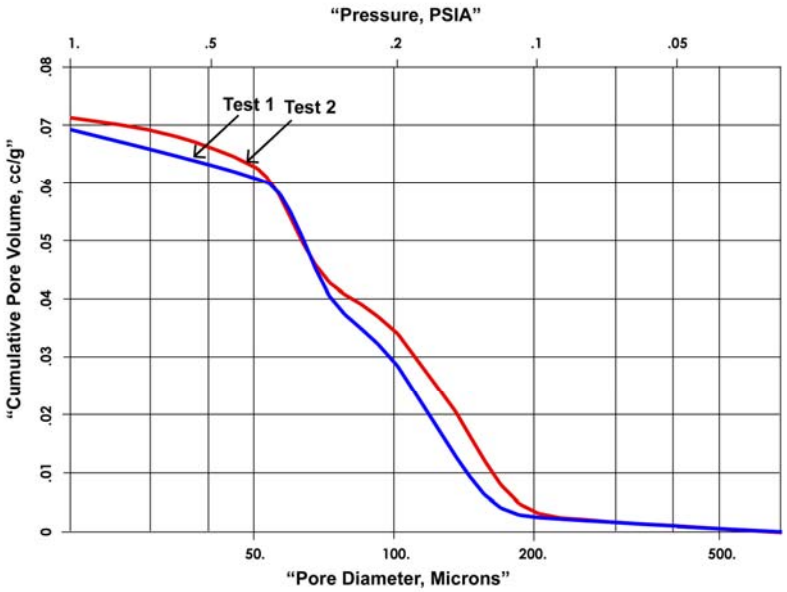


Figure 1 Pore volume of sintered metal implant

- Only through pores relevant for biotechnology measured
- Large 500 μm pores measurable
- Highly repeatable

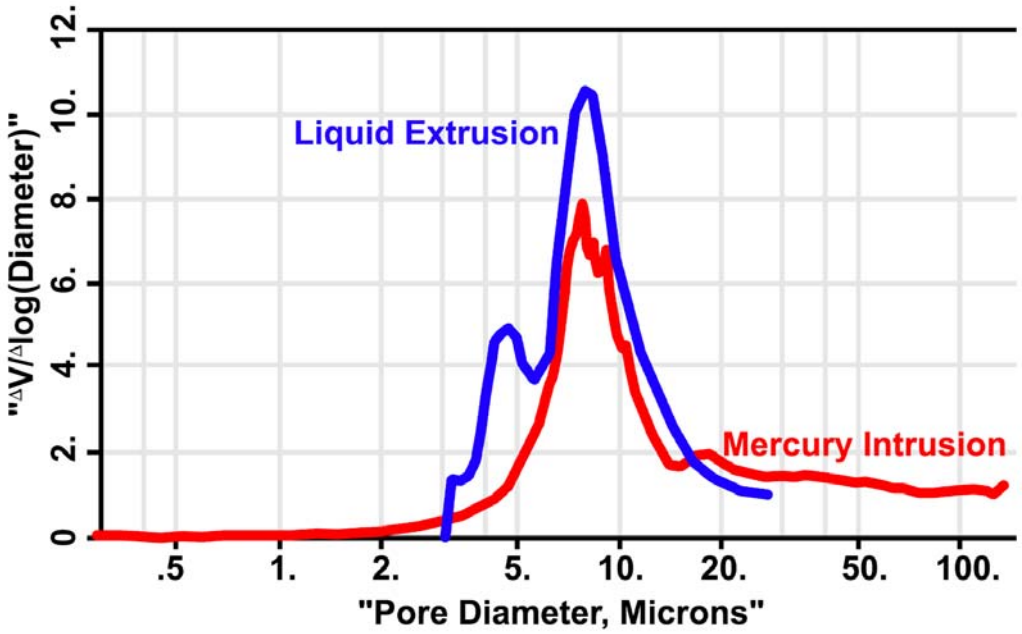


Figure 2. Pore volume distribution in a nonofiber substrate
(Distribution due to mercury shown for comparison)

- Much higher resolution by liquid extrusion technique
- Expected identical pore volume by both methods (No blind pore in nanofiber substrate)
- Expected high Porosity, 81.7 %
- Extrusion Technique completely avoided high pressure & toxic mercury

Characteristics of Through Pores Throat diameters

Through pore throat diameters determine nutrient and drug permeation rate and govern separation of entities like bacteria from body fluids, drugs, drinking water, food and beverages. Characteristics of pore throat diameters of through pores were determined by **PMI Capillary Flow Porometer.** This technique measures the largest pore throat diameter, the mean flow pore throat diameter, and the pore distribution. The pore throat distribution is shown in Figure 3.

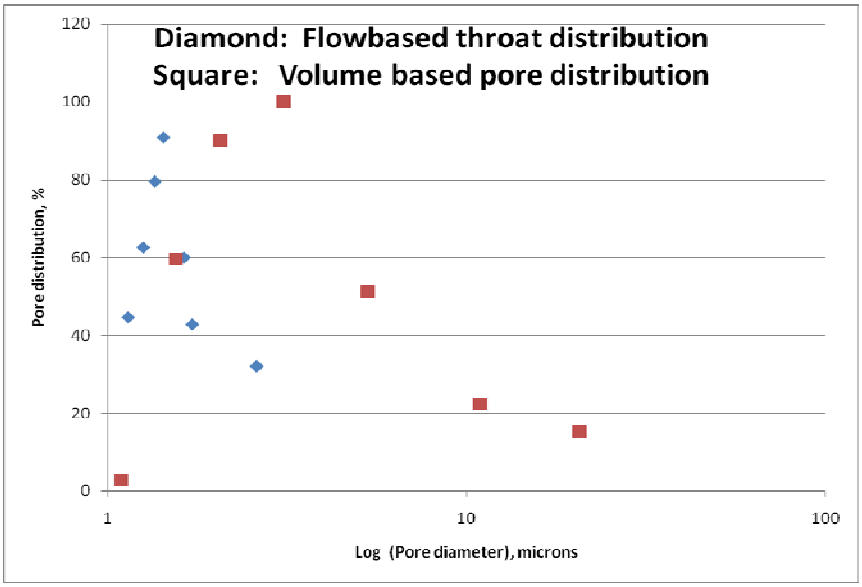


Figure 3. Distribution of through pore throat diameters in a thin nanopore substrate

- Narrow distribution demonstrated by both techniques (The largest throat diameter is 2.91 μm and the mean is 1.65 μm)
- The flow based mean throat diameter (1.65 μm) is close to the volume based median pore diameter (3.27 μm)
- Results suggest inappreciable change in pore diameter along pore path

Liquid Permeability

Tissue in-growth occurs in fluid accessible passages. Low permeability samples show very little tissue in-growth. The **PMI Liquid Permeameter** was used to measure liquid permeability. Figure 4 shows excellent tissue in-growth in a high permeability substrate.

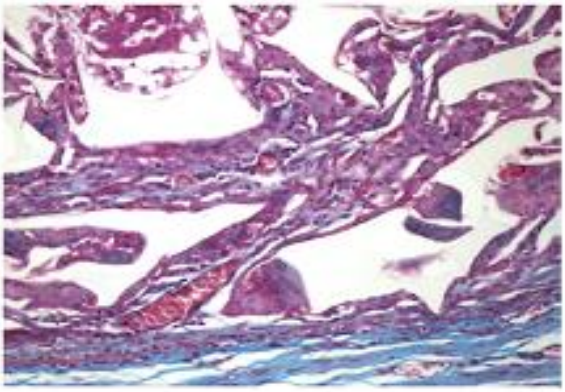


Figure 4 Tissue in-growth in high permeable reticulated foam substrate

Results of liquid permeability were combined with the mean flow pore diameters measured by **PMI Capillary Flow Porometer.** Figure 5 shows the dependence of tissue in-growth on the ratio of liquid permeability and mean flow pore diameter.

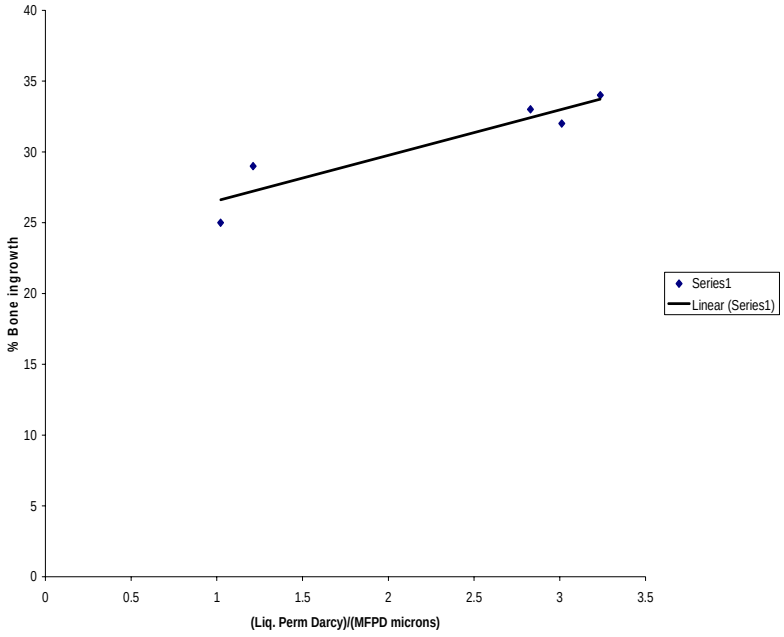


Figure 5. Correlation between percent tissue in-growth and ratio of liquid permeability and mean flow pore diameter for an implant

Special Needs

You can visit our website to see the wide spectrum of pore structure capability that our instruments offer. If you have a special need that is not part of PMI's standard test list, the engineers at PMI will be happy to talk to you about your testing requirements and come up with a suitable solution. Please free to contact us:

Porous Materials, Inc.
20 Dutch Mill Rd
Ithaca, NY 14850

Ph: 607-257-5544
Toll Free (US & Canada only): 1-800-825-5764
Fax: 607-257-5639

Online: www.pmiapp.com
Email: info@pmiapp.com

Porometers

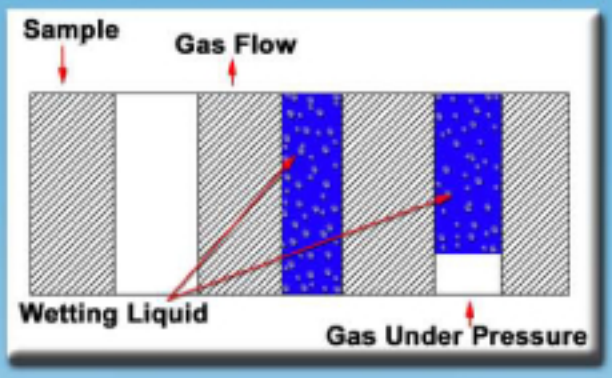
Porous Materials, Inc
20 Dutch Mill Rd
Ithaca, New York 14850 USA
Toll Free in Canada and USA: 1-800-TALK-PMI
Phone: (607) 257-5544 Fax: (607) 257-5639
Email: info@pmiapp.com

Principle

A wetting liquid spontaneously fills the pores of a sample by displacing the gas present in the pores. Application of differential pressure of a non-reacting gas on the sample is required to remove the wetting liquid from pores. The differential pressure p, required to remove the wetting liquid from a pore of diameter D, is given by:

$$p = 4\gamma \cos\theta / D$$

Where γ is the surface tension of wetting liquid and θ is the contact angle of the wetting liquid on the sample.Gas flow through the sample starts after the largest pore in the sample is emptied. The flow rate increases with increase in differential pressure. Pressure and flow rates of fluids through wet and dry samples are measured. Such measurements are used to compute various pore characteristics.



Unique Distinguishing Features

Hardware

- Sample chamber for almost any sample geometry like sheets, plates, discs, rods, tubes, hollow fibers, cartridges, powders, gels, pen tips & sponges.
- See-through sample chamber for visual confirmation of test.
- Multiple sample chamber for simultaneous testing of many samples.
- Mobile sample chamber for avoiding cutting of samples from the bulk product.
- Use of chemical resistant construction material for testing with strong chemicals like potassium hydroxide, phosphoric acid and salt solutions.
- Robust construction for use in industrial environments.
- Hardware option to permit measurement of pore volume and liquid permeability.
- Hardware for application of controlled compressive stress on the sample.
- Hardware for applying cyclic compression on the sample.
- Test temperature variable from sub ambient to 300 °C.
- Liquid permeability test at pressures up to 200 psi.
- Testing of samples saturated with chemicals.
- Options for tests performed under controlled humidity environment.

Automation

- Execution of test, data acquisition, data storage and data reduction are automated.
- User friendly interface and menu driven Windows based software make operation very simple.
- Number of data points is user definable.
- Real time status and results of test in progress are graphically displayed.
- Results can be plotted in many useful user specified formats.
- In research mode, user has the option to conduct tests by specifying parameters relevant to the investigation.
- In QC mode, operator involvement is minimal, and test duration is small.

Reliability & Validity

- Pore sizes determined in PMI porometer are in excellent agreement with those measured in Scanning Electron Microscope.

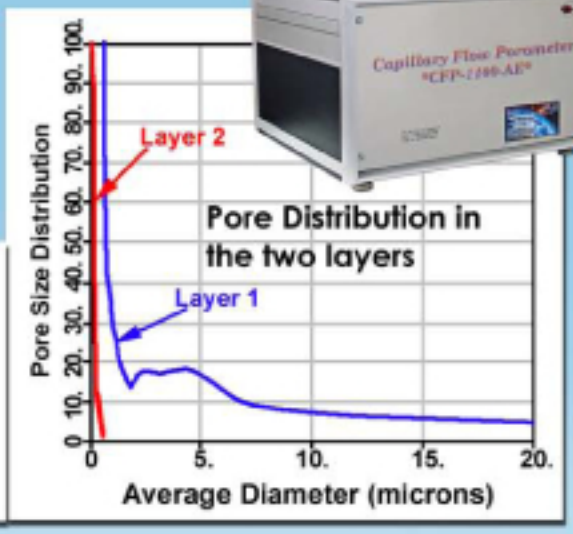
Etched Circular pore diameter, µm	
SEM	POROMETER
87.1 ± 5.2	86.7 ± 4.1
4.5 ± 0.5	4.6 ± 0.1

- Published data based on thirty repetitions show better than 1% repeatability.

In-Plane Porometer

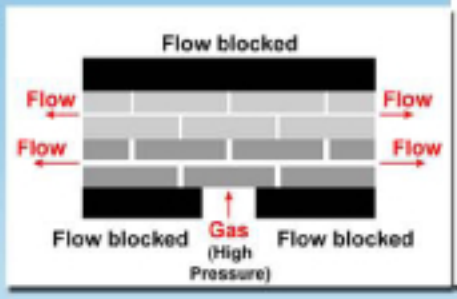
Operation

The In-Plane Porometer is such that gas moves radially from the center to the periphery of a sheet shaped sample. Both wet and dry samples are used Pore characteristics responsible for flow in the plane of the sheet material are computed.



Application

In-Plane pore structures are important for applications of products like paper, separators, textiles, electrodes, pharmaceuticals, biotech products and felts. This instrument is also used for in-situ pore structure characterization of individual layers of multi-layer products.



Average Fiber Diameter Analyzer

Operation

The average fiber diameter analyzer measures flow rate and differential pressure across a dry sample and computes the average fiber diameter using the relation by Davies (C.N. Davies, Proceedings of the Institute of Mechanical Engineers, London. 1B. 1952, pp.185-194)

Application

Determination of average fiber diameter takes only a few minutes in this completely automated instrument that is simple to use, robust and inexpensive. The instrument is suitable for quality control and production control.

Envelope Surface Area & Average Particle Size Analyzer

Operation

The instrument measures flow rate through a dry sample as a function of differential pressure and computes external surface area (surface area of through pores in a porous material) using Carman-Kazeny equation. The average particle diameter, D is computed using the following relation.

$$D = 4V/S = 4/S\rho$$

Where V is the volume of solid per unit mass, S is the envelope surface area per unit mass and ρ is the absolute density of powders. Results compare well with those by gas adsorption. Envelope Surfae Area Analyzer: 0.56 m^2/g and Gas Adsorption: 0.52 m^2/g

Application

The envelope surface area analyzer takes only minutes to perform a test, is fully automated, requires very little maintenance and is robust and inexpensive. One of its important applications is in quality control and process control.

QC Porometer

Operation

QC porometer is a capillary flow porometer incorporating special design features so that it is simple to operate, requires very little operator involvement, is user friendly, takes only a few minutes per test, is compact, and requires very little maintenance.

Application

The unique features of the instrument, especially its ability to generate highly reproducible data quickly, make it highly suitable for quality control and process control operations. This instrument is used in these applications in a wide variety of industries.

On-Line Porometer

Operation

The On-Line porometer is a capillary flow porometer containing a unique mobile sample chamber. The sample chamber can be attached to the area of the bulk sample to be tested or the sample could be made to slide through the sample chamber. Thus, the need for cutting samples and damaging the bulk product is eliminated.

Application

The instrument is ideal for testing samples on line and bulk samples without damaging those in any way. The unit is robust and simple to use, and testing is fast and reproducible. Process control is one of the important application areas for this instrument.

Microflow Porometer

Operation

Gas pressure on one side of the sample is increased and the increase in pressure on the other side due to gas flow through the sample is monitored. The inlet gas pressure and the outlet gas pressure are measured. The permeability is calculated from these data. Use of wetted samples permits determination of pore size and pore distribution.

Application

The instrument can measure permeability in nearly impermeable materials. All other properties determined by capillary flow porometry can also be measured. The instrument is used for characterization of membranes and other filtration media.

Bubble Point Tester

Operation

The instrument uses a wetted sample and measures pressure that starts flow through the sample. This bubble point pressure yields bubble point pore diameter. Multi-head testers can simultaneously test many samples. Pass/fail or go/no-go material selection procedures can be adopted.

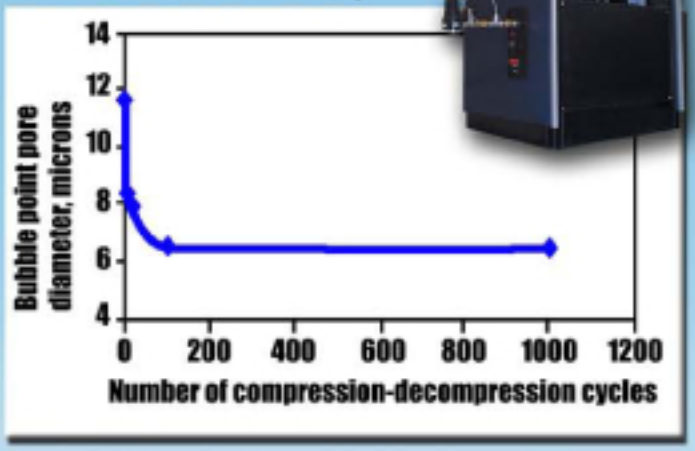
Application

Large volumes of samples are conveniently tested. The option of testing samples individually or simultaneously is available. Many options for segregating materials on the basis of the test results can be incorporated. This is widely used in industries for characterization of a variety of materials.

Cyclic Compression Porometer

Operation

The Cyclic Compression Porometer subjects the sample to cyclic compressive stress in the specified stress limit for the desired number of cycles and measures the pore characteristics like a capillary flow porometer. The instrument is fully automated and can perform many tests on the same sample after executing the number of stress cycles specified for each test.



