Wetting Liquid Extrusion Techniques: Part II- Liquid Extrusion Porosimetry

Dr. Krishna Gupta

Porous Materials, Inc.

OVERVIEW

- Basic Principles
- Liquid Extrusion Porosimeter
- Measurable Pore Structure Characteristics
- Special Features
- Comparison with Mercury Intrusion Porosimetry
- Wide Applicability
- Conclusions

BASIC PRINCIPLES

This is a Wetting Liquid Extrusion Technique

- Wetting liquids spontaneously fill the pores of sample
- Differential gas pressure can be used for extrusion of wetting liquid from pores
- Pore diameter determines the required pressure

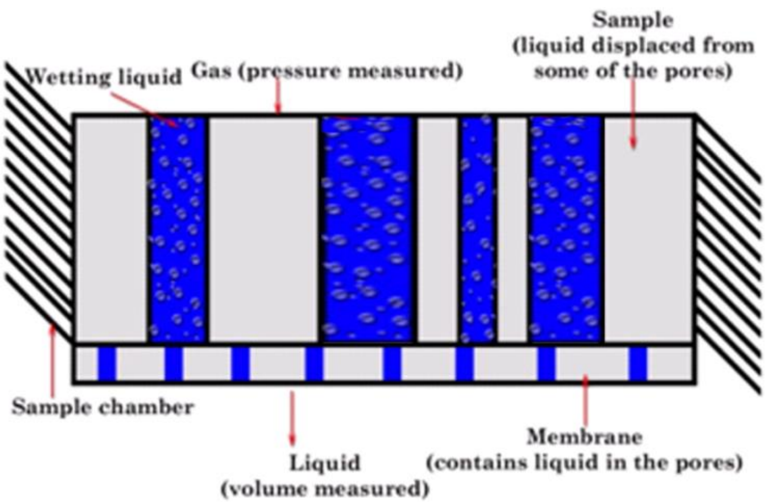
$$p = \frac{\left[4 \cdot \gamma_{l/g} \cdot \cos\theta\right]}{D}$$

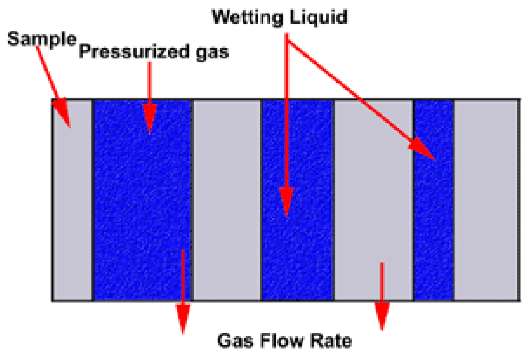
p = differential pressure
γ = surface tension of the liquid
D = pore diameter
θ = contact angle

BASIC PRINCIPLES

Volume of extruded liquid = Pore volume

- How can the liquid be collected and measured without interference from gas?
- Consider supporting sample on a membrane containing wetting liquid filled pores smaller than sample pores





BASIC PRINCIPLES

Gas & liquid flowing out of the pore

↓

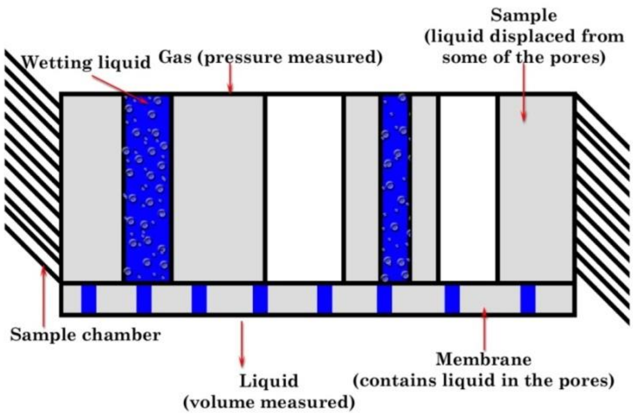
Test pressure required to empty sample pores