Application

Many products like felts used for dewatering paper, filtration media and battery and fuel cell components are subjected to cyclic stresses. Such products need to be characterized under simulated true service conditions to give realistic data.

Integrity Tester

Operation

Gas pressure on the sample wetted with the wetting liquid is increased and the amount of gas flowing through the sample is measured. Gas often diffuses through the sample even before the largest pore in the sample is emptied.

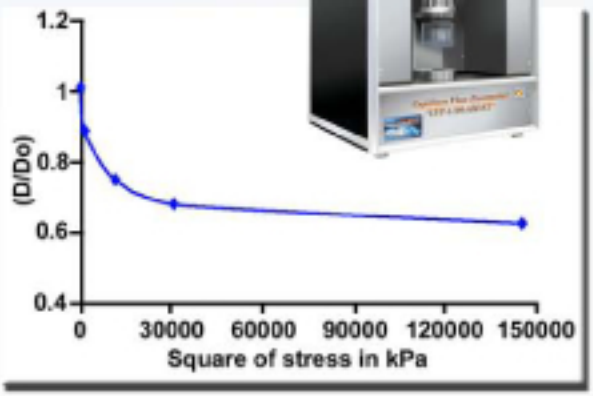
Application

Integrity of many materials including filter media, membranes, paper and battery separators is tested in this instrument. Bubble point can also be determined in the same instrument. The addition of optional features to the instrument allows testing of samples under tension, compression and elevated temperatures.

Compression Porometer

Operation

The Compression Porometer is a capillary flow porometer that is capable of maintaining the sample under controlled compressive stress while the test is being performed. Compressive stress up to 1000 psi may be applied. The data shows the variation of bubble point pore diameter (D) in relation to that at zero stress (Do).



Application

The pore characteristics of products experiencing considerable stress during service could be considerably different from those evaluated in the laboratory. This instrument provides a unique opportunity for evaluating the component under true service conditions. The instrument is invaluable for products such as filtration media and battery components.

Versatility

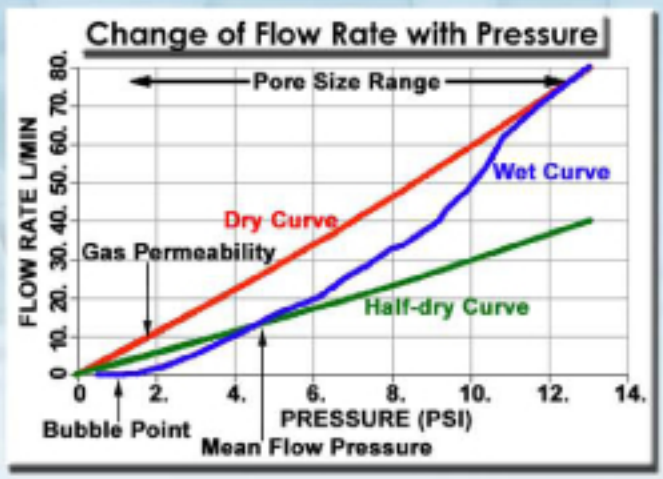
- Instruments may be custom built to suit the requirements of customers.
- One instrument can have the ability to perform many tests.
- On-line test can be performed.
- Many test options may be added to a basic unit.
- Tests can be performed with any wetting liquid.
- Tests can be performed under simulated service conditions.

Measurable Characteristics

- The largest constricted pore diameter (Bubble pointpore diameter)
- The mean flow pore diameter
- Pore size distribution
- Pore volume
- Pore volume distribution
- Envelope (external) surface area
- Average fiber diameter/Particle Size
- Microflow permeability
- Liquid permeability
- Effect of compressive stress
- Effect of cyclic compression
- Effect of temperature
- Effect of chemical environment
- Effect of orientation (x y & z directions)
- Hydro-head
- Integrity
- Gas permeability(Darcy, Gurley, Frazier & Rayle)

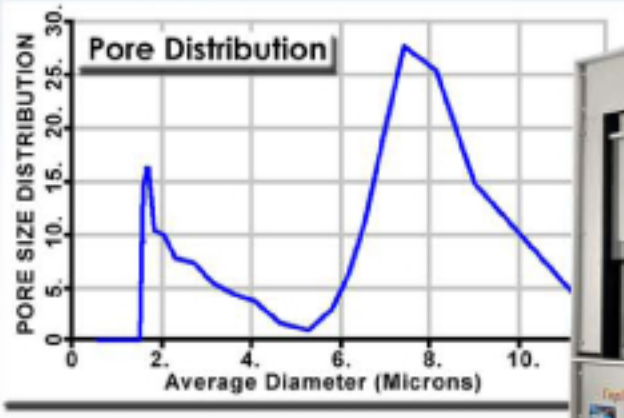
Instruments

Capillary Flow Porometer



Operation

The pores of the sample are filled with a wetting liquid. Gas pressure is increased on one side of the sample. At a certain pressure the largest pore is emptied and gas flow starts. On further increase of pressure, smaller pores are emptied and gas flow increases. Gas pressure and flow rate through wet and dry samples are measured. Pore diameters are computed from differential pressures and pore distribution is given by the distribution function, f = [-d(fw/fd)/dD] where fw & fd are flow rates through wet and dry samples respectively. The flow rate through dry sample gives gas permeability and envelope surface area.



Application

Constricted pore diameters in the 500 - 0.013 micron range, the largest pore diameter, mean flow pore diameter, and gas permeability (Darcy, Frazier, Gurley & Rayle) of filter media, nonwovens, battery parts, fuel cell parts, ceramics, paper, membranes, textiles, powders, chemicals, pharmaceuticals and biotech products are measurable. The instrument can have options to measure external surface area, average fiber diameter, average particle size, hydro-head and many other properties.

Multi-Chamber and Multi-Mode Porometer

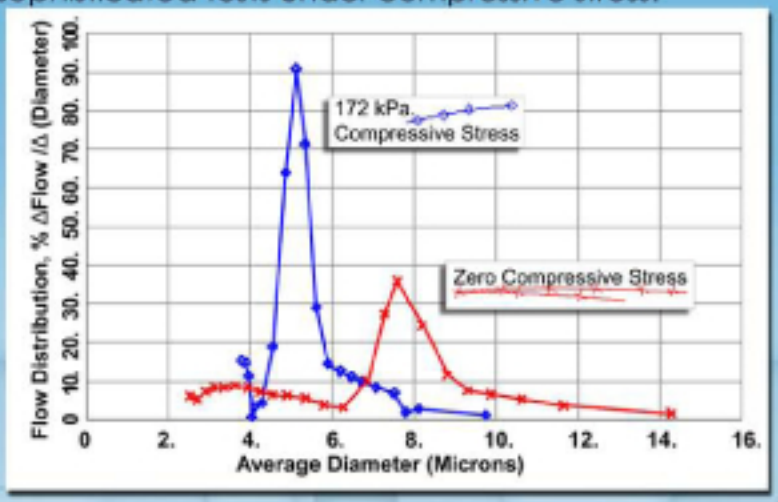
Operation

Multi-Chamber Porometers have multiple sample chambers. The Multi-Mode Porometers have the ability to characterize several different properties in different modes of testing. The Perm Porometer is a capillary flow porometer and liquid Permeameter that uses a penetrometer to measure liquid flow rate. The five chamber Multi-Mode Porometer combines the features of the QC Porometer and Compression Porometer. Four chambers test samples in the QC mode, while the fifth chamber performs sophisticated tests under compressive stress.

The Multi-chamber & Multi-mode Porometer

Application

Multi-Chamber and Multi-Mode Porometers are used in the industry to test large volumes of samples. The instruments are usually custom made.

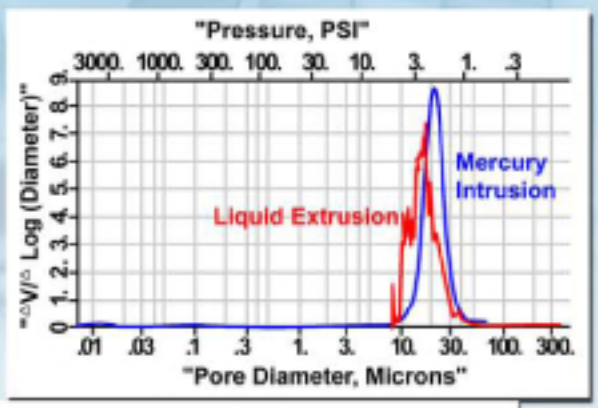


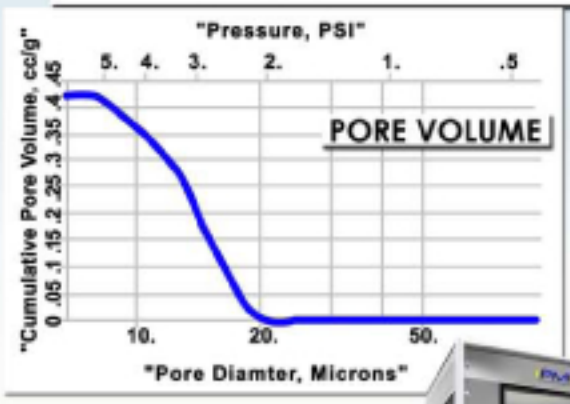
The Perm Porometer

Liquid Extrusion Porosimeter

Operation

The pores of the sample and a membrane are filled with a wetting liquid and the sample is placed on the membrane. The membrane is such that its largest pore is smaller than the smallest pore of interest in the sample. Gas pressure of a non-reacting gas is increased on the sample to extrude the liquid from pores. The volume of displaced liquid passing through the membrane is measured, while the liquid containing membrane prevents the gas from passing through. The gas pressure gives the pore diameter. The volume of displaced liquid gives the pore volume. Measurement of liquid flow rate without the membrane under the sample yields liquid permeability.





Application

Extrusion Porosimeter is the only instrument that has the capability to measure liquid flow rate, pore volume and pore volume distribution. The instrument does not use any toxic and harmful material like mercury. The pressure required is much less than that required by mercury intrusion porosimetry. Pore volume and liquid flow rates through powder and solid porous materials having pores in the range of 800 to 0.05 microns are measurable by this technique.

Specifications

Pressure Capability	up to 500 psi
Pressure Accuracy	0.15% of reading
Flow Capability	up to 200 LPM (Standard)
	up to 10,000 LPM (Optional)
Flow Accuracy	1:60,000 F.S
Resolution	1% of F.S.
Pore Diameter (min.)	0.013 µm
Pore Diameter (max.)	1000 µm

Wetting Liquid	Water	Mineral Oil	Alcohol	Silwick	Porewick	Galwick

Models

Many models are available. Instruments can be custom made to incorporate all the required features. Detailed information is available on request.

Sale **Lease** **Rent**

Other Products From PMI

Mercury Intrusion Porosimeters

Mercury intrusion porosimeters are fully automated instruments capable of measuring pore volume, pore volume distribution, surface area and porosity by mercury intrusion at pressures up to 60,000 psi.

Non-mercury Intrusion Porosimeters

These instruments measure pore volume and pore volume distribution using a non-wetting liquid other than mercury as the intrusion liquid. The use of the toxic mercury is avoided. The pressures needed are small so that concerns related to safety and the effect of pressure on pore structure are avoided. Small pore sizes are measurable. Use of actual application liquid permits service conditions to be simulated.

Gas Adsorption (BET) Sorptometers

The Sorptometers have the ability to measure single point and multi-point surface area, pore volume, micropores, chemisorption, and adsorption-desorption isotherms. Fully automated instruments with a variety of combination of options are available. Adsorption of a wide variety of gases and vapors is measurable.

Pycnometers

Fully automated gas pycnometers measure absolute density. Mercury intrusion pycnometers are used to measure bulk density of materials.

Contract Testing Service

The analytical services division of PMI has extensive experience in providing reliable, prompt and comprehensive contract testing service.

PMI
Porous Materials, Inc.

Permeameters

Gas & Liquid

PMI
Porous Materials, Inc.

Porous Materials, Inc.
20 dutch Mill Road, Ithaca, New York 14580 USA
Toll Free US & Canada: 1-800-TALK-PMI Phone: (607) 257-5544
Fax:(607) 257-5639 Email: info@pmiapp.com

Applications

Permeameters measure:

- Liquid Permeability
- Gas Permeability
- Microflow Permeability
- Diffusion Permeability
- Water Vapor Transmission rate

Permeameters are used in many industries such as chemical, biotech, pharmaceutical, food, beverage, fuel cells, batteries, and pollution control. Materials tested in permeameters include membranes, ceramics, filter media, sintered metal filters, hydrogels, paper, textiles, battery seperators, powder beds, electrodes, foams, sponges, and pen tips.

Principle

Permeameters measure fluid flow rates. The measured flow rates are expressed in liters per minute (LPM) or any other desired unit. Flow rate is often used to compute permeability defined by Darcy's Law:

$(k/\mu) = F / [A(\Delta p / l)]$

The flow at average pressure (F) per unit area (A) of the sample per unit pressure gradient ($\Delta p / l$) across the sample is defined as the ratio of permeability (k) of the samples and viscosity (μ) of the fluid. The cgs unit of permeability is cm^2. Permeability is often given in terms of Darcy, Fraizer, Gurley, or Rayle.

Liquid Permeameter

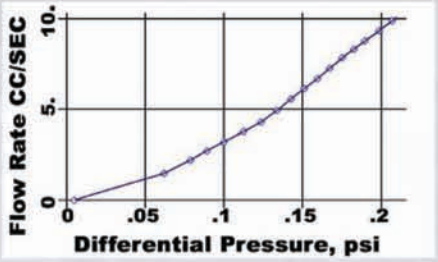
Instrument

Liquid from a penetrometer tube is forced through the sample. The Differential pressure on the liquid across the sample and the flow rate of the liquid are measured. Pressure is measured by a pressure transducer and liquid flow is measured by the penetrometer.

The fully automated instrument executes tests, acquires data, stores data, and displays data in the desired unit. Windows based operation of the instrument is simple.

Operational Features

- Permeability of a variety of chemicals like phosphoric acid, oil, salt solutions, fat, and body fluids.
- Multiple Sample Chambers for high volume testing
- Measurement at high pressures
- Measurement at elevated temperatures up to 200° C
- Permeability through sample under compression
- Sample chamber that does not require samples to be cut out from the bulk sample



Permeability of KOH solution

Microflow Liquid Permeameters

Instrument

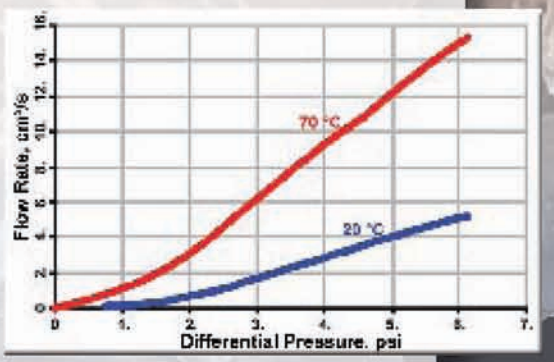
A Microflow liquid permeameter is a liquid permeameter that uses a programmable microbalance to accurately measure a small amount of liquid that may permeate through the sample.

Gas Permeameters

Instrument

Gas under pressure is forced through the sample. The differential pressure and the flow rate of the gas are measured with pressure and flow transducers.

The fully automated instrument executes test, acquires data, stores data, and displays data in the desired unit. Windows based operation of the instrument is simple.



Optional Features

- Permeability of a wide variety of gases
- Multiple sample chamber for high volume testing
- Measurement at high pressures
- Measurement at elevated temperatures
- Sample chamber that does not require samples to be cut out
- Permeability through sample under compression

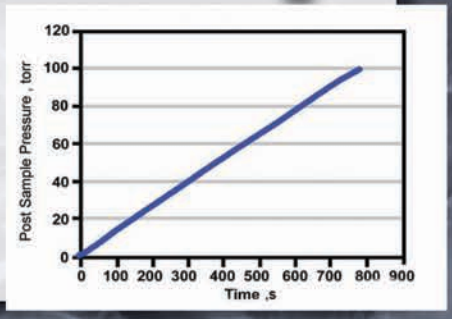
Microflow Gas Permeameters

Instrument

Gas permeameters cannot measure gas flow rate accurately when the flow rate through the sample is low. Such samples can be examined by microflow permeameters. In these instruments gas is brought to the inlet side of the sample at a known pressure and the increase in pressure on the outlet side is measured. The gas flow rate F at STP is computed from the following relation:

$F = (T_s V/Tps) (dp/dt)$

Where V is volume of the outlet chamber, ps is the standard pressure, (dp/dt) is the rate of pressure increase in the outlet chamber, the test temperature is T, the standard temperature is T_s . The instrument is fully automated. It can have many optional features.



Optional Features

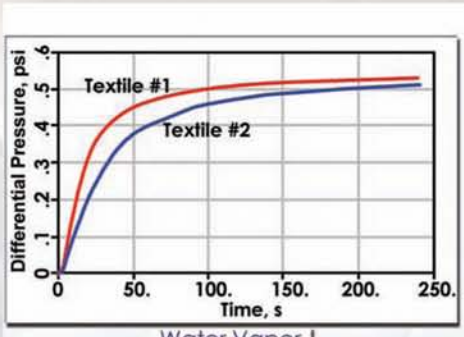
- Microflow permeability can be part of capillary flow porometry
- Measurement at elevated temperatures
- Measurement while sample is under compressive stress
- A wide variety of test gases can be used

Diffusion Permeameter

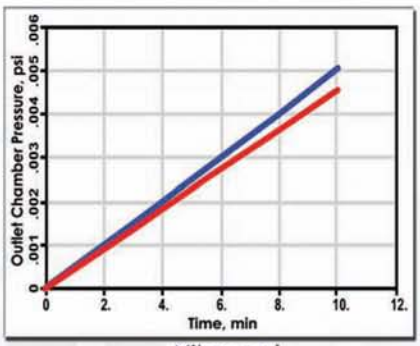
Instrument

When the gas flow rate through the sample is so low that a microflow permeameter cannot determine the permeability accurately, the diffusion permeameter can be used to measure gas permeability. The sample chamber of the instrument is evacuated first. Gas is maintained at a constant pressure in the inlet side and the increase in pressure in the outlet side is measured. Flow rates are computed as in microflow permeametry.

The instrument is fully automated. Because of evacuation, the instrument is capable of yielding very accurate results and permeability of a variety of gases is measurable. Flow rates as low as 10^{-4} cm^3/s are measurable.



Water Vapor

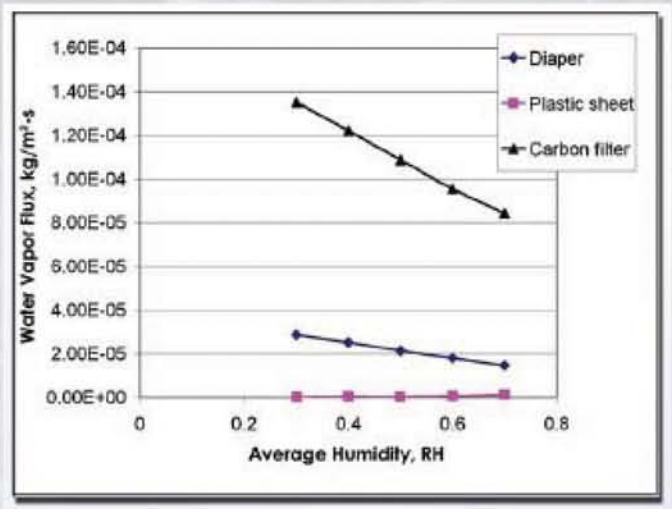


Nitrogen

Water Vapor Transmission Analyzer

Instrument

This instrument uses the dynamic moisture cell (ASTM F2298-03) for measurement of water vapor transmission rate. Transmission across a sample due to imposed humidity gradient, pressure gradient, or both gradients can be measured. The instrument is capable of measuring transmission rate and flow resistance as functions of humidity, pressure, and temperature.



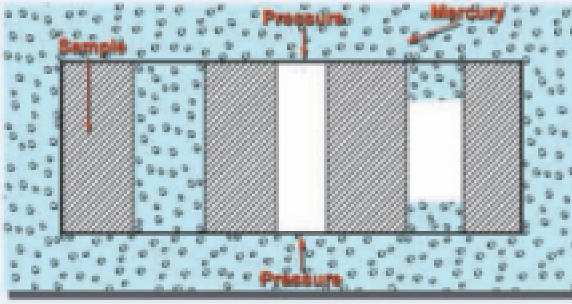
Porosimeters

"Not Just Products ... Solutions"!

Porous Materials, Inc.
20 Dutch Mill Road, Ithaca, NY 14850
Phone: (607) 257-5544 Fax: (607)257-5639
Toll Free (Canada & USA): 1-800-TALK-PMI
Email: info@pmiapp.com Website: www.pmiapp.com

Principle

A nonwetting liquid like mercury does not spontaneously fill pores of a sample because the sample/nonwetting liquid surface free energy is greater than the sample/gas surface free energy. However, application of pressure can force a nonwetting liquid into the pores of a sample. The differential pressure required to force the nonwetting liquid into a pore is given by:



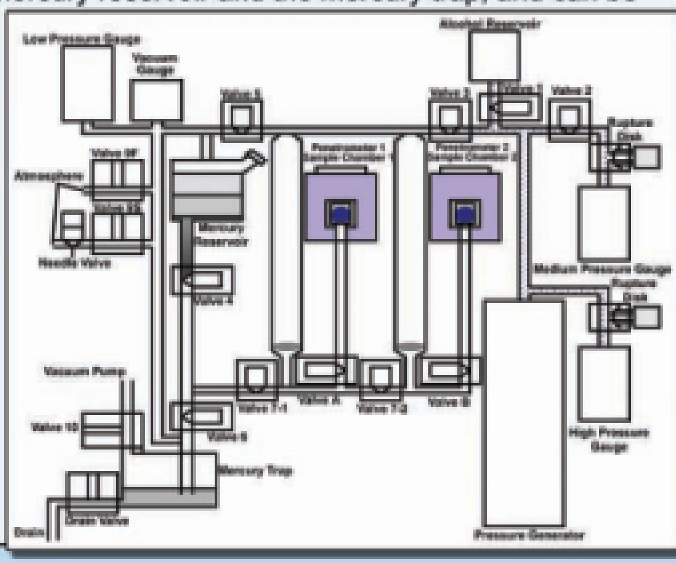
$$P = -4\,\gamma \cos\theta / D$$

P = differential pressure
γ = surface tension of nonwetting liquid
θ = contact angle of the nonwetting liquid with the sample
D = pore diameter

The pore diameter at any location in a pore is defined such that (dS/dV) = (4/D), where (dS/dV) is the ratio of small increase in surface area due to a small increase in volume of nonwetting liquid in the pore. In this technique the pressure and the volume of intruded nonwetting liquid are accurately measured. Combining these data with the surface tension and the contact angle of the liquid, pore diameter, pore volume, pore volume distribution and pore surface area are computed.

Operating Procedure

The PMI Porosimeter consists of a low pressure section, a high pressure section, and penetrometers connected to the sample chambers. The low pressure section includes the mercury reservoir and the mercury trap, and can be opened to the atmosphere or evacuated. The high pressure section includes a pressure generator that uses isopropyl alcohol as the hydraulic fluid to pressurize mercury. The sample is placed inside a stainless steel cell that has a hole on one of its sides for evacuation and entry of mercury into the cell. The cell is closed with a lid and placed in the sample chamber. Intrusion volume of mercury is measured by noting changes in mercury level in the penetrometer with a magnetic sensor. Exposure to mercury is negligible due to the unique design of the instrument. The unique design also permits use of nonmercury nonwetting liquids for intrusion.



Another instrument design uses water as the nonwetting liquid and uses absolutely no mercury. The instrument is known as the Aquapore.

Unique Features

Hardware

- Mercury and Nonmercury (other nonwetting liquid) Porosimetry can be performed in the Porosimeter.
- A unique system design allows use of the sample chamber to be connected to or isolated from the low pressure and high pressure sections of the instrument. This allows fully automated testing with no requirement to transfer samples from a low pressure station to a high pressure station in the middle of the test.
- Presence of three pressure transducers (low, medium, high) ensures maximum resolution and accuracy.
- Equipment design allows extrusion to be performed even at subatmospheric pressures.
- Operator involvement is minimum.
- Automatic clean-out routines after test limit the amount of mercury exposure.
- Samples can be tested at elevated or sub-ambient temperatures.
- Sample chamber and penetrometer are made out of stainless steel.
- Novel design enables sample chamber and penetrometer tube to be changed individually and independently.
- Simultaneous testing of multiple samples in a multiple sample chamber instrument is possible.
- In-situ pretreatment of samples is preformed to avoid contamination.

Safety

- A mercury level sensor warns the user to empty the mercury trap (where used mercury is collected).
- Doors of sample chamber area and the mercury drain reservoir area minimize operator exposure to mercury.
- The instrument cabinet has its own internal ventilation system. Mercury vapors within the cabinet are captured by an activated carbon filter.
- Burst pressure disks and relief valves prevent over pressurization of the system.
- The internal computer monitors the position of all of the valves and does not allow valve combinations that could impede the operation of the instrument. (The low and high pressure sections can not both be connected to the sample chamber, the mercury fill and drain valves can not be open at the same time, etc....)
- The hydraulic pressure generator system (in medium and high pressure instruments) has built-in limit switches that prevent mechanical damage that could be caused by over travel of the piston.
- Both the user software and the internal computer system monitor the pressure and do not allow the pressure generator to operate beyond the maximum pressure specifications of the instrument. In case of an over pressure situation, the internal computer will automatically retract the piston to relieve the pressure in a controlled and safe manner.

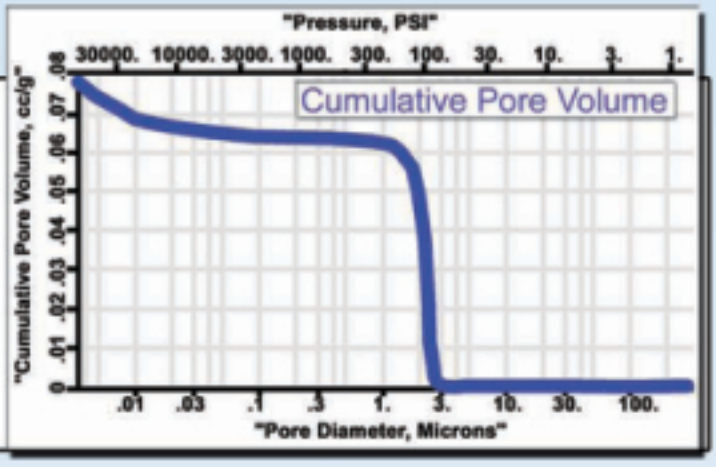
Software

- Windows 98/NT/2000/XP compatible software enables convenient use of the instrument.
- User defined contact angle and surface tension can be entered.
- Data can be collected either at user-specified pressure values, at user-specified intrusion volume values, or at evenly spaced user-specified number of data points. Equilibrium conditions are also user adjustable.
- Multiple users and/or sample types can be given their own software "group". The software settings are remembered independently for each "group".
- Outgassing and pretreatment performed either automatically or manually. The user can skip the normal automatic outgassing procedure.
- Testing can be paused at any time. While a test is paused, the software allows full manual operation of the instrument while retaining the ability to continue the test when manual control is completed.
- Separate software for report generation enables the user to plot up to seven test results on the same graph. Report generation does not have to be performed on the same computer that controls the instrument.
- The report software allows data to be converted directly into an Excel spreadsheet or exported to standard text or tab-delimited database or other programs.
- The software features curve fitting and interpolation routines that help in better analysis of the data.
- Desired pressure units can be selected for the reports independent of the units in test.
- Report software enables user to view reports, modify scales in graphs and print reports.

Applications

Mercury Porosimetry

PMI Porosimeters are capable of measuring properties such as pore volume, surface area, pore volume distribution, percent porosity and density. Pore volume is determined by the total volume of mercury that intrudes into the pores. Measured pore volume is the volume of all pores accessible to the mercury. The percent porosity can also be obtained as a function of pressure (pore size).



Pore volume distribution is presented in terms of a distribution function, Fv.

$$Fv = -\frac{dV}{d \log D}$$

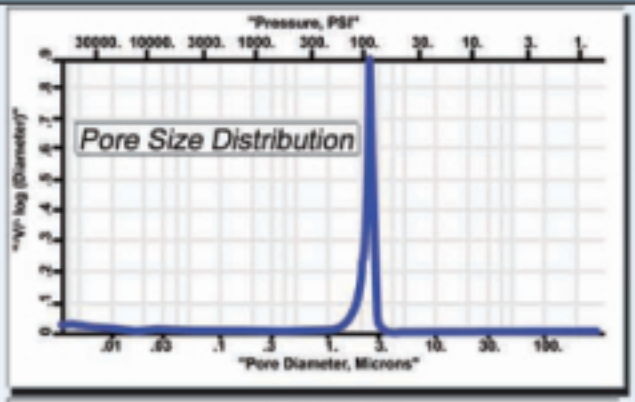
V = cumulative pore volume

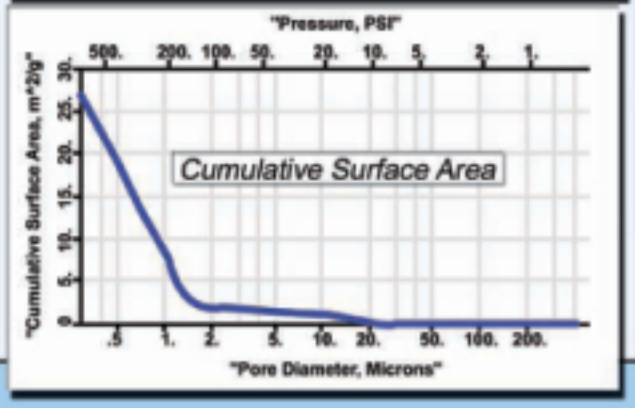
The distribution function is such that area under the function in any pore diameter range yields pore volume of pores in that range.

Measurement of surface area through porosimetry gives a quick estimate of the surface area. Surface area is computed from volume measured as a function of pressure through the use of the following relation derived from the equations given earlier.

$$S = \int PdV/(-\gamma \cos \theta)$$

S = surface area

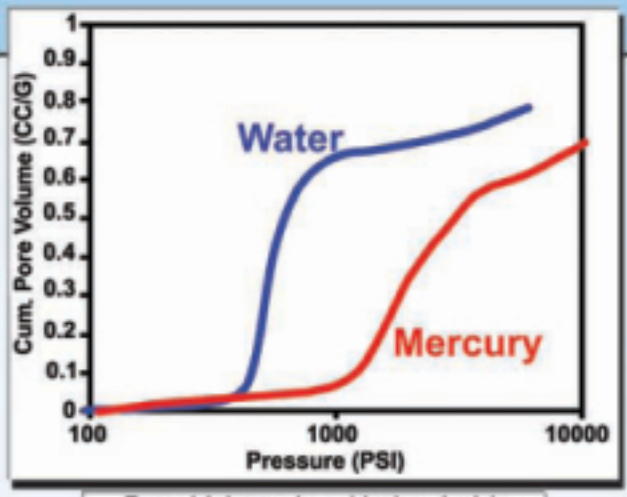




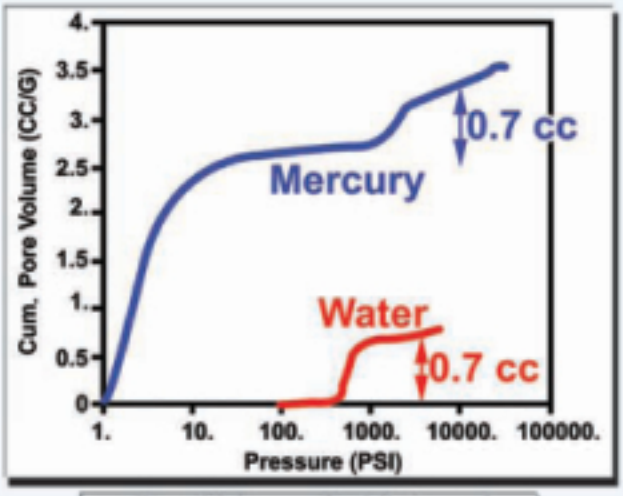
Nonmercury Porosimetry

PMI Porosimeters are unique in providing the dual capability to perform nonmercury porosimetry as well as conventional mercury porosimetry in the same instrument. The nonmercury porosimetry uses a fluid that is nonwetting to the material being tested. The use of nonmercury porosimetry over mercury porosimetry for certain materials may be advantageous due to the following reasons:

- Lower intrusion pressures: Most liquids have much lower surface tension values than mercury. The intrusion into pores can be accomplished at much lower pressures than those required with mercury. This greatly reduces the risk of the sample crushing at high pressures.



Pore Volume in a Hydrophobic Material as a Function of Pressure



Pore Volume of a Mixture of Hydrophobic & Hydrophilic Materials as a Function of Pressure

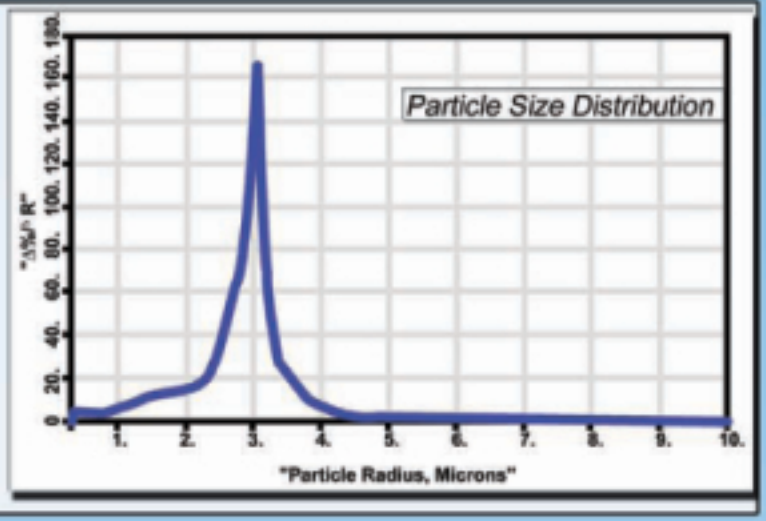
- Use of application liquid: Many applications require that the porous material be in constant contact with certain fluid. If this fluid is nonwetting to the material, it can be used for testing.
- Lower pore sizes: The pore sizes that can be measured in nonmercury porosimetry are much lower than those measured by mercury porosimetry.

For hydrophobic materials the use of water as the intruding fluid allows the measurement of very small pores that are beyond the scope of measurement by mercury intrusion method. When testing a sample that is a mixture of hydrophobic and hydrophilic materials, the combination of mercury intrusion and water porosimetry can give a comprehensive picture of the characteristics of both the hydrophobic and hydrophilic pores of the sample. The PMI **Aquapore** has the ability to perform water intrusion test without using any mercury.

Testing of Powders

Porosimetry of powders often results in bimodal distributions. Mercury enters the large interparticle voids at low pressures and intraparticle pores at high pressures.

The PMI Porosimeter uses a sample cell specifically designed for powders. This prevents the powder from contaminating other parts of the system. A slow evacuation routine prevents the powder from being pulled out of the sample cell. The particle size characterization is an important aspect in the manufacture of many powders including pharmaceutical powders.



Specifications

PMI's Automated Porosimeters are capable of measuring pore volume, surface area, pore size distribution, particle size distribution and percent porosity of porous materials based on intrusion and extrusion (below atmospheric pressure) of mercury or another nonwetting fluid.

Pore diameter range: 0.0035 – 400 microns (mercury) Smaller pores with other liquids

Sample volume: Up to 10 cc for sample cells (others available)

Pressure transducer: Three separate transducers, low, medium and high, for each instrument (some low pressure instruments have only two transducers)

Test pressure: 0 – 60,000 psi (instruments available with different pressure ranges)

Vacuum gauge: 0 – 1000 microns (0 – 0.1 cm) Hg

Penetrometer: Up to 1.5 cc (nominal) with unlimited refill capability

Resolution: 0.001 cc

Accuracy: 0.005 cc

Pressurizing fluid: Air/ Isopropyl Alcohol/Water

Power requirements: 110/220 Vac, 50/60 Hz / 15 Amps.

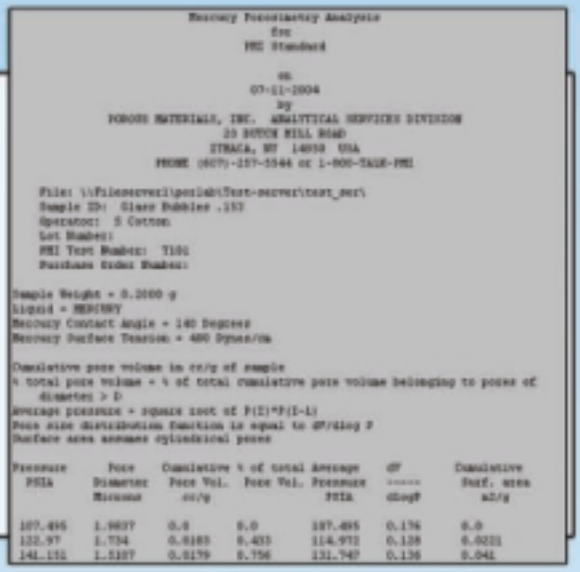
Size: 72" H, 30" W, 30"D, (approximately)

Weight: 400 lbs (approximately)

Software: For Windows 98//NT/XP

Data Report

- Pore volume versus diameter
- Delta volume histogram
- Pore distribution (histogram)
- Percent total pore volume
- Pore distribution (pressure and diameter)
- Particle size distribution (histogram)
- Percent porosity and density
- Cumulative surface area
- Porosimetry text data
- Summary sheet (reporting median pore sizes based on volume and surface area along with standard deviations)



Mercury Porosimetry Analysis
for:
PMI Standard

on:
07-11-2004
by:
POROUS MATERIALS, INC. ANALYTICAL SERVICES DIVISION
20 DUTCH MILL ROAD
ITHACA, NY 14850 USA
PHONE (607)-257-5544 or 1-800-TALK-PMI

File: \\Fileserver1\porlab\Test-server\test_ser\
Sample ID: Glass Bubbles .153
Operator: S Cotton
Lot Number:
PMI Test Number: T101
Purchase Order Number:

Sample Weight = 0.2000 g
Liquid = MERCURY
Mercury Contact Angle = 140 Degrees
Mercury Surface Tension = 480 Dynes/cm

Cumulative pore volume in cc/g of sample
% total pore volume = % of total cumulative pore volume belonging to pores of diameter > D
Average pressure = square root of P(I)*P(I-1)
Pore size distribution function is equal to dV/dlog P
Surface area assumes cylindrical pores

Pressure PSIA	Pore Diameter Microns	Cumulative Pore Vol. cc/g	% of total Pore Vol.	Average Pressure PSIA	dV ----- dlogP	Cumulative Surf. area m2/g
107.495	1.9837	0.0	0.0	107.495	0.176	0.0
122.97	1.734	0.0165	8.433	114.972	0.128	0.0221
141.131	1.5107	0.0179	8.756	131.747	0.136	0.041

PMI Analytical Services

PMI has over two decades of experience in analyzing porous materials using mercury/nonmercury intrusion porosimetry and other techniques. The analytical services division of PMI is well known for providing timely, accurate and reliable contract testing services. Contact PMI for details.