<

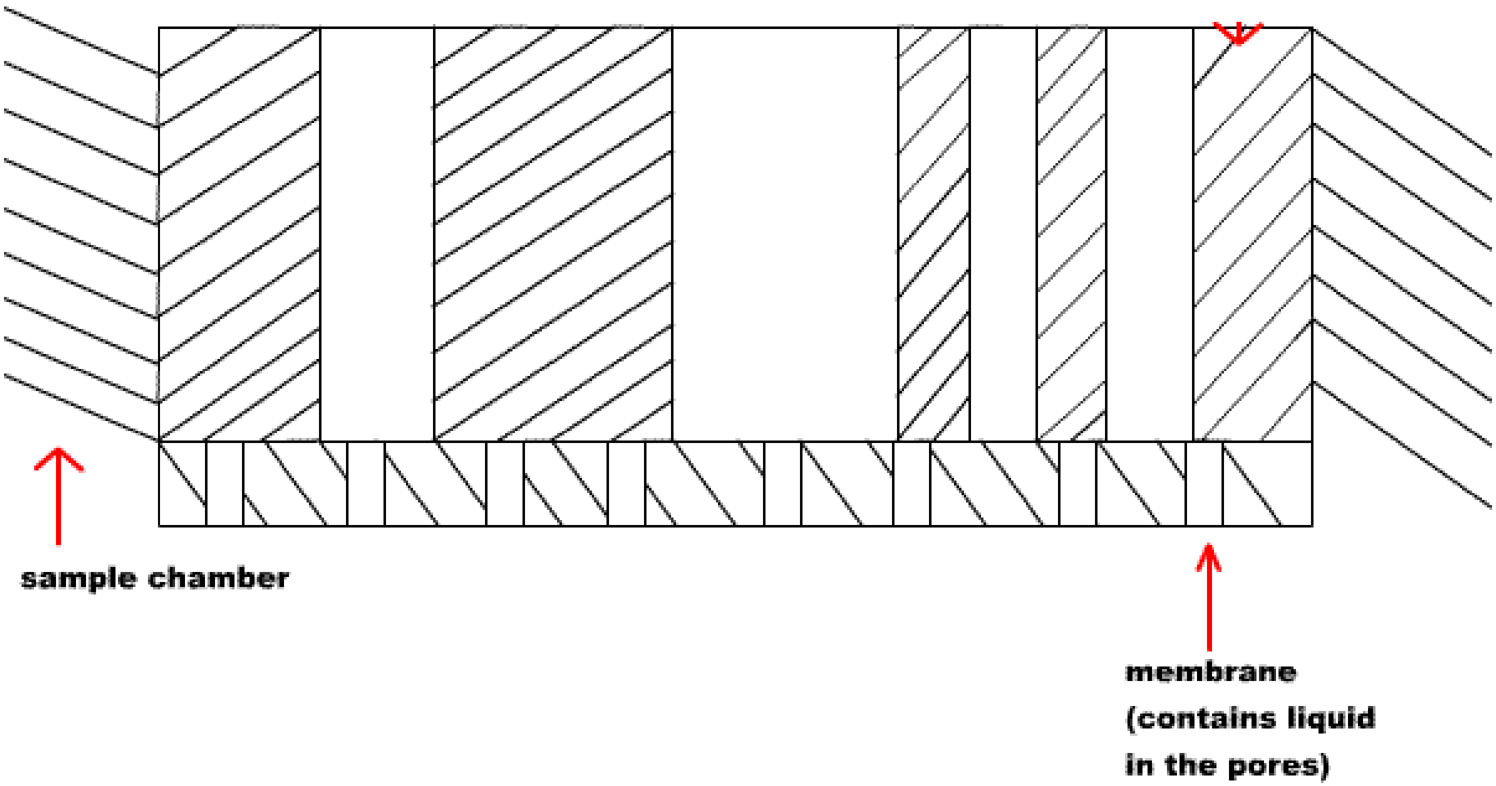
Pressure needed to empty membrane pores

↓

Gas does not pass through liquid filled pores of membrane ← → Extruded liquid flows out through liquid filled pores of membrane