Models

Model: AMP–200–A–1

This is a fully automated unit capable of pressures up to 200 psi. It uses mercury as the penetration liquid and is able to detect pores >1 micron. It has one sample chamber. This is an economical unit good for regular testing of small volume of samples with large pores. Samples that are sensitive to pressure can be tested in this porosimeter. The unit is robust and does not require much maintenance.

Model: AMP–200–A–1–NM

This model incorporates special design features to permit use of nonmercury nonwetting liquid for intrusion. Otherwise it is identical with Model: AMP–200–A–1. Measurement of smaller pores (>0.04 microns with water) becomes possible. All advantages of using a nonmercury intrusion liquid are available to the user.

Model: AMP–200–A–2

This model has two sample chambers. In all other respects this model and the Model: AMP–200–A–1 are identical. Two samples can be tested simultaneously. This feature provides the capability of testing a sample of a material and a standard under identical conditions, so that the material may be compared with the standard and decisions on the selection or rejection of the material, quality control concerns and process control may be taken intelligently.

Model: AMP–200–A–2–NM

The porosimeter has all the features of Model: AMP–200–A–1 and the optional feature to test two samples in its two sample chambers with any nonmercury nonwetting liquid. Small pores are easily detectable (>0.04 microns with water). The porosimeter has all the advantages of a two sample chamber unit (see Model: AMP–200–A–2) and those associated with the use of nonmercury

Model: AMP–200–A–3

This model is identical with Model: AMP–200–A–1 except that it has three sample chambers permitting three samples to be tested simultaneously. This is an inexpensive model to test a large volume of samples with larger pores (>1 micron).

Model: AMP–200–A–3–NM

This Model has three sample chambers and is designed to work with any nonmercury nonwetting liquid. It has all the features of Model: AMP–200–A–1. This instrument has the advantages of using nonmercury nonwetting liquid, permits detection of smaller pores (<0.04 microns with water) and can test a large volume of samples.

Model: AMP–2K–A–1

This is a fully automated low pressure model with maximum pressure capability of 2000 psi. Mercury is used as the intrusion liquid and the detectable pore size is 0.1 micron. The unit has one sample chamber and is good for testing a small volume of samples with pores >0.1 micron. Because of low pressures, the effect of pressure on the structure of the sample is not expected to be appreciable. This is also a robust model that requires very little maintenance.

Model: AMP–2K–A–1–NM

This model is identical with the Model: AMP–2K–A–1 except that it has features that permit any nonmercury nonwetting liquid to be used. Pores less than 0.015 microns in size are measurable. It gives all the advantages associated with its ability to use any liquid.

Model: AMP–2K–A–2

This model has two sample chambers. All other features of the model are identical with those of Model: AMP–2K–A–1. The instrument is capable of testing two samples simultaneously and has all the advantages associated with the use of two sample chambers (see Model: AMP–200–A–2).

Model: AMP–2K–A–2–NM

This low pressure model has been designed to use nonmercury liquid for intrusion into samples in two sample chambers and has the features of Model: AMP–2K–A–1. The porosimeter has all the advantages associated with use of any nonmercury intrusion liquid and two sample chambers (See Model: AMP–200–A–2).

Model: AMP–2K–A–3

This low pressure porosimeter has three sample chambers. Other features of the porosimeter are identical with those of Model: AMP–2K–A–1. Three samples can be tested simultaneously. The porosimeter is very useful for testing large volume of samples with large pores (>0.1 micron) on a regular basis.

Model: AMP–2K–A–3–NM

This model is identical with Model: AMP–2K–A–1, except that it has three sample chambers and it is capable of using any nonmercury nonwetting liquid. The instrument has all the advantages of using any nonmercury intrusion liquid and can test a large volume of samples with pores less than 0.1 microns in size.

Model: AMP–10K–A–1

This is a completely automated medium pressure model capable of going up to 10,000 psi. It has one sample chamber. It uses mercury as the intrusion liquid and measures pores with diameters down to 0.02 microns. The effect of pressure on pore structure is normally not appreciable. The instrument is suitable for testing small volume of samples with not very small pore diameters.

Model: AMP–10K–A–1–NM

This model can use any nonmercury intrusion liquid and has all the features of Model: AMP–10K–A–1. The pore size detectable can be less than 0.02 microns. It has all the advantages of using any nonmercury liquid.

Model: AMP–10K–A–2

This model has two sample chambers. Other features of this model are the same as those of Model: AMP–10K–A–1. Simultaneous testing capability for two samples gives the instrument all the advantages of a two–sample chamber instrument (See Model: AMP–200–A–2).

Model: AMP–10K–A–2–NM

This unit has the ability to test with any nonmercury intrusion liquid and has two sample chambers. In all other respects it is identical with Model: AMP–10K–A–1. The pore size detectable can be less than 0.02 microns. The porosimeter has all the advantages of a machine that has two sample chambers (See Model: AMP–200–2) and has the ability to use any nonmercury nonwetting liquid.

Model: AMP–10K–A–3

This model contains three sample chambers and has all the features of Model: AMP–10K–A–1. Simultaneous testing of three samples is possible. The instrument is good for testing a large volume of samples containing not too small pores (> 0.02 microns).

Model: AMP–10K–A–3–NM

This medium pressure model is identical with Model: AMP–10K–A–1 and has additional features that permit use of any nonmercury intrusion liquid and simultaneous testing of three samples. Pores less than 0.02 microns may be detected. It has the advantages due to the use of any nomercury liquid. It is ideal for testing a large volume of samples with smaller pore sizes (0.02 microns).

Model: AMP–15K–A–1

This is another completely automated medium pressure unit in which the intrusion pressure can go up to 15,000 psi. Using mercury as the intrusion liquid, pores down to 0.015 microns can be measured. The instrument has one sample chamber. The effect of pressure on pore structure is usually small. The instrument is suitable for testing a small volume of samples.

Model: AMP–15K–A–1–NM

This instrument can use any nonmercury intrusion liquid. In all other respects it is identical with Model: AMP–15K–A–1. Measurable pore size can be less than 0.015 microns, depending on the fluid. The instrument has all the advantages associated with the use of a nonmercury liquid.

Model: AMP–15K–A–2

This is a two-sample chamber model with all the capabilities of Model: AMP–15K–A–1. Two samples can be tested simultaneously. This capability gives the instrument all the advantages of a two sample chamber instrument. (See Model: AMP–200–A–2).

Model: AMP–15K–A–2–NM

This two–sample chamber model is capable of using any nonmercury intrusion liquid. Other features of the instrument are identical with the Model: AMP–15K–A–1. The porosimeter has the advantages of a two sample chamber machine that is capable of using any nonmercury liquid.

Model: AMP–15K–A–3

This is a three–sample chamber model that has all the features of Model: AMP–15K–A–1. Three samples can be tested simultaneously. The instrument can handle a large volume of samples with pores greater than 0.015 microns.

Model: AMP–15K–A–3–NM

This porosimeter is identical with Model: AMP-15K-A-1 except that it has three sample chambers and it can use any nonmercury intrusion liquid. The instrument has all the advantages due to the use of nonmercury liquid. It is ideal for testing a large volume of samples with pores which can be less than 0.015 microns.

Model: AMP–30K–A–1

This is a sophisticated and fully automated high pressure model. It is capable of attaining pressures up to 30,000 psi. The smallest pore size measurable is about 0.007 microns with mercury as the intrusion liquid. This is a single sample chamber unit, which is cost effective when a small number of samples need to be tested or when testing is infrequent. The high pressure attainable by this instrument can cause crushing of porous particles of powders so that the total surface area including surface area of internal pores in the particles could be measured.

Model: AMP–30K–A–1–NM

This model is identical with Model: AMP–30K–A–1 in addition to having the ability to use any nonmercury intrusion liquid. Pore sizes less than 0.007 microns can be measured, depending on the fluid used. The instrument has all the advantages associated with the use of nonmercury intrusion liquid.

Model: AMP–30K–A–2

This high pressure model has two sample chambers and all of the capabilities of Model: AMP–30K–A–1. Simultaneous testing of two samples is possible. The instrument has all the advantages of a two-sample chamber instrument (See Model: AMP–200–A–2).

Model: AMP–30K–A–2–NM

The model has the ability to use any nonmercury intrusion liquid and two samples in its two sample chambers. In all other respects it is identical with Model: AMP–30K–A–1. It has all the advantages due to the use of nonmercury liquid and simultaneous testing of two samples (See Model: AMP–200–A–2).

Model: AMP–30K–A–3

This sophisticated model is capable of testing three samples simultaneously in its three sample chambers. All other features of this model are the same as those of Model: AMP–30K–A–1. The porosimeter is suitable for testing a large volume of samples with pores > 0.007 microns.

Model: AMP–30K–A–3–NM

This is a model whose features are the same as those of Model: AMP–30K–A–1, but it has three sample chambers and is capable of using any nonmercury intrusion liquid. It has all the advantages of using nonmercury intrusion liquid. The instrument is suitable for testing a large volume of samples and for detecting < 0.007 micron pores.

Model: AMP–60K–A–1

This unit is sophisticated and fully automated. It is capable of attaining 60,000 psi intrusion pressure. Using mercury as the intrusion liquid, the minimum pore size detectable is 0.0035 microns. It has one sample chamber. It is the most economical unit for testing a small volume of samples with small pores. The high pressure can crush most powders with internal pores. Therefore, it is possible to determine areas of external surfaces of powders and surfaces of pores in powders.

Model: AMP–60K–A–1–NM

This model has all the features of Model: AMP–60K–A–1 and the ability to use nonmercury intrusion liquid. The instrument has all the advantages associated with the use of nonmercury intrusion liquid.

Model: AMP–60K–A–2

This porosimeter has two sample chambers. Otherwise it is identical with Model: AMP–60K–A–1. Two samples can be simultaneously tested. This unit has all the advantages of two-sample chamber model (See Model: AMP–200–A–2).

Model: AMP–60K–A–2–NM

This model is identical with Model: AMP–60K–A–1 and has the ability to test samples using mercury or any other nonmercury nonwetting intrusion liquid. The instrument can measure small pores and has all the advantages due to the use of nonmercury nonwetting liquid and two sample chambers (See Model: AMP–200–A–2).

Model: AMP–60K–A–3

This porosimeter model has all the features of Model: AMP–60K–A–1 and three sample chambers so that three samples can be tested simultaneously at high pressures. This instrument is ideal for testing of large volume of samples with small pores.

Model: AMP–60K–A–3–NM

This is a high pressure three–sample chamber porosimeter that has been designed to use any nonmercury intrusion liquid. All other features of the model are identical with those of Model: AMP–60K–A–1. The porosimeter is suitable for testing a large volume of samples with small pores and has all the special advantages due to the use of nonmercury intrusion liquid.

Liquid Extrusion Porosimeter

Description

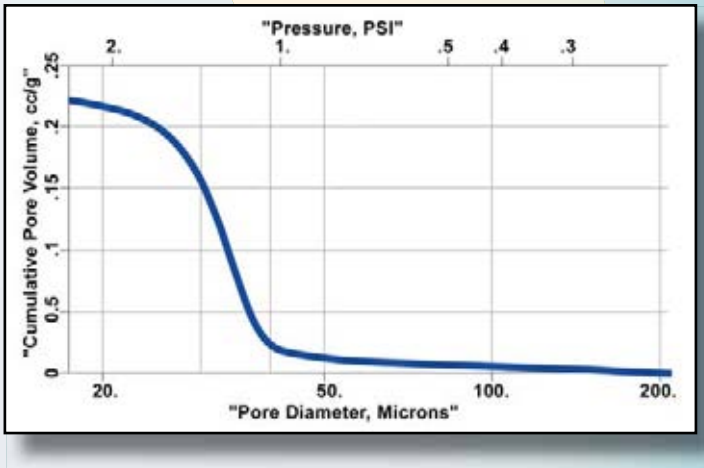
The PMI Liquid Extrusion Porosimeter is a unique instrument with the ability to measure through–pore volume, volume distribution and liquid permeability without using mercury. The instrument is employed for characterization of porous materials used in many industries such as biotech, pharmaceutical, filtration, food, and environment. It produces no harmful effects on personnel or environment.

Principle

The sample is placed on a membrane in the sample chamber. The membrane is such that its largest pore is smaller than the smallest pore to be tested. The pores of the sample and the membrane are filled with a weting liquid. The pressure of a nonreacting gas is increased on the sample to extrude the liquid from the pores. The differential pressure, p, required to displace liquid from a pore is related to its diameter, D, surface tension of the liquid, γ, and contact angle of the liquid, θ.

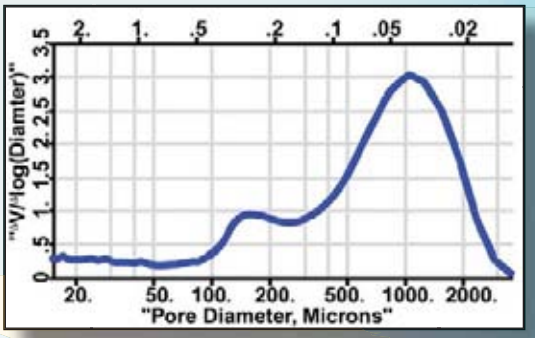
$$p = 4 \gamma \cos \theta / D$$

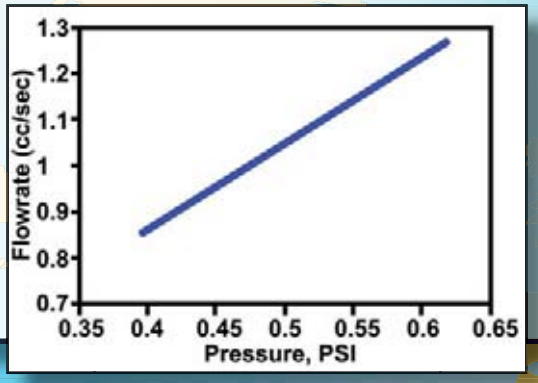
The displaced liquid passes through the liquid–filled pores of the membrane and its volume is measured, while the liquid-filled pores of the membrane prevent the gas from passing through because of insufficient pressure. The gas pressure gives the pore diameter. The volume of displaced liquid gives the pore volume. Measurement of liquid flow rate without the membrane under the sample yields liquid permeability of the sample.



Features

- One Instrument performs like two. Measures liquid permeability like a permeameter and pore volume like a Mercury Intrusion Porosimeter.
- No toxic material like mercury is used.
  No health hazard.
  No disposal-related cost.
- Fully automated. Simple to use.
  Very little operator involvement.
- Highly reproducible & accurate.
- A wide variety of samples can be investigated.
- Pressure required almost an order of magnitude less than that required for mercury intrusion.
- Can be used for pressure sensitive materials.
- Only instrument capable of measuring through-pore volume.
- Effects of application environment measurable. (stress, temperature, chemical environment).
- Capable of measuring very large pores (up to 1000 microns).





Specifications

Pressure Range
0 - 100 psi (Others Available)

Pore Size Range
1000 μm - 0.05 μm

Resolution
1 in 20,000

Intrusion Volume Range
0.01 cc

Sample Size
1.5” Diameter, 1” Thick
(Others Available)

Other Products

Average Fiber Diameter Analyzer
Bubble Point Tester
Capillary Flow Porometer
Capillary Condensation Flow Porometer
Complete Filter Cartridge Analyzer
Clamp-On Porometer
Compression Porometer
Custom Porometer
Cyclic Compression Porometer
Envelope Surface Area Analyzer
Filtration Media Analyzer
High Flow Porometer
Integrity Analyzer
In-Plane Porometer
Microflow Porometer
Nanopore Flow Porometer
QC Porometer
Diffusion Permeameter
Gas Permeameter
Liquid Permeameter
Vapor Permeameter
Water Vapor Transmission Analyzer
Liquid Extrusion Porosimeter
Mercury/Nonmercury Intrusion Porosimeter
Vacuapore
Water Intrusion Porosimeter (Aquapore)
BET Liquisorb
BET Sorptometer
Gas Pycnometer
Mercury Pycnometer

Also Available:
Testing Services
Consulting Services
Short Courses

Buy Rent Lease

Porous Materials, Inc.
20 Dutch Mill Rd, Ithaca, NY 14850 USA
Tel: (607)-257-5544 Toll Free in USA & Canada: 1-800-TALK-PMI
Fax: (607) 257-5639 Email: info@pmiapp.com WWW.PMIAPP.COM

07/08

Water Vapor Transmission Analyzer

Applications

The PMI Water Vapor Transmission Analyzer is capable of measuring water vapor transmission through porous media such as textiles, leathers, man made materials, membranes, nonwovens, and fabrics used in numerous high technology components and consumer products manufactured by a variety of industries. The instrument has the unique ability to measure vapor transmission rate over a wide range of humidity, temperature, and pressure under gradients of humidity, temperature, and pressure encountered in application environments.

Principle of Operation

Two independent gas streams are maintained on the two sides of a sample at the desired temperature. Humidity and gas flow rates are measured. The transmission rate through the sample is computed using mass balance.

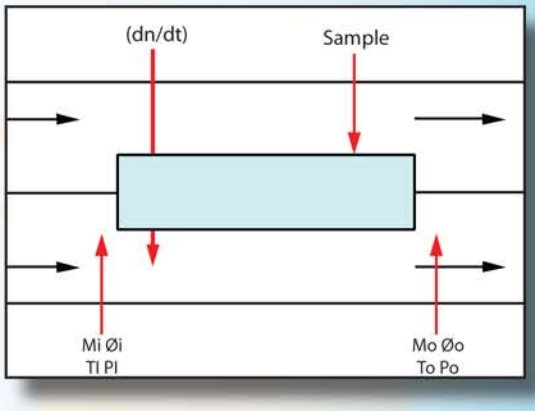
$$(dn/dt) + [(p_{e,i}\Phi_i / P_i)\, M_i] = [(p_{e,o}\Phi_o / P_o)\, M_o]$$

Where

n = moles Φ = humidity M = flow rate

t = time p_e = equilibrium vapor pressure

í = inlet o = outlet P = total pressure



Specifications

Humidity (φ) measurement
Range: 5 - 95%
Accuracy: ± 2%

Humidity (φ) control
Range: 0 - 100%
Accuracy: ± 1.5% (φ = 0.5)
± 5% (high & low φ)

Differential pressure transducers
Range: 4 torr (2 mm Hg)
Accuracy: 0.015%

Temperature
Range: RT - 100 °C
Accuracy: 0.4 °C (low φ) - 0.8 °C (high φ) at 100 °C
Control: ± 2%

Mass Flow Transducers
Range: 5 L/min
Accuracy: 1%

Mass Flow Controller
Range: 2000 cc/min
Accuracy: 1%

Instrument

The sample is enclosed in a sample chamber. A part of the gas flowing through each independent stream is allowed to go through bubblers while the other part bypasses the bubblers and mixes with the gas passing through the bubblers. For maintaining constant humidity in the inlet gas stream, the flow rate in each part of the gas stream is controlled. The gas pressure is controlled by the valve at the end of each gas flow line. The valves automatically control and maintain either zero differential pressure or a finite definite pressure difference. Absolute pressure remains close to the standard pressure. The inlet and outlet flow rates and humidity are measured. The water vapor transmission rate through the sample is computed using the following relation.

$$(dn/dt) = [(p_e/P)\Phi_o - (p_e/P)\Phi_i]M_o/[1-(p_e/P)\Phi_i]$$

Features

- Humidity on any side can be maintained between 5 and 95%
- Any desired pressure gradient can be maintained.
- Any desired test temperature can be achieved
- Simultaneous pressure and humidity gradients can be maintained
- Flat samples in a wide range of sizes can be accommodated
- Completely automated
  - « Test execution
  - « Data acquisition
  - « Data storage
  - « Data reduction

Other Products

Average Fiber Diameter Analyzer
Bubble Point Tester
Capillary Flow Porometer
Capillary Condensation Flow Porometer
Complete Filter Cartridge Analyzer
Clamp-On Porometer
Compression Porometer
Custom Porometer
Cyclic Compression Porometer
Envelope Surface Area Analyzer
Filtration Media Analyzer
High Flow Porometer
Integrity Analyzer
In-Plane Porometer
Microflow Porometer
Nanopore Flow Porometer
QC Porometer
Diffusion Permeameter
Gas Permeameter
Liquid Permeameter
Vapor Permeameter
Water Vapor Transmission Analyzer
Liquid Extrusion Porosimeter
Mercury/Nonmercury Intrusion Porosimeter
Vacuapore
Water Intrusion Porosimeter (Aquapore)
BET Liquisorb
BET Sorptometer
Gas Pycnometer
Mercury Pycnometer

Also Available:
Testing Services
Consulting Services
Short Courses

Buy Rent Lease

Porous Materials, Inc.
20 Dutch Mill Rd, Ithaca, NY 14850 USA
Tel: (607)-257-5544 Toll Free in USA & Canada: 1-800-TALK-PMI
Fax: (607) 257-5639 Email: info@pmiapp.com WWW.PMIAPP.COM

02/09

Aquapore

Principle

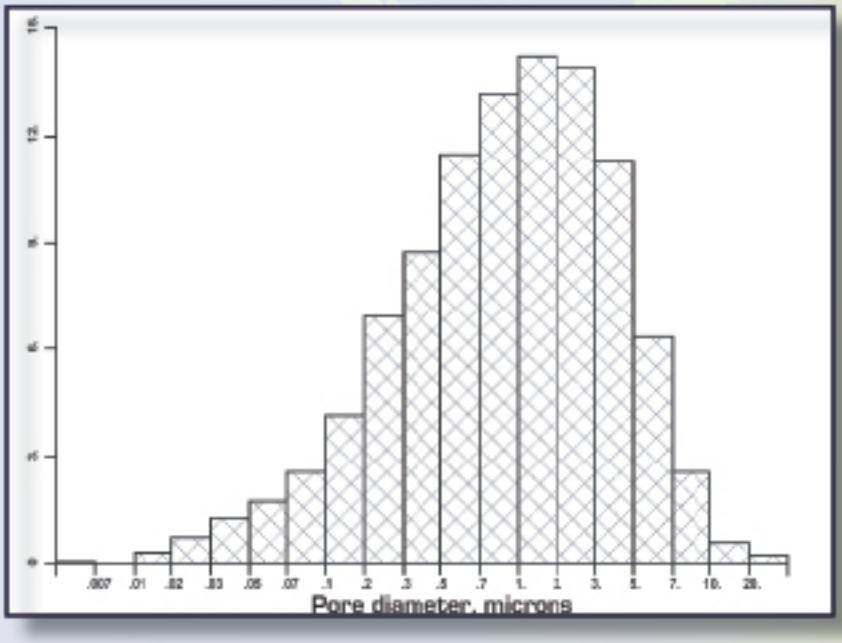
Aquapore is a liquid intrusion porosimeter used for characterization of hydrophobic pores. Water does not wet hydrophobic pores. Intrusion of water into pores occurs on application of pressure. Measured intrusion volume of water yields pore volume and measured intrusion pressure yields pore diameter.

$p = - 4 g \cos q / D$

p= differential pressure on water g= surface tension of water
q= contact angle of water D= pore diameter

Capability

Measures:
- Pore volume
- Pore diameter
- Pore volume distribution
- Hydrophobic through pores
- Hydrophobic blind pore



Specifications

Pore Size Range
3.5 - 2000 microns

Surface Area Range
0.01 m2/g and higher

Sample Size
Up to 1 cm in diameter

Pressure Range
Vacuum - 1 Atm (14.5 PSI)

Pressurizing Gas
Any non-corrosive gas including nitrogen, krypton, argon, ammonia

Pressure Transducer Range
10 Torr - 1000 Torr

Accuracy
0.15%

Resolution
1 in 10,000

Power Requirements
110/120 VAC, 50/60 Hz (Other available)

Dimensions
70" H x 29" W x 13" D

Weight
250 lbs

Features

Handles micropores and mesopores
Evacuation and testing under sub-freezing to elevated temperature ranges
Unlimited number of user defined data points
Windows-based software handles all control, measurement, data collection, and report generation; manual control also possible
Compatible with Windows 95 or higher
Real-time visual test display depicts testing status and results throughout operation
Non-destructive testing
Length of test varies from 2.5 hrs to 2 days depending on sample type and tests performed
Wide range of acceptable sample types and sizes
Multiple sample chambers available
Minimal maintenance required
Chemisorption over a wide range of pressures and temperatures

Other Products

Average Fiber Diameter Analyzer
Bubble Point Tester
Capillary Flow Porometer
Complete Filter Cartridge Analyzer
Clamp-On Porometer
Compression Porometer
Cyclic Compression Porometer
Envelope Surface Area Analyzer
Filtration Media Analyzer
High Flow Porometer
Integrity Analyzer
Integrity Analyzer
In-Plane Porometer
Microflow Porometer
Multi-Chamber and Multi-Mode Porometer
QC Porometer
Diffusion Permeameter
Gas Permeameter
Liquid Permeameter
Vapor Permeameter
Water Vapor Transmission Analyzer
Liquid Extrusion Porosimeter
Mercury/Nonmercury Intrusion Porosimeter
Mercury Pycnometer
BET Liquisorb
BET Sorptometer
Gas Pycnometer

Also Available:
Testing Services
Consulting Services
Short Courses

Buy Rent Lease

Porous Materials, Inc.
20 Dutch Mill Rd, Ithaca, NY 14850 USA
Tel: (607)-257-5544 Toll Free in USA & Canada: 1-800-TALK-PMI
Fax: (607) 257-5639 Email: info@pmiapp.com WWW.PMIAPP.COM

December 2007

BET SORPTOMETER

Description

PMI's BET Sorptometer accurately measures total surface area (via single and multi-point methods), adsorption and desorption isotherms, mean pore size, pore size distribution, pore volume, and pore structure. PMI's BET Sorptometer can assess a wide variety of samples, including powders and bulk solids, and can analyze both micropores and mesopores. Chemisorption of a wide variety of chemicals is measurable.

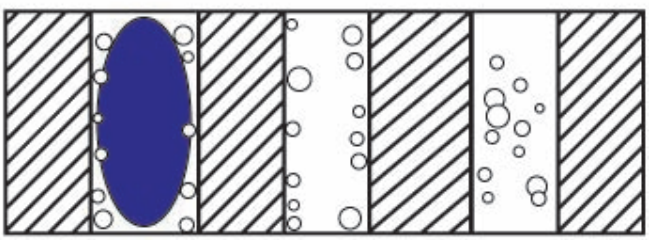
Applications

PMI's BET Sorptometer has a multitude of applications in industries worldwide. Industries that utilize the versatile BET Sorptometer include the automotive, battery, and pharmaceutical. Specific applications include the characterization of catalysts in the chemical industry, pulp characterization in the paper industry, and testing of the powder pre-cursors to predict adhesion and final porosity in the powder metallurgy industry.

Applicable industries are:

- Automotive
- Chemical
- Ceramic
- Paper
- Battery Separator
- Fuel Cells
- Filtration
- Pharmaceuticals
- Powder Metallurgy

Principle of the Gas Adsorption Technique



Principle

When a clean surface is exposed to a gas, an adsorbed film forms on the surface. Adsorbed films also form on the surface of pores within a material and vapor can condense in the pores. At a constant temperature, the amount of adsorbed/condensed gas on a surface depends on the pressure of the gas. Measurement of the amount of adsorption/condensation as a function of pressure can give information on the pore structure. The PMI Sorptometers use gas adsorption/condensation to analyze pore characteristics.

Physical Adsorption

Weak van der Waal's type interaction of molecules with a pore surface leads to physical adsorption. The Brunauer, Emmett and Teller (BET) theory of physical adsorption is normally used for analysis of adsorption data to compute surface area.

$$\frac{P}{W(P_o - P)} = \frac{1}{CW_m} + \frac{C-1}{CW_m} X \frac{P}{P_o}$$

where:
W = amount of adsorbed gas
Wm = amount of gas adsorbed in a monolayer
P = gas pressure
Po = equilibrium (saturation) vapor pressure at the test temperature
C = dimensionless constant that depends on the temperature and the gas/solid system

When vapor pressure, P, is low compared with Po ($0.05 < P/Po < 0.3$), the plot of [P/W (Po – P)] versus [P/Po] is linear and the plot yields the magnitudes of C and Wm. The surface area S per unit mass, m, of the sample is computed using the cross–sectional area of the adsorbed gas molecule:

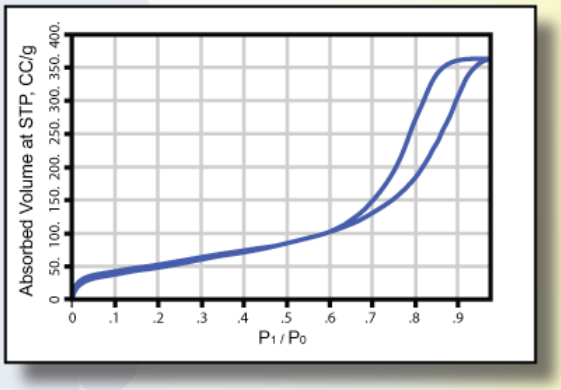
$$S = \frac{W_m N_o \alpha}{m}$$

where:
No = Avogadro's number
a = cross–sectional area of the adsorbed gas molecule
Wm = amount of gas absorbed in moles.

Vapor Condensation

As the relative vapor pressure (P/Po) increases, vapor eventually condenses in the pores utilizing the surface free energy available due to replacement of the solid/vapor interface by solid/liquid interface. The amount of vapor condensed in pores gives the pore volume, and the Kelvin equation gives the pore diameter.

$$\ln\left(\frac{P}{P_o}\right) = - \frac{4 \gamma V \cos \theta}{D R T}$$



Adsorption and Desorption Isotherms at Liquid N2 temperature

where:
g = surface tension of condensed liquid
V = molar volume of condensed liquid
q = contact angle
D = pore diameter
R = gas constant
T = absolute test temperature

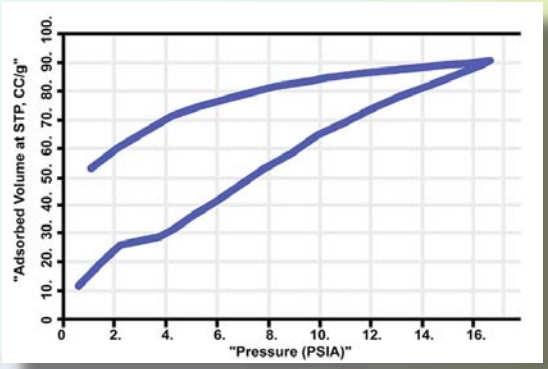
Adsorbed layers of molecules form on the pore walls before condensation fills the pores. Therefore, the actual pore diameters are computed by adding two times the thickness of the adsorbed gas layer to D.

A complete adsorption isotherm is determined by measuring the amount of vapor adsorbed as a function of increasing pressure. A desorption isotherm is determined by measuring the amount of adsorption as a function of deceasing pressure. Based on this technique, characteristics of materials related to adsorption, desorption, surface area and pore volume can be determined.

Chemisorption Chemical reaction between a gas and a surface accompanied by a high heat of adsorption results in chemisorption. Chemisorption data can be analyzed using various theories. Langmuir equation yields:

$$\frac{W}{W_m} = \frac{KP}{1 + KP}$$

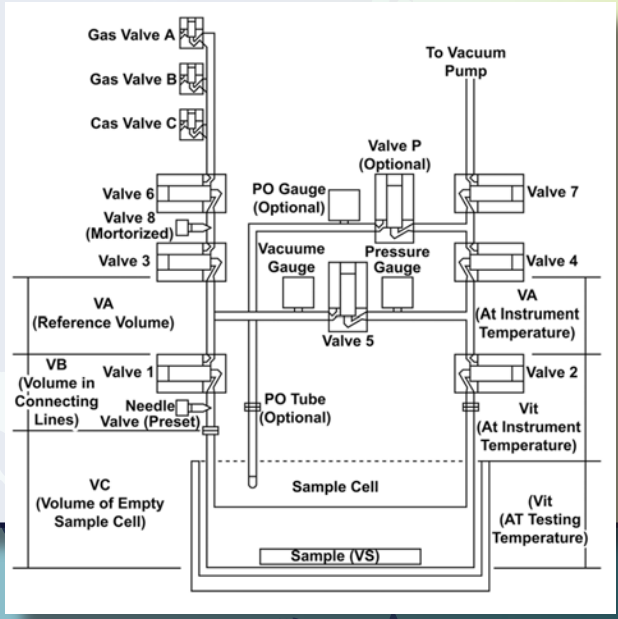
where:
W = amount of adsorbed gas
Wm = amount of gas adsorbed in a monolayer
K = equilibrium constant
P = gas pressure



Ammonia Adsorption and Desorption Isotherms at 0 °C

Equipment

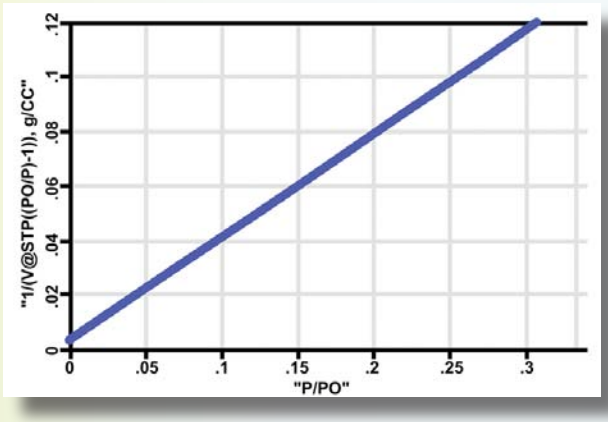
The PMI Sorptometer uses volumetric method to compute the amount of gas. The sample chamber is connected through valves to the reference volume, gas supply, & vacuum line. For a test, a weighed sample is placed in the sample chamber. The sample chamber of known volume is heated and evacuated to remove moisture and adsorbed gases. The desired adsorption temperature is then established in the sample chamber using a constant temperature bath, and the chamber is isolated. The reference volume is pressurized with adsorbate gas, and isolated. The pressure of reference volume is measured. The gas is allowed to expand into the sample chamber. After equilibration the gas pressure is measured. The amount of gas adsorbed by the material is calculated by making use of the gas law.



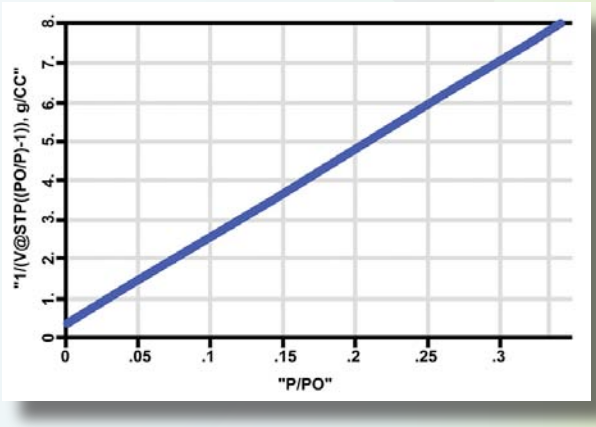
Capabilities

Surface Area:

PMI BET Sorptometers are capable of testing samples with moderate to high surface areas by using nitrogen gas. For samples with very low surface area, a larger amount of sample or krypton gas can be used for greater accuracy. The number and spacing of data points in multipoint surface area measurement are user adjustable.



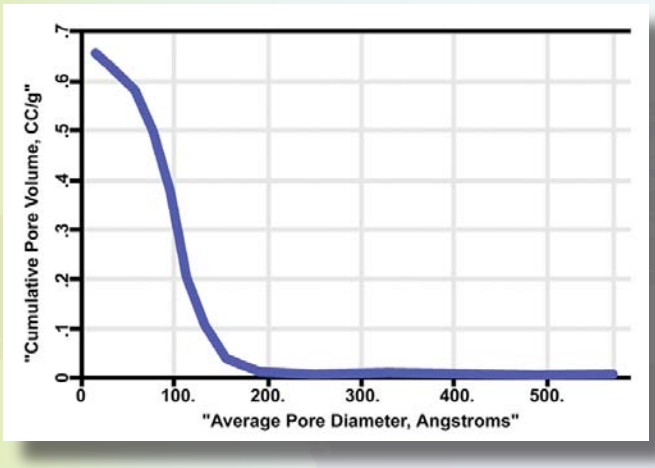
Surface Area Analysis — Nitrogen



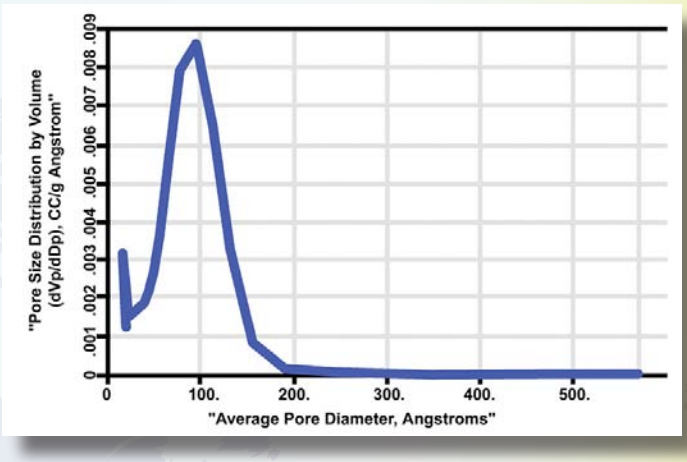
Surface Area Analysis — Krypton

Pore Volume and Pore Diameter

Pore volume, pore diameter and pore volume distribution can be determined accurately by the PMI Sorptometer. The distribution function is such that area under the function in any pore diameter range is the volume of pore in that range.