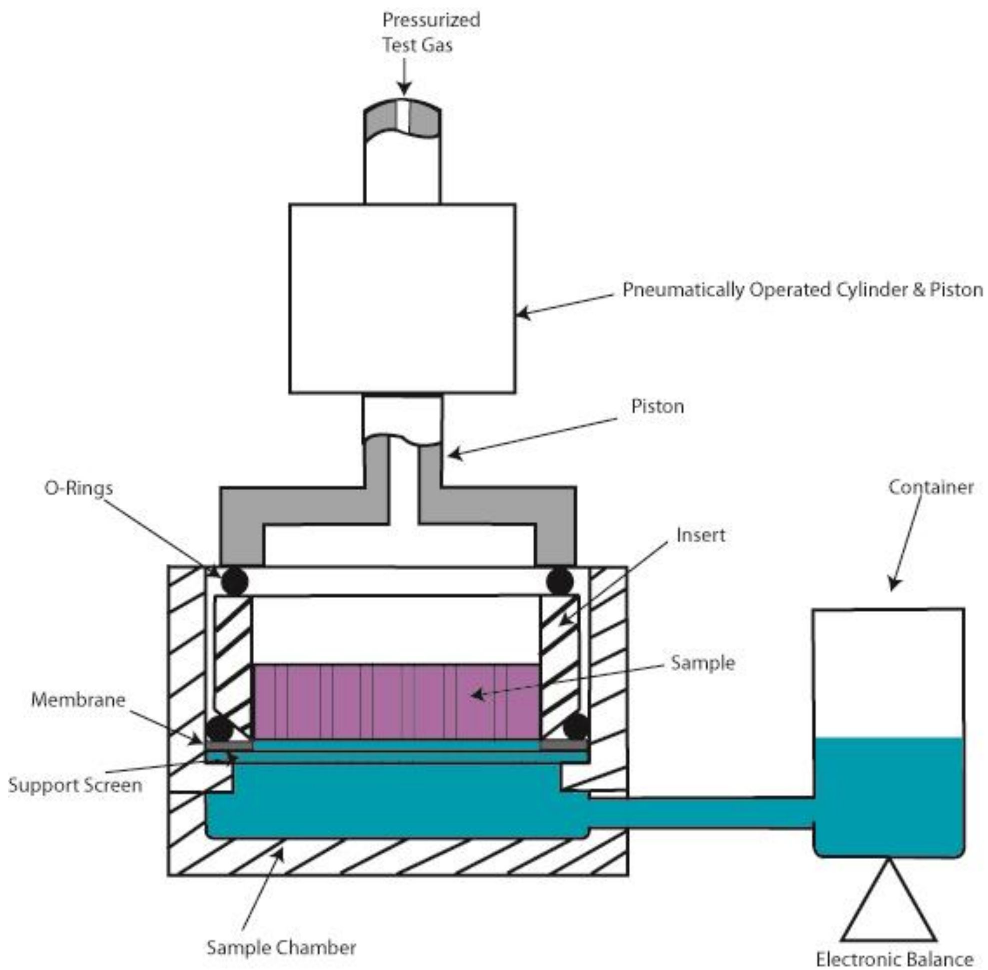


BASIC PRINCIPLES



THE LIQUID EXTRUSION POSIMETER

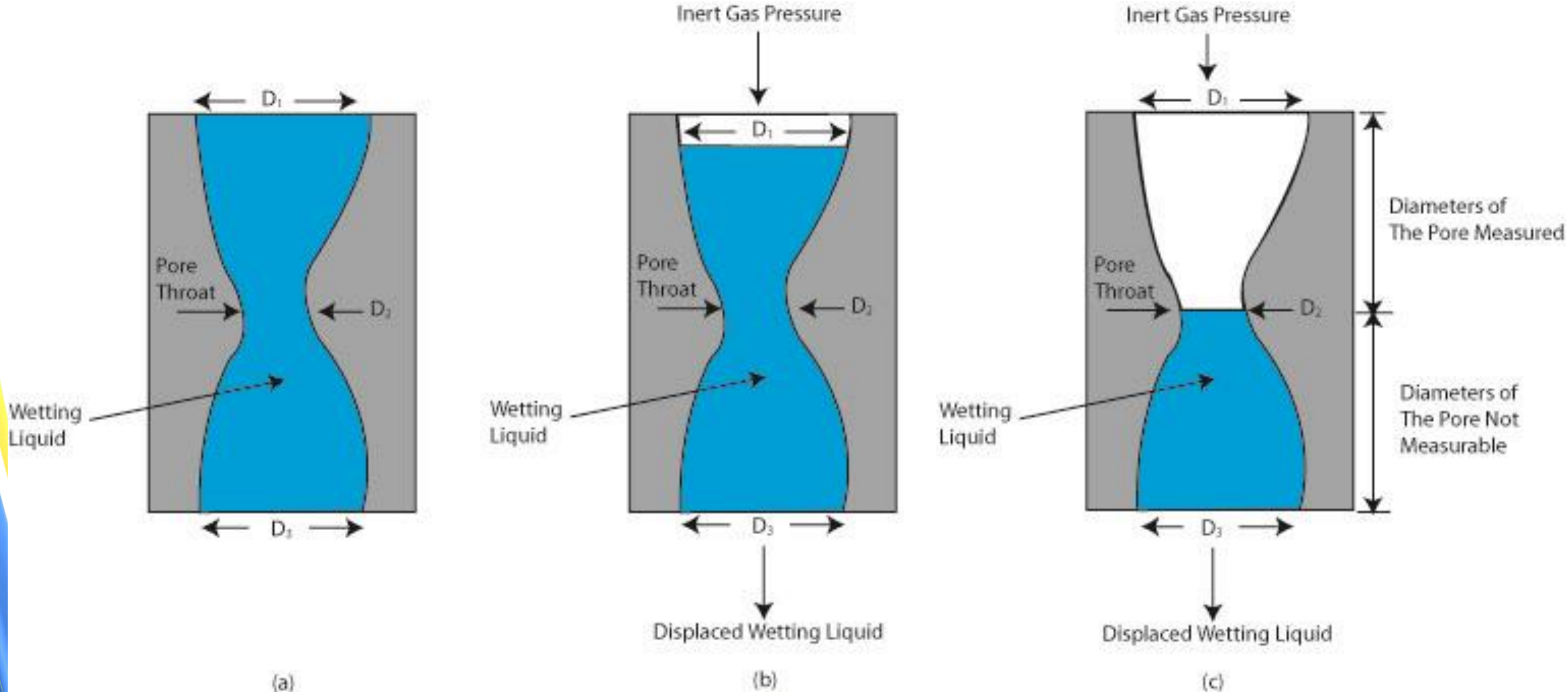
THE INSTRUMENT