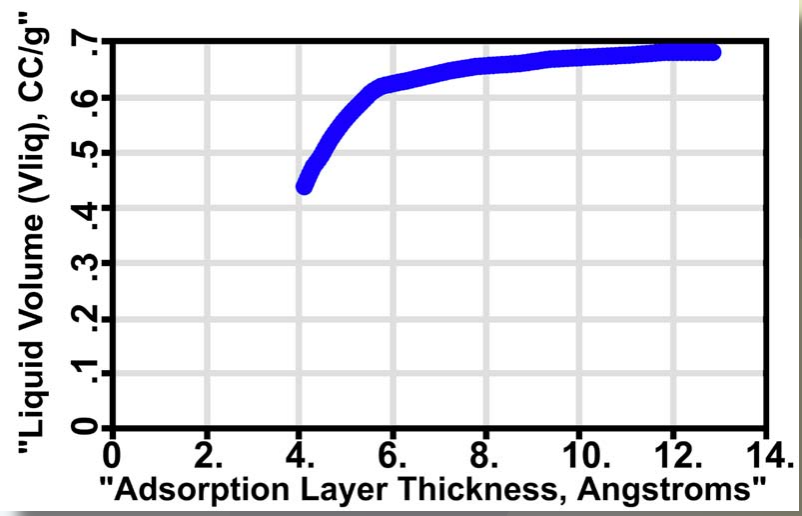
Cumulative Pore Volume



Pore Volume Distribution

Adsorption and Desorption Isotherms

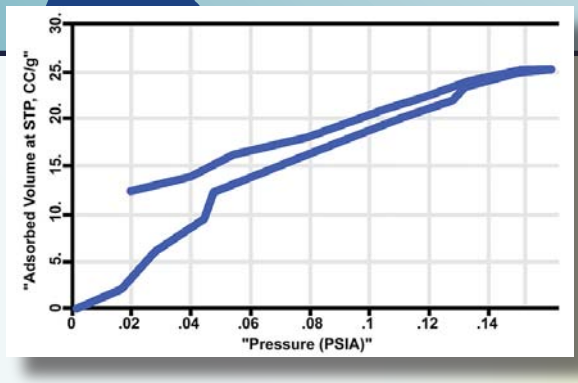
Adsorption and desorption of gasses on samples can be accurately measured using PMI BET sorptometers. The user has independent control over the quantity and spacing of pressures used in both adsorption and desorption testing. Many different kinds of analyses are available to interpret data using the supplied report generation software. Pore size calculation for adsorption and desorption include Pierce, BJK, and DH models. Microporous solids can be analyzed using T–Plot and H–K analysis.



T – Plot Method — Micropore Volume Analysis

Chemisorption:

PMI BET Sorptometers can use specialty gasses like NH3 and H2O for measuring chemisorption. Other gasses can be used by creating additional gas specification files. The user can specify any temperature and pressure of gas, limited only by the capabilities of the instrument and the equilibrium vapor pressure of the gas at the temperature selected.



Adsorption and Desorption Isotherms – Water Vapor at 0 °C

Specifications

Surface Area range: 0.01 m2 and higher.
Pore diameter range: Micropore to 2000Å.
Sample volume: Up to 10 cc (others available).
Pressure gauge: 10 torr to 500 psi.
Resolution: 1 part in 20,000
Accuracy: 0.15% of reading
Adsorption temperature: –195.6 deg C (liquid nitrogen) to 300 deg C. (higher temperature available)
Adsorbate: Any noncorrosive gas including N2, H2, CO, CO2, H2O, Kr, Ar.
Degassing system: Heater oven up to 500 deg C (Higher temperatures available).
Degassing and testing performed in–situ.
Power requirements: 110/220 Vac, 50/60 Hz

Unique Features

- Adsorption of chemically active substances like NH3, CO & CO2
- Adsorption of vapors of liquids like water, benzene and alcohol
- Adsorption studies using H2, N2, Ar & Kr
- Adsorption under pressures up to 500 psi
- Adsorption under extra low pressures down to 10 – 5 psi
- Adsorption studies at temperatures up to 300°C & higher
- Use of flow method by QBET series permits fast and reproducible surface area measurements
- Volumetric method employed measures equilibrium amount of adsorbed gas precisely without the possibility of any contamination
- Design features modifiable to satisfy your special requirement
- Any number of user specified data points between specified pressure limits
- Automated calibration routine for different sample chambers
- Continuous status display
- Continuous recording of equilibrium (saturation) pressure
- Data reduction software allows data analysis by many available procedures
  - Surface area: Single point, Multipoint
  - Pore Size: Pierce, NJH and DH models
  - Micropore: t–plot, Langmuir, D–R and H–K model
- Software enables curvefitting and interpolation of data, output to be written in text and Excel files, and multigraph analysis that can analyze up to seven graphs
- Fully automated and minimal operator involvement
- Windows based menu–driven procedure makes test execution, data acquisition, and data reduction very simple
- In–situ outgassing of samples at temperatures up to 800°C
- No need for transfer from outgassing station to test station and increase possibility of contamination
- Multiple sample chambers with provision to use different gases in different sample chambers, and simultaneous testing of multiple samples
- Unique design permits measurement of very low and high surface areas in the same instrument

PMI Analytical Services

PMI has many years of experience in analyzing porous materials using the gas adsorption technique. The analytical services division of PMI is well known for providing timely, accurate and reliable contract testing services. Contact PMI for details.

Models

QBET Series

Units in this series are basic simple instruments dedicated to quick generation of highly reproducible data on surface area using nitrogen or argon. Each unit has one sample chamber. Liquid nitrogen is the only accessory required to perform the test. Various models in this series are capable of measuring single point or multipoint surface area and pore volume. Theses units are least expensive, physically small, robust and require very little maintenance. Model Number: QBET–G–X–Y–A

CBET Series

Instruments in this series are volumetric compact units, but the capabilities are much better than those of the QBET series. These instruments can measure surface area, adsorption and desorption isotherms, pore volume and pore volume distribution using nitrogen, argon and krypton. The results are reproducible and accurate. Models in this series are inexpensive, and robust. These models require very little maintenance. Model Number: CBET–G–D–Y–A

ABET Series

Models in this series are the most advanced instruments in which many novel and advanced designs are incorporated. These instruments are capable of measuring very low adsorption, chemical adsorption and water vapor adsorption. The results are highly accurate and reproducible. Multiple sample chambers permit testing of as many as six samples at a time. A variety of gases may be used. The advanced series provides many options to the user. Model Number: ABET–G–D–Y–A–L–C–W

Brief Descriptions of Selected Models

Model: QBET–N–V–1–A
This fully automated sorptometer is a single–chamber instrument that can determine single point surface area, multipoint surface area and pore volume using nitrogen as the adsorption gas. The instrument is small, robust and inexpensive. It requires very little maintenance. The sorptometer is ideal for applications such as quality control requiring rapid generation of reproducible data.

Model: CBET–N / Kr/Ar–D–2–A
This table top compact model can measure single point surface area, multipoint surface area and pore volume using N2, Kr or Ar as the adsorption gas. The instrument is fully automated and has two sample chambers. Two samples can be tested simultaneously. This option gives the opportunity to compare samples with the standard. The model is also robust and inexpensive.

Model: ABET–G–D–6–A–L–C–W
This is a sophisticated unit with many capabilities. It uses gases like N2, Ar, Kr, H2 and CO2 for adsorption and measures single point surface area and pore volume distribution. Chemical adsorption of substances like ammonia, alcohol and benzene is measurable. The results are accurate and reproducible. The instrument is fully automated and requires very little operator involvement. The six sample chambers of the instrument permit simultaneous testing of six samples. The sorptometers are ideal for determination of a variety of physical and chemical characteristics.

Symbols

A = fully automated
C = chemical adsorption of many chemical species
D = surface area, pore volume and pore distribution
G = adsorption gas that could be N2, Ar, Kr, H2 or others
L = very low adsorption pressure
V = single point and multipoint surface area and pore volume
W = water vapor adsorption capability
X = type of test
Y = number of sample chambers

Highlights of PMI BET Sorptometer Models

Features	QBET SERIES	CBET SERIES	ABET SERIES
Principle	Flow	Volumetric	Volumetric
Design:	Simple	Moderately Complex	Complex
Gases	N_2/Ar	N_2/Ar/Kr	N_2/Ar/Kr/CO_2/ H_2/NH_3/Others
Liquid N_2 Bath:	Container with cover	Container with Cu–block and cover	**Refill system**
Measurable Properties:	Single point and multipoint surface area. Pore volume	Single point & mulitipoint surface area. Pore volume. Pore distribution. Desorption.	Single point & multipoint surface area. Pore volume. Pore distribution. Desorption. Very low adsorption. Chemisorption. Water vapor adsorption.
Reliability:	Highly reproducible	Highly reproducible and accurate	Highly reproducible and very accurate
Duration of Test:	Short	Moderate	Long
Size:	Small	Table Top	Floor Model
Maintenance:	Very Little	Very Little	Regular
Cost:	Low	Medium	High

New Sorptometers

PVT Apparatus

Other Products

Average Fiber Diameter Analyzer
Bubble Point Tester
Capillary Flow Porometer
Capillary Condensation Flow Porometer
Complete Filter Cartridge Analyzer
Clamp-On Porometer
Compression Porometer
Custom Porometer
Cyclic Compression Porometer
Envelope Surface Area Analyzer
Filtration Media Analyzer
High Flow Porometer
Integrity Analyzer
In-Plane Porometer
Microflow Porometer
Nanopore Flow Porometer
QC Porometer
Diffusion Permeameter
Gas Permeameter
Liquid Permeameter
Vapor Permeameter
Water Vapor Transmission Analyzer
Liquid Extrusion Porosimeter
Mercury/Nonmercury Intrusion Porosimeter
Vacuapore
Water Intrusion Porosimeter (Aquapore)
BET Liquisorb
BET Sorptometer
Gas Pycnometer
Mercury Pycnometer

Also Available:
Testing Services
Consulting Services
Short Courses

Buy Rent Lease

Porous Materials, Inc.
20 Dutch Mill Rd, Ithaca, NY 14850 USA
Tel: (607)-257-5544 Toll Free in USA & Canada: 1-800-TALK-PMI
Fax: (607) 257-5639 Email: info@pmiapp.com **WWW.PMIAPP.COM**

PMI Porous Materials, Inc.

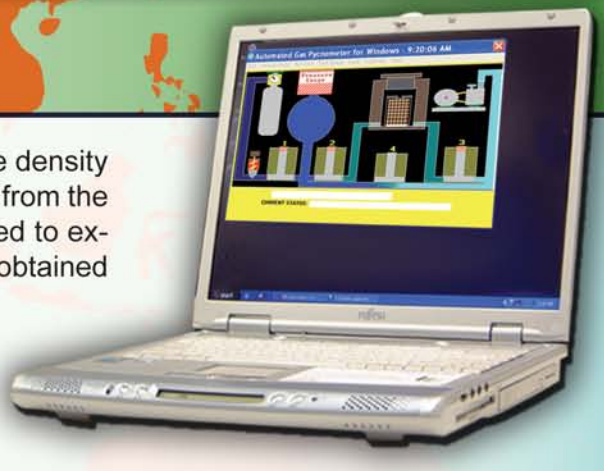
Gas Pycnometer

Principle

PMI gas pycnometer is used to determine the true volume and true density of powders and bulk solids. The true volume of a solid is calculated from the measured drop in pressure when a known amount of gas is allowed to expand into a chamber containing the sample. Thus, the true volume obtained by pycnometry excludes any pore volume accessible to the gas. Helium is the preferred gas, because it exhibits ideal gas behavior. However, almost any non-corrosive gas including air can be used. The true density is obtained by dividing the weight of the sample by true volume.

Operating Procedure

PMI gas pycnometer is used to determine the true volume and true density of powders and bulk solids. The true volume of a solid is calculated from the measured drop in pressure when a known amount of gas is allowed to expand into a chamber containing the sample. Thus, the true volume obtained by pycnometry excludes any pore volume accessible to the gas. Helium is the preferred gas, because it exhibits ideal gas behavior. However, almost any non-corrosive gas including air can be used. The true density is obtained by dividing the weight of the sample by true volume.



Specifications

Sample Size: 25 cc and 100 cc (others available upon request).
Number of sample chambers: 1, 2, 3, 4, or 5
Accuracy & Reproducibility: 0.1 %
Power requirements: 110/220 VAC, 50/60 Hz
Size: 12" X 28" X 22" (25 X 70 X 55 cm) (approximately).
Weight: 60 lbs (27 kg) (approximately).
Software: Windows 95/98/NT/00/ME compatible.

Porous Materials, Inc. Analytical Services Department
20 Dutch Mill Road
Ithaca, NY 14850 USA
Phone 607-257-4267, 2575544 or 1-800-825-5764
E-mail: info@pmiapp.com
www.pmiapp.com

GAS PYCNOMETRY ANALYSIS

Test Type: VACUUM
Test Date: 06-22-2001

Sample ID: Polypropylene resin
Mass: 10.481 gm

Reference Volume: 11.31 cc
Sample Chamber Volume: 24.96 cc

PFO PSIA	PIO PSIA	PI PSIA	PF PSIA	VOLUME (cc)	DENSITY (gm/cc)
00.003	00.003	09.798	04.399	11.073	00.947
-00.003	-00.002	09.802	04.394	11.051	00.948
-00.003	-00.002	09.796	04.392	11.055	00.948
-00.003	-00.003	09.792	04.394	11.076	00.946
-00.003	-00.003	09.796	04.393	11.060	00.948

Average Volume : 11.063 cc
Average Density : 0.947 +/- 0.001

Hardware

- Three different test methods provide the most accurate results: Vacuum, high pressure and ambient pressure.
- Pressure relief valve prevents over pressurization of pressure gauge.
- Any non-corrosive and non-absorbing gas can be used.
- Use of metering valve provides excellent control on the amount of gas (pressure) that can be used for the test.
- Slow evacuation for powder samples prevents powder from being dragged into the vacuum pump.
- Minimal operator involvement.

Software

- Windows 95/98/NT compatible software enables convenient use of the instrument.
- User defined pressures can be used to test the samples.
- The user can specify the number of times the test is to be repeated within the specified standard deviation.
- Automatic pressure and volume calibration routines for different kind of test methods.
- Software allows the user to perform a test in the manual mode.
- Software allows the user to store different test set tings so that the settings can be recalled and used for future testing.
- Test results can be stored to disk and printed directly.

Optional Features

- Elevated temperature testing (density of the sample at high temperature)
- Multiple sample chambers and reference volumes

Other Products

Average Fiber Diameter Analyzer
Bubble Point Tester
Capillary Flow Porometer
Capillary Condensation Flow Porometer
Complete Filter Cartridge Analyzer
Clamp-On Porometer
Compression Porometer
Custom Porometer
Cyclic Compression Porometer
Envelope Surface Area Analyzer
Filtration Media Analyzer
High Flow Porometer
Integrity Analyzer
In-Plane Porometer
Microflow Porometer
Nanopore Flow Porometer
QC Porometer
Diffusion Permeameter
Gas Permeameter
Liquid Permeameter
Vapor Permeameter
Water Vapor Transmission Analyzer
Liquid Extrusion Porosimeter
Mercury/Nonmercury Intrusion Porosimeter
Vacuapore
Water Intrusion Porosimeter (Aquapore)
BET Liquisorb
BET Sorptometer
Gas Pycnometer
Mercury Pycnometer

Also Available:
Testing Services
Consulting Services
Short Courses

Buy Rent Lease

Porous Materials, Inc.
20 Dutch Mill Rd, Ithaca, NY 14850 USA
Tel: (607)-257-5544 Toll Free in USA & Canada: 1-800-TALK-PMI
Fax: (607) 257-5639 Email: info@pmiapp.com **WWW.PMIAPP.COM**

02/09

Novel Characterization Techniques for Prediction of Tissue In-Growth in Reticulated Porous Materials

A. Datta, C. Friedman, Maybelle Jordan
Biomerix, New York, NY
and
Krishna Gupta, Akshaya Jena
Porous Materials, Inc, Ithaca, NY

Abstract

Pore volumes accessible to flow and liquid permeability of un-reticulated and reticulated foams were measured. The foams were implanted in rabbits, pigs, and dogs. The harvested implants were examined for tissue in-growth. Un-reticulated foam with low pore volume and liquid permeability showed no in-growth. Reticulated foam had high pore volume and liquid permeability and showed considerable tissue in-growth. Characterization techniques for pore volume and liquid permeability were very effective in predicting tissue in-growth characteristics of foams.

Introduction

Biocompatible substrates can serve as scaffolds onto which cells may attach, grow and proliferate and this tissue engineering approach can be used to regenerate new tissues and to replace and repair defective ones. Open cell biocompatible elastomeric and resilient foams have significant potential for use as scaffolds for repair and regeneration of defective tissue in orthopedic, wound healing, vascular embolization, and aneurysm control. A high void content together with an interconnected and intercommunicating network of pores that provide fluid permeability throughout the implantable device can permit cellular in-growth and proliferation leading to eventual bio-integration of the elastomeric and resilient foam substrate or implant.

Suitable characterization techniques for the interconnected and intercommunicating network of pores will, thus, be of great value for prediction of tissue in-growth in open cell porous materials. We have developed two novel techniques for quantifying parameters that can be used as predictors for tissue in-growth. The parameters are liquid permeability and accessible pore volume. These material characteristics were further correlated to the tissue response by examining the behavior of these materials as implants in animals.

Materials

- Porous matrix made from biodurable cross-linked elastomeric and resilient polyurethane was obtained by foaming using water as a foaming agent.
- The thin windows formed between the pores during the foaming process were removed by reticulation.
- Reticulation created a continuous passage throughout the entire porous matrix characterized by an inter-connected and inter communicating pore structure, improved the mechanical response by reinforcing the struts with deposition of the melted polymer from the cell windows, and enhanced the material's potential for fluid transport and tissue in-growth and proliferation.

Experimental Techniques

Liquid Extrusion Porosimetry

Principle

In this technique, the sample is placed on a membrane whose largest pore diameter is smaller than the smallest pore of interest in the sample that is being measured. The pores of the sample and the membrane are filled with a wetting liquid. The liquid from pores of the sample is displaced by application of differential pressure on a non-reacting gas on the sample. The gas pressure is progressively increased until all the fluid is removed from pores. Pressure needed to displace the wetting liquid from a pore is related to pore diameter by the following relation [1].

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

where:
p = differential pressure on the liquid in the pore
θ = contact angle of the wetting liquid on the sample
γ = surface tension of wetting liquid
D = pore diameter

Pressure required to displace liquid from the pores of the sample is insufficient to remove liquid from pores of the supporting membrane. Therefore, gas does not flow through the membrane, but the liquid displaced from pores of the sample flows out through the membrane, and is collected and measured. Measurement of pressure yields pore diameter and corresponding measurement of displaced liquid yields pore volume and pore distribution.

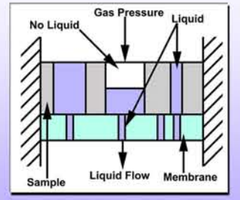


Figure 1. Principle of liquid extrusion porosimetry

Instrument

The fully automated instrument performs the test, regulates the pressures, acquires data, and calculates results in a controlled and reproducible fashion.

Capability

- Uses very low pressures. Thus, the distortion of pore structure is minimal.
- Does not use any toxic material.
- Customer specified liquid can be used in the test.

Liquid Permeametry

Principle

Liquid permeability is computed from the liquid flow rates measured through the sample using the liquid permeameter. In this instrument, the application liquid is pushed from the top or bottom of the sample and the liquid is made to flow under desired differential pressure applied using an inert gas. The pressure difference across the sample and the flow rate of the liquid through the sample are accurately measured [2]. Liquid permeability is computed from flow rate using Darcy's law [3]:

$$v = -(k / \mu)(dp/dx) \quad (2)$$

where
v = linear fluid flow rate
p = pressure
k = permeability
x = linear displacement
μ = viscosity of fluid

Assuming permeability to be a constant over the thickness of the sample and converting linear velocity, v, to volume flow rate, F, integration of Equation 2 yields:

$$F = -(k A / \mu l)(p_i - p_o) \quad (3)$$

where
l = thickness of the sample
A = area of sample
p_i = inlet pressure
Permeability, k, is computed in Darcies
p_o =outlet pressure

Figure 2. Liquid Extrusion Porosimeter

Figure 3. Liquid Permeameter used in this study

Instrument

The fully automated instrument performs the test, regulates the pressures, acquires data, and calculates results in a controlled and reproducible fashion.

Capability

- Minimal pressure on sample and minimal structural distortion of the pores
- Customer specified liquid usable in the test
- Wide range of measurement capability from very high to very low permeability
- Permeability measurable on materials subjected to different compression

Animal Test

Study 1 - Implant placed in the carotid artery of rabbits

- Implant: Oversized occlusive implant measuring 3 mm diameter and 10 mm length
- Implants placement: By open surgical procedure
- Animals sacrifice: At 24 hours, 2 weeks and 4 weeks
- Harvested implant: Examined for tissue in-growth, biocompatibility and biointegration

Study 2 - Implant placed in the right iliac artery of pigs

- Implant: Oversized occlusive implant measuring 4 - 8 mm diameter and 15 mm length
- Implants delivery: Percutaneously via catheter for peripheral embolization
- Animals sacrifice: At 1 week, 1 month and 3 months
- Harvested implant: Examined for tissue in-growth, biocompatibility and biointegration

Study 3 - Implant placed in the spinal annulotomy of mini pigs.

- Implant: Implant measuring 12 mm diameter and 18 mm length
- Implants delivery: By open surgical procedure
- Animals sacrifice: At 3 week and 6 week
- Harvested implant: Examined for tissue in-growth, biocompatibility and biointegration

Study 4 - Implant placed adjunctive to stent-graft in aneurysm sac in dogs

- Implant: Implants measuring 10 mm diameter and 20 mm length
- Implants delivery: Percutaneously by femorally accessed catheters to prevent and treat Type II endoleaks
- Animals sacrifice: At 1month and 3 months
- Monitoring of pressure: Intra aneurysmal pressure (defined as the pressure on the aneurysmal vessel wall from any blood flow within the perigraft space in the sac) and systemic pressures
- Harvested implant: Examined for tissue in-growth, biocompatibility and biointegration

Results and Discussion

SEM Foam Structure

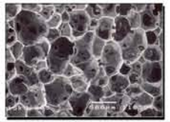


Figure 4. Reticulated foam structure showing an inter-connected and inter-communicating pore structure. Reticulation created a continuous passage throughout the entire foam.

Material Properties

- Material has been tested extensively to demonstrate that it is non-cytotoxic, non-mutagenic, non-toxic, non-pyrogenic, and non-clastogenic

Density	2.70 – 3.20 pcf
Tensile Strength	15 – 35 psi
Elongation at Break	175 – 225 %
Compressive Strength @ 50% strain	0.75 – 1.25 psi
Compression Set	5 – 10 %
Average pore size by SEM	250 – 300 microns
Dynamic Recovery (90%)	< 5 s after 10 minutes @ 75 % strain

Characterization of Pore Structure

A – Accessible Pore Volume and Volume Distribution

- Pore volumes of reticulated (Figure 5) and un-reticulated foams were measured
- Inert wetting liquid Galwick® was used for the test
- Less than 0.5 psi pressure was needed for the test

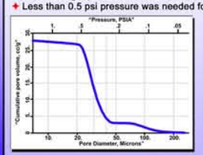


Figure 5. Measured cumulative pore volume of the reticulated foam

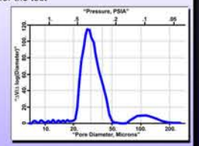


Figure 6. Measured pore volume distribution of the reticulated foam

- Distribution function (Figure 6) is such that area under the curve in any pore diameter range is the volume of pores in that particular range.
- In this technique as the liquid is displaced in the pore structure consisting of cells connected to each other through small openings or windows, the volume of the liquid in the pore downstream from the window is measured as the volume of the pore having the diameter equal to the diameter of the window. Thus, the diameter of the windows, which normally would have gone undetected is detected and measured by this technique.
- The technique measures cell diameters in the range of about 80 -300 microns, which is in the same range provided by microscopic observations. However, the technique also provides additional information about average small openings or window diameters of around 30 microns not measurable by microscopy.
- Measurement of window diameter is important because it provides the primary resistance to flow as well as in-growth and proliferation of tissue through adjacent pores in the overall pore structure.

B - Liquid Permeability

- Liquid (water) permeability of reticulated and un-reticulated foams were measured (Figure 7).

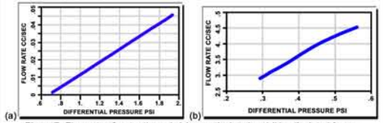


Figure 7. Flow rate of water through (a) un-reticulated and (b) reticulated foams

- Liquid permeability was computed from flow rate using Darcy's law
- The flow rate is very low for un-reticulated and high for reticulated matrix.
- Effects of compressive strain or compressive stress on the foam can change the permeability of the implant considerable. These effects can be quantitatively evaluated.

C - Effect of Reticulation on Accessible Pore Volume and Liquid Permeability

Foam Characteristics	Intrusion volume, cc/gm	Permeability, Darcy [Flow rate of water = (Darcy)×4.08×10-3 l/min-(psi/cm)-cm2]
Un-reticulated	4	0.54
Reticulated	28	205

- Reticulation significantly improved the fluid accessibility by the presence of the continuous passage throughout the matrix.
- It is measured by the increase in intrusion volume or the volume of inter-connected and intercommunicating pores which permit fluid flow, which in turn is determined by the dimensions of the opening of the window between cells.
- Tissue in-growth can occur in fluid accessible continuous passage and the extent and amount of proliferation can be potentially predicted depending on intrusion volume.
- Liquid Extrusion Technique was able to differentiate between un-reticulated foam with very small volume of accessible pores and reticulated foam with much larger volume of accessible pores.
- Un-reticulated foam permitted very little flow of fluid while reticulated foam, owing to inter-connected and intercommunicating pores, permitted flow that is several orders of magnitude higher.
- Presence of higher flow again indicates potential for tissue in-growth and proliferation.
- Liquid Permeability was sensitive in characterizing the transformation in the matrix morphology as it relates to flow of fluid through the matrix.

D - Effect of compression on Darcy's Constant

	Percentage Drop in Darcy's Constant as a function of Compression Strain			
	0% compression	25% compression	50% compression	100% compression
Reticulated Matrix	198.90	130	56	89

- The variability of flow arising out of different compressive strains on the matrix was measurable.
- Changes in Liquid Permeability was measured as a function of compressive strain.

Animal Studies

Study 1 - Implant placed in the carotid artery of rabbits

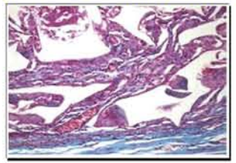


Figure 8. Histology of harvested carotid artery implanted with reticulated foams at 4 weeks

Study 2 - Implant placed in the right iliac artery of pigs

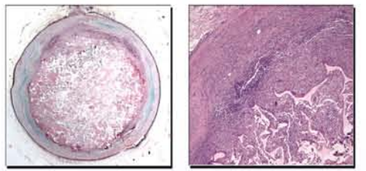


Figure 9. Harvested pig iliac artery containing reticulated foams and the associated histology at 3 months

Study 3 - Implant placed in the spinal annulotomy of mini pigs.

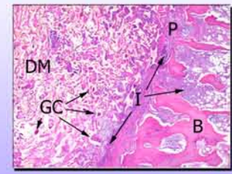


Figure 10. Histology of harvested mini pig spine annulus with reticulated foams at 6 weeks

Study 4 - Implant placed in the aneurysm sac adjunctive to stent-graft in dogs

Pressure Measurements of Treatment and Control Animals in an Canine Model of AAA Endoleaks

Test Systems	Systolic Pressure*	Mean Pressure*	Endoleak Patency
Patent Type II Endoleak	0.702	0.784	Patent
Polyurethane Treated Type II Endoleak	0.183	0.142	Thrombosed
Control (No Endoleak/ No Branches)	0.172	0.137	Thrombosed
Systemic Pressure	1.0	1.0	NA
P-Value (Patent vs. Polyurethane Treated)	<0.001	<0.001	<0.001

Note *All pressures listed were measured after antegrade AAA exclusion and are indexed as a percentage the systemic pressure

- All arterial vessels into which the implant were placed showed total occlusion following placement of the implant and remained as occlusive barrier through the end of the study.
- Treatment of endoleaks with reticulated implants leads to complete elimination of intra aneurysmal pressure in the sac making it indistinguishable from controls with no aneurysm.
- As the healing progressed, extensive tissue in-growth and proliferation occurred into the reticulated matrix accompanied by organizing fibrin/platelet rich thrombus.
- No evidence of fibrous encapsulation was observed for any of reticulated implants – a phenomenon expected and observed with un-reticulated matrices.
- The extent of tissue infiltration scaled fairly well with the accessible pore volume of the implants.
- Expected cellular response to porous materials with no necrosis, mild to moderate inflammation that subsides with time and none to decreasing granular tissue formation across multiple animal studies
- Implants were atraumatic to vessel walls.
- Overall, the reticulated implants were successfully bio-integrated [4, 5].

Summary and Conclusions

- A reticulated, biodurable, cross-linked, elastomeric, and resilient polyurethane matrix was used for this study.
- Implants made from this matrix were surgically implanted in a variety of tissues using different animal models through various time points from 1 week through 3 months.
- Material supported extensive tissue in-growth and proliferation including soft tissue (rat RCR and rabbit subcutaneous – not shown here), fibrocollagenous (mini-pig & rabbit annulus), and vascular (carotid artery, abdominal & carotid bifurcation aneurysms, external iliac).
- No adverse reaction was noticed and the material was shown to be very biocompatible with complete bio-integration across various tissue types.
- Accessible Pore Volume and Liquid Permeability of un-reticulated and reticulated matrices were measured using novel techniques developed by Porous Materials, Inc, Ithaca, NY.
- Measurement of pore volume accessible to flow and, thus, to tissue is an excellent qualitative and quantitative indicator for predicting the degree or extent of tissue in-growth and proliferation. The extent of tissue infiltration scaled fairly well with the accessible pore volume of the implants
- Permeability of matrix under relaxed and compressed conditions is indicative of the degree of interconnectivity of the matrix. These results can be used as indicators for tissue in-growth and proliferation especially when the implant configuration and geometry are expected to change from post implantation to delivery.

References

1. Akshaya Jena and Krishna Gupta, Liquid Extrusion Techniques for Pore Structure Evaluation of Nonwovens, International Nonwovens Journal, Fall, pp.45-53, 2003.
2. Akshaya Jena and Krishna Gupta, 'Evaluation of Permeability of Strong Chemicals at Elevated Temperatures and High Pressures', Proceedings of the 2002 17th Annual Battery Conference on Applications and Advances, California State University, Long Beach, California, December 2001, IEEE Catalog Number 02TH8576.
3. A. E. Scheidegger, The Physics of Flow Through Porous Media, Macmillan, 1957.
4. S.J. Peter, M.J. Miller, A.W. Yasko, M.J. Yaszemski, and A.G. Mikos, "Polymer Concepts in Tissue Engineering," J. Biomed. Mater. Res. (Appl. Biomater.), 43, 422-427 (1998).
5. S. Yang, K. Leong, Z. Du and C. Chua, "The Design of Scaffolds for Use in Tissue Engineering Part I", Tissue Engineering, 7, 679 - 689

PORE VOLUME OF NANOFIBER NONWOVENS

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

ABSTRACT

Pore volume, pore diameter, pore volume distribution and pore throat diameters of nanofiber mats were measured using mercury intrusion porosimetry, liquid extrusion porosimetry and capillary flow porometry. Analysis of results showed that mercury intrusion distorts the structure due to application of high pressure. Liquid extrusion does not require high pressures, gives good resolution and measures pore structure relevant for application. Capillary flow porometry uses low pressures, measures pore throat diameter, but does not measure pore volume.

Key Words: Pore Volume. Pore Structure. Nanofiber. Liquid Extrusion. Mercury Intrusion. Flow Porometry

INTRODUCTION

Nanofiber nonwovens are finding increasing applications in filtration industry particularly in processes involving biotechnology. For such applications, pore volume is very important. Through pore diameter, pore throat diameter and permeability are also important pore structure characteristics. Nanofiber mats are normally sensitive to pressure and are often brittle. Therefore, the characterization technique should be such that the pore structure is not distorted. In this investigation, the applicability of the techniques, Mercury Intrusion Porosimetry, Liquid Extrusion Porosimetry and Liquid Extrusion Flow Porometry for pore structure characterization of nanofiber mats have been investigated. The results obtained by the three techniques have been critically examined.

CHARACTERIZATION TECHNIQUE

Mercury Intrusion Porosimetry

Mercury is non-wetting to most materials. It does not enter the pores spontaneously. Intrusion of mercury into pores occurs due to pressure applied on mercury. Pressure is used to compute pore diameter [1].

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

where D is pore diameter, γ is surface tension of mercury , θ is contact angle of mercury and p is pressure on mercury for intrusion into the pore. Intrusion volume gives pore volume.

The technique measures pore volume and pore diameter of through and blind pores. As shown in Figure 1, all diameters and volumes of through and blind pores are measured.

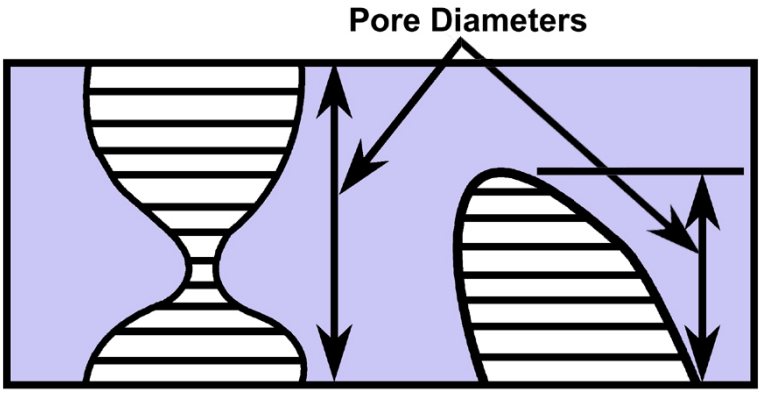


Figure 1. All diameters and volumes of through and blind pores measurable by mercury intrusion porosimetry.

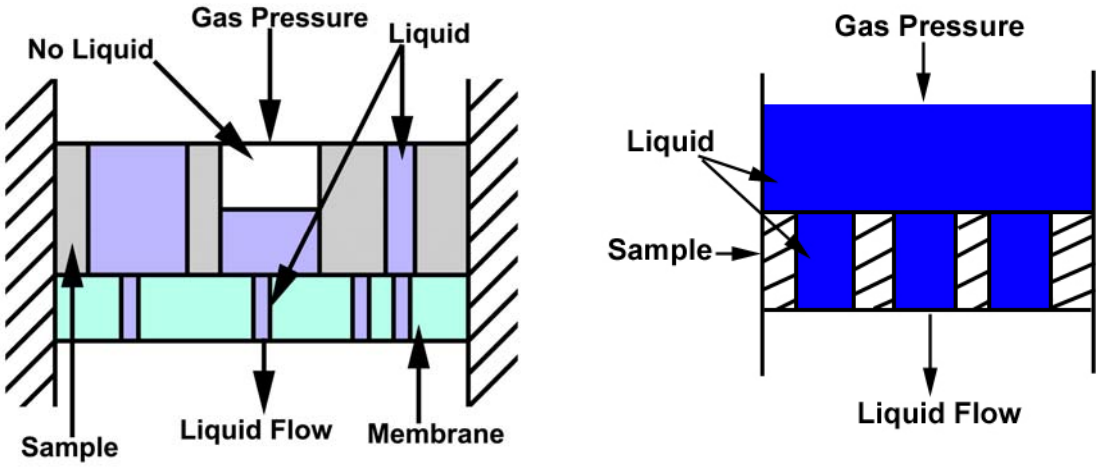
Liquid Extrusion Porosimetry

In liquid extrusion porosimetry, the pores are spontaneously filled with a wetting liquid and the liquid is extruded from pores by a non-reacting gas. It can be shown that the differential pressure is related to pore diameter [2,3].

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

where D is pore diameter, γ is surface tension of wetting liquid, θ is the contact angle of the wetting liquid and p is differential pressure.

In this method, the volume of extruded liquid is measured in addition to the differential pressure. In order to allow the extruded liquid to flow out and prevent the gas to escape, a membrane is placed under the sample such that the largest pore of the membrane is smaller than the smallest pore of interest in the sample (Figure 3a). The pores of the sample and the membrane are filled with a wetting liquid and pressure on gas is increased to displace the liquid from pores of the sample. The gas pressure is inadequate to empty the pores of the membrane. Therefore, the liquid filled pores of the membrane allow the extruded liquid from the pores of the sample to flow out while preventing the gas to escape. The measured volume of the liquid flowing out of the membrane gives through pore volume. Differential pressure yields through pore diameter and variation of volume with pressure yields through pore surface area. Flow rate of excess liquid maintained on the sample yields liquid permeability (Figure 3b).



(a) Pore Volume (b) Permeability

Figure 3. Principle of liquid extrusion porosimetry.

This technique measures only the volume and diameters of through pores (Figure 4) [3]. Blind pores are not measured (Figure 1). As shown in Figure 4, only some of the diameters of the through pore are measured, where as mercury intrusion measures all pore diameters (Figure 1).