THE PMI LIQUID EXTRUSION POSIMETER

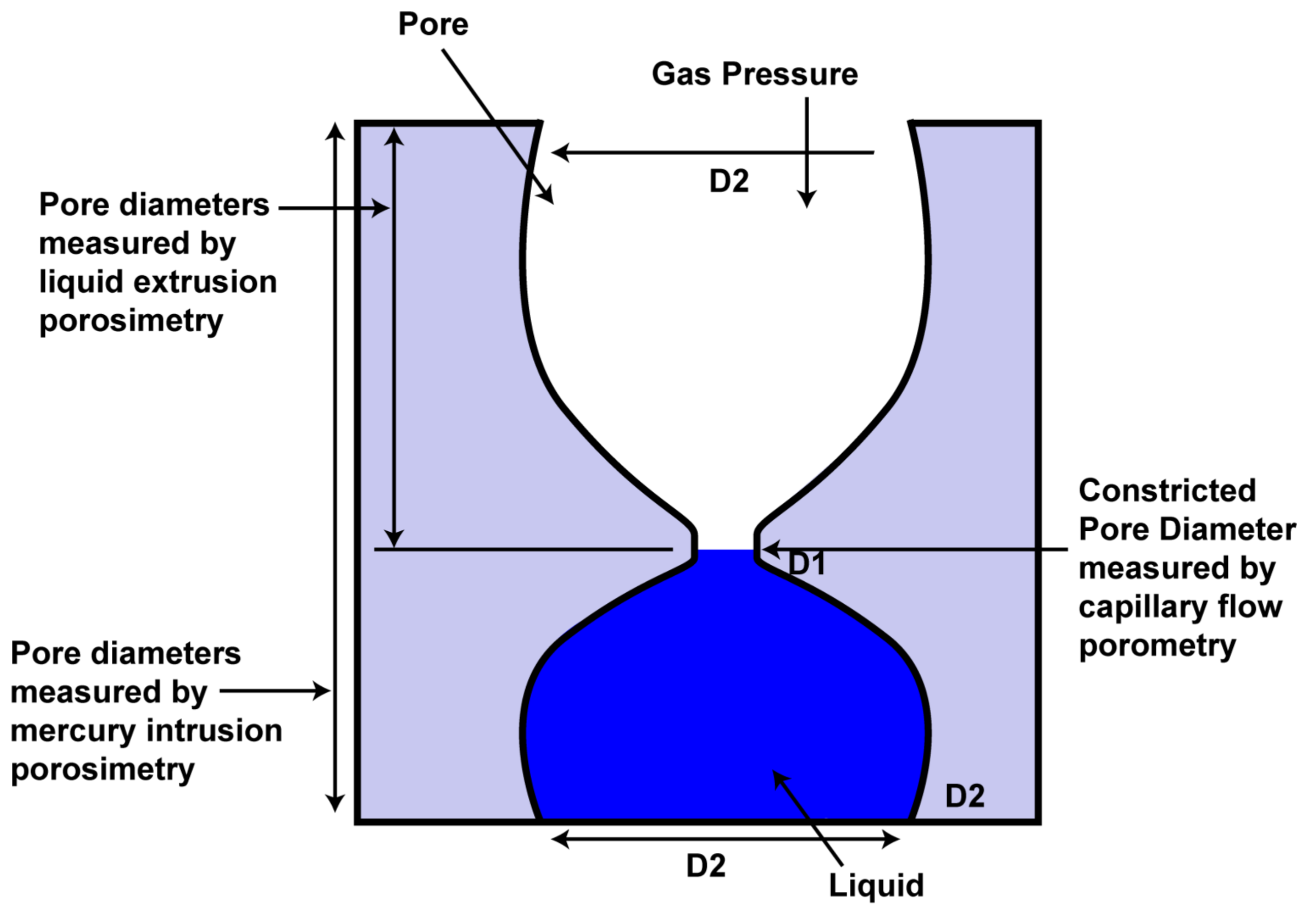
DATA ANALYSIS

Through Pore Diameter ($D = 4\gamma\cos\theta/p$)