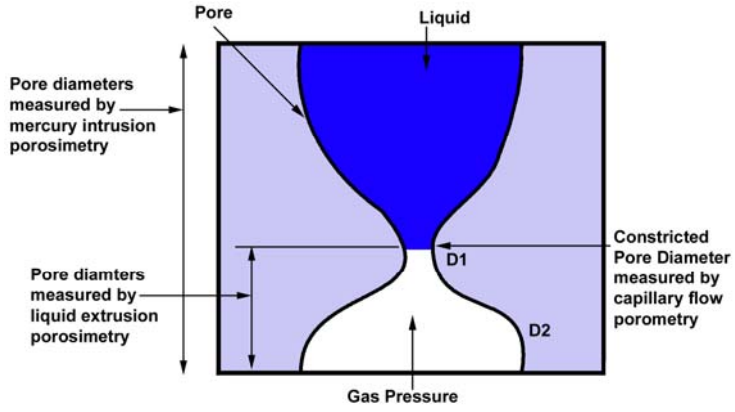
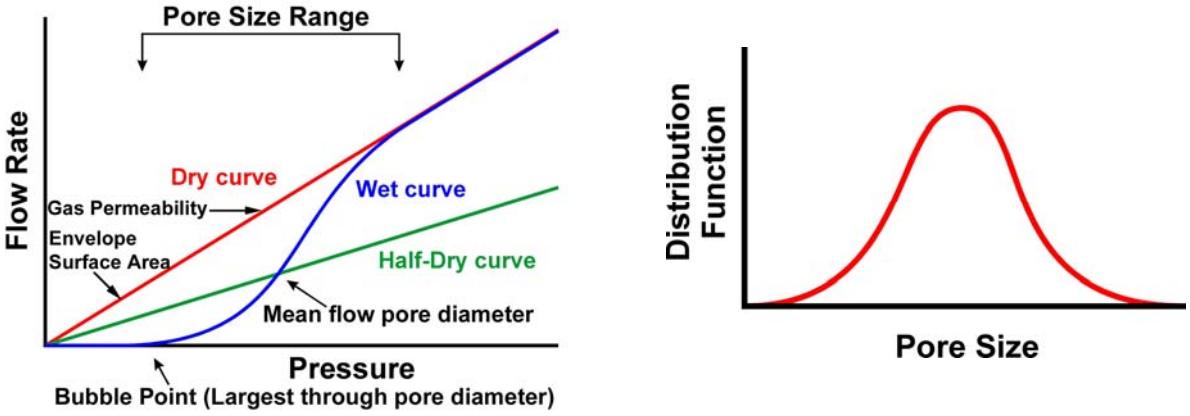


Figure 4 Pore diameters of a through pore measurable by Liquid Extrusion Porosimetry

Liquid Extrusion Flow Porometry (Capillary Flow Porometry)

In this method, the pores of the sample are filled with a wetting liquid, the liquid is emptied by a pressurized gas permitting gas to flow through the empty pores. The differential pressure required to empty a pore of diameter D is given by Equation 2 [1, 4]. It shows that the largest pore is emptied at the lowest pressure and initiates gas flow. With increasing pressure smaller pores are emptied and gas flow increases. The differential pressures and gas flow rates through dry and wet samples are measured. In the dry sample, the flow rate 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 2) 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 5. 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. The dry curve yields gas permeability and envelope (through pore) surface area. Liquid flow rate gives liquid permeability.



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

(b) Pore distribution

Figure 5. Through pore characteristics measurable by capillary flow porometry.

Capillary flow porometry measures only the throat diameter of each through pore (Figure 4). One diameter per through pore is measured. Blind pores are not measured (Figure 1).

RESULTS AND DISCUSSION

Through Pore Volume

Mercury Intrusion Porosimetry yields volume of through and blind pores as a function of intrusion pressure. However, nanofiber mats are not expected to contain appreciable amounts of

blind pores. Therefore, the measured intrusion volume is due to the volumes of through pores. Extrusion porosimetry measures only the through pore volume as a function of extrusion pressure. The pore volumes measured in both techniques are shown as function of pressure in Figure 6.

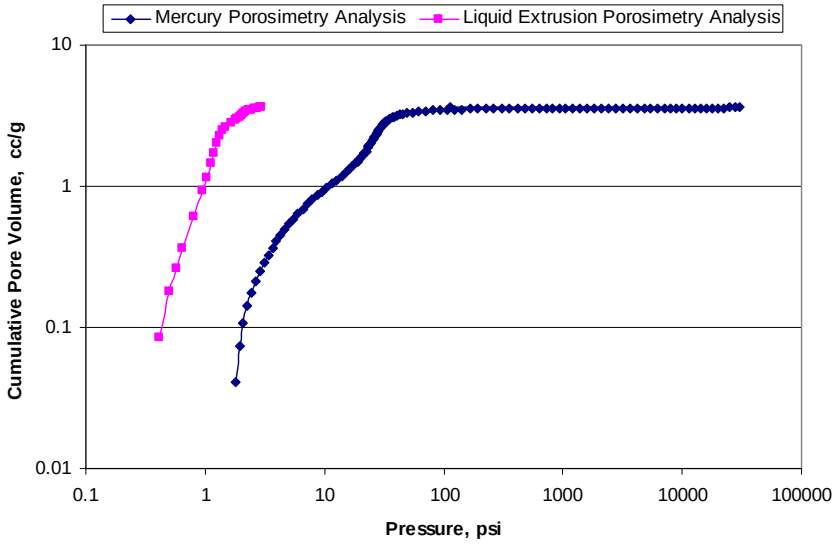


Figure 6. Through pore volume measured as functions of pressure in nanofiber mats using PMI Mercury Intrusion Porosimeter and Liquid Extrusion Porosimeter.

The total pore volumes measured by both techniques are the same (Table 1). This observation confirms that the blind pores in the nanofiber mats are negligible. However, the measurement pressure for mercury intrusion is almost twenty times higher than the test pressure required for liquid extrusion. The high test-pressure for mercury intrusion is likely to distort the pore structure of nanofiber mats.

Table 1 Through pore volume, test pressure and porosity measured by the mercury intrusion and liquid extrusion techniques.

Technique	Through Pore Volume	Approximate Test Pressure	Porosity
Mercury Intrusion	3.62 cc/g	>100 psi	81.1 %
Liquid Extrusion	3.65 cc/g	5 psi	81.7 %

Porosity

Blind pores in the nanofiber mat are negligible. Therefore, porosity of the nanofiber mat is computed from the measured pore volume and bulk density of the material. As expected porosity is high for the nanofiber material. Both techniques yield the same value (Table 1).

Pore Diameter

Figure 7 shows a plot of pore volume against pore diameter measured by mercury intrusion porosimetry and liquid extrusion porosimetry. Pore volume of wide pores measured by liquid extrusion is less than the pore volume of wide pores measured by mercury intrusion. But pore volumes of small pores measured by liquid extrusion are larger than those measured by mercury intrusion. Such behavior is expected. As explained above, the diameters and volumes of wider parts of a wide mouth pore (Figure 4) beyond the throat are not measured by liquid extrusion porosimetry, but the volumes of these parts of the pore are measured as the volume of the small pore of constricted diameter. Therefore, volume of large pores measured by liquid extrusion is smaller and volume of small pores measured by liquid extrusion is larger than the volumes measured by mercury intrusion.

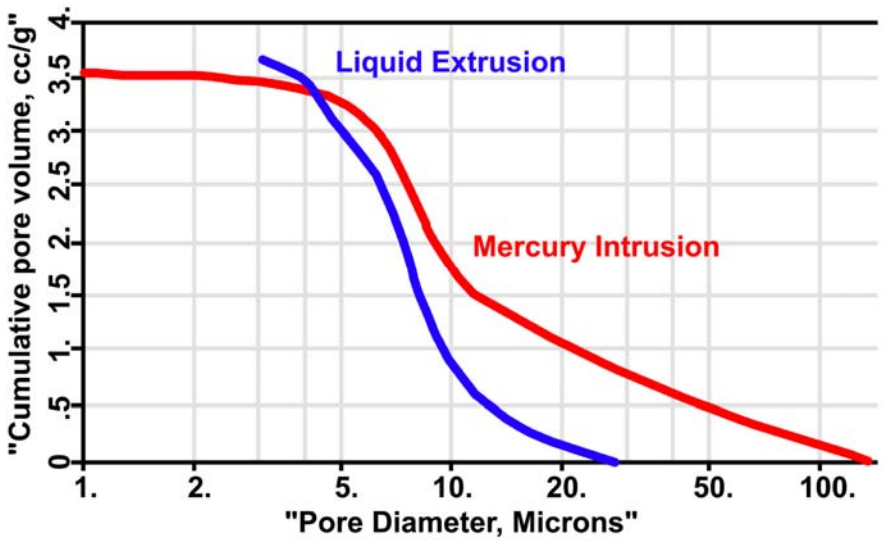


Figure 7. Pore volume plotted against pore diameter measured by liquid extrusion and mercury intrusion.

High intrusion pressure of mercury can also shift the mercury intrusion curve relative to the liquid extrusion curve by distorting the pore distribution. Before mercury intrusion could start, the pressure of mercury can compress the sample and measure intrusion volume due to volume reduction of the sample by compression. Thus, mercury intrusion will show higher intrusion volume when the pores are large. With increasing mercury pressure smaller pores will be reduced in diameter. Although, the volume reduction by compression of the sample is measured as the pressure goes up, the volume of smaller pores are not measured. Therefore, volume of small pores measured by mercury intrusion will be smaller than the volume measured by liquid extrusion.

Liquid extrusion and mercury intrusion techniques do not give the pore throat diameter, the largest pore throat diameter and the mean flow throat diameter of the nanofiber mat. Capillary flow porometry is capable of such measurements. The results obtained with capillary flow porometry are shown in Figure 8.

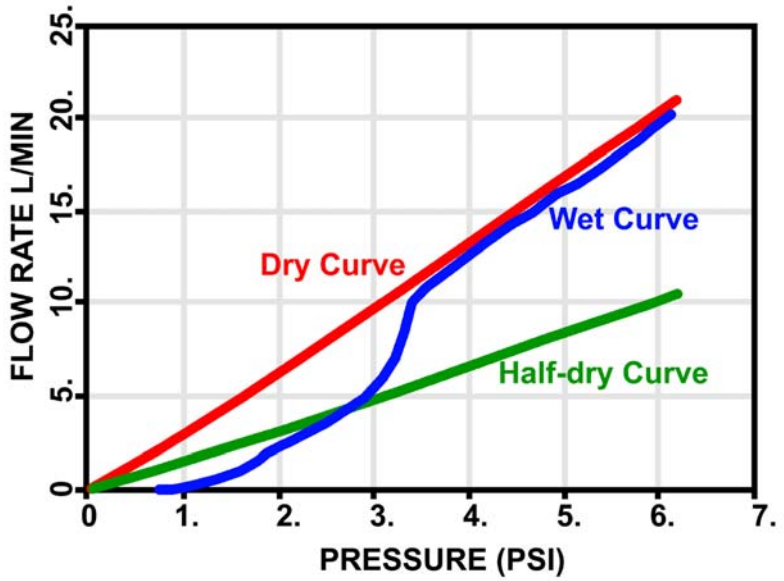


Figure 8. Differential pressure and flow rate through wet and dry samples of nanofiber mat measured by the PMI Capillary Flow Porometer.

The largest pore throat diameter (bubble point pore diameter) and the mean flow pore diameter computed from the data in Figure 8 are 12.50 and 3.36 μm respectively. Figure 8 shows that the test pressure for this technique is also very low so that the influence of pressure may be taken to be negligible.

Pore Volume Distribution

The pore volume distribution over pore diameter is expressed in terms of the distribution function F_v

$$F_v = - (dV / d \log D) \qquad (3)$$

where V is pore volume. The function is such that area under the function in any pore diameter range yields volume of pores in that range. Figure 9 shows the pore volume distribution computed from data in Figure 7 based on mercury intrusion and liquid extrusion.

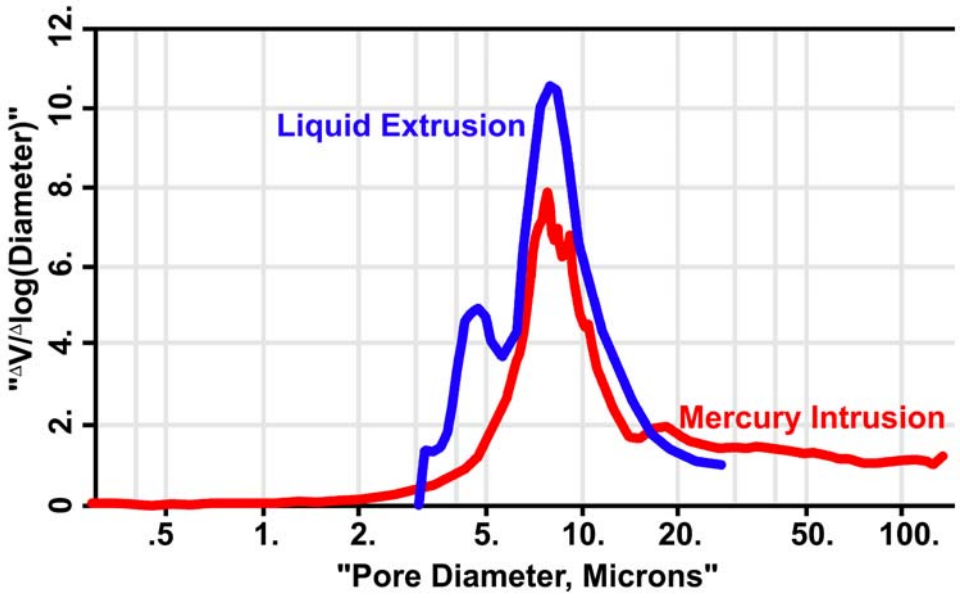


Figure 9. Through pore volume distribution over pore diameter by mercury intrusion and liquid extrusion

Mercury intrusion yields only a single peak where as liquid extrusion yields two peaks. The first peak represents about 75 % of pore volume and the second peak represents about 25 % of pore volume. Thus the resolution is much higher in liquid extrusion. In this technique, smaller pores are more easily detectable because volume of pores beyond the pore constriction is associated with the constricted pore diameter. The smaller pores are not seen by mercury intrusion because of the high pressure. With increase in pressure mercury enters large pores and reduces the diameters of small pores by compressing them. Thus, the peak due to the small pores is suppressed.

The flow distribution is expressed in terms of distribution function F.

$$F = - [(F_w/F_d)\times 100] /dD \qquad (4)$$

where F_w and F_d are flow through wet and dry samples respectively. The function is such that area under the function in any pore diameter range is the percentage flow in that range. Figure 10

shows the flow distribution over pore diameter computed from data in Figure 8. The peak of the flow distribution curve is at 2 μm. Characteristics of distributions are listed in Table 2.

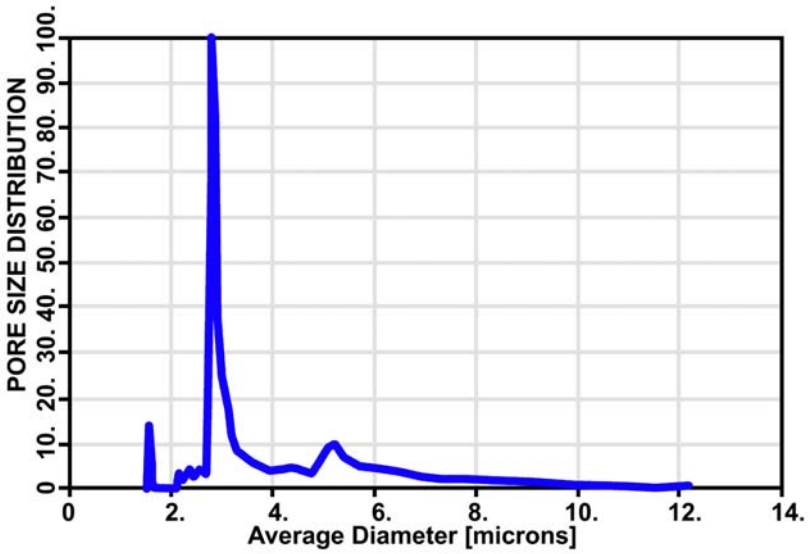


Figure 10. Flow distribution over pore throat diameters of through pores of nanofiber mats.

Table 2. Characteristic diameters of through pores.

Characteristic	Mercury Intrusion Porosimetry	Liquid Extrusion Porosimetry	Capillary Flow Porometry
3. Median pore diameter (Volume based), μm	9.73	7.69	—
4. Peak of volume distribution, μm	7.72	4.78, 7.83	—
5. Largest throat diameter, μm	—	—	12.50
6. Flow based mean throat diameter, μm	—	—	3.36
7. Peak of flow distribution, μm	—	—	2.80

Mercury intrusion yields a larger median pore diameter because the pore volume of large pores measured by mercury intrusion is larger than that measured by liquid extrusion. The volume

distribution peak at 4.78 μm is completely suppressed by mercury intrusion. The bubble point diameter is similar to the median pore diameter suggesting that the widest parts of pores are not much larger than the median value. The mean throat diameter and the throat diameter corresponding to the distribution peak are close to the volume distribution peak at 4.78 μm.

Appropriate Technique for Characterization of Nanofiber Mats

Through pore volume of nanofiber mats can be measured by liquid extrusion porosimetry and mercury intrusion porosimetry. However, mercury intrusion requires high pressures. High pressures reduce pore diameter and further increase in pressure is required to measure the smaller pores. Thus, pore diameters relevant for application are not measured. Pore distribution is suppressed. Peak due to smaller pores is not observed. On the other hand, liquid extrusion requires very little pressure (about 5 % of the pressure needed for mercury intrusion), produces excellent resolution of peaks and yields the same total pore volume as mercury porosimetry. In addition, liquid extrusion technique is capable of measuring liquid permeability and surface area of through pores, which are important for attachment. Mercury intrusion cannot measure these properties. Therefore, liquid extrusion technique is undoubtedly more appropriate for characterization of pore volumes of nanofiber mats.

For filtration and tissue growth applications, pore throat diameters of nanofiber mats are required in addition to pore volume. Mercury intrusion and liquid extrusion techniques cannot measure pore throat diameter. Capillary flow porometry measures pore throat diameters without distorting pore structure because this technique uses low pressures. Therefore, capillary flow porometry is a good technique to use along with liquid extrusion technique when nanofiber mats are used for filtration applications.

SUMMARY AND CONCLUSION

1. Mercury intrusion porosimetry, liquid extrusion porosimetry and capillary flow porometry were used to characterize nanofiber mats. Through pore volume, pore diameter, volume distribution and pore throat diameters were measured.
2. Analysis of results showed that both mercury intrusion and liquid extrusion yielded the same total pore volume and porosity, but pore diameter and distribution were distorted by high pressure applied during mercury intrusion. The pore diameters relevant for application were not measured. Liquid extrusion technique did not have these disadvantages and had much higher resolution. The peak due to small pores was clearly seen
3. Liquid extrusion technique is the appropriate technique for testing nanofiber mats.
4. Capillary flow porometry cannot measure pore volume, but uses low pressures and measures pore throat diameters, which are not measurable by liquid extrusion or mercury intrusion. This is the appropriate technique to support the liquid extrusion technique

REFERENCES

1. Akshaya Jena and Krishna Gupta, ‘Characterization of Pore Structure of Filtration Media’, Fluid particle Separation Journal, Vol. 14, No. 3, 2002, pp. 227-241.
2. A. Jena and K. Gupta, ‘A Novel Technique for Pore Structure Characterization without the Use of Any Toxic Material’, Nondestructive Characterization of Materials XI, Eds. R. E. Green Jr., B. B. Djordjevic and M. P. Hentschel, Springer, 2002, pp. 813-821.
3. Akshaya Jena and Krishna Gupta ‘Liquid Extrusion Techniques for Pore Structure Evaluation of Nonwovens’, International Nonwovens Journal, Fall, 2003, pp. 45-53.
4. A. K. Jena and K. M. Gupta, ‘In-Plane Compression Porometry of battery separators’, Journal of Power Sources, Vol. 80, No. 1-2, 1999, pp. 46-52.