All pore diameters from pore entrance to pore throat are measured

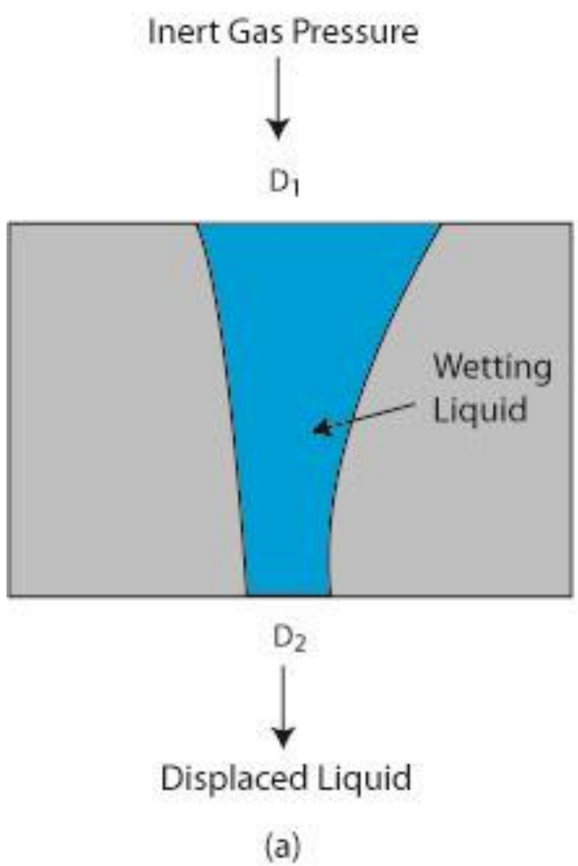
PORE DIAMETERS MEASURABLE BY SOME OTHER TECHNIQUES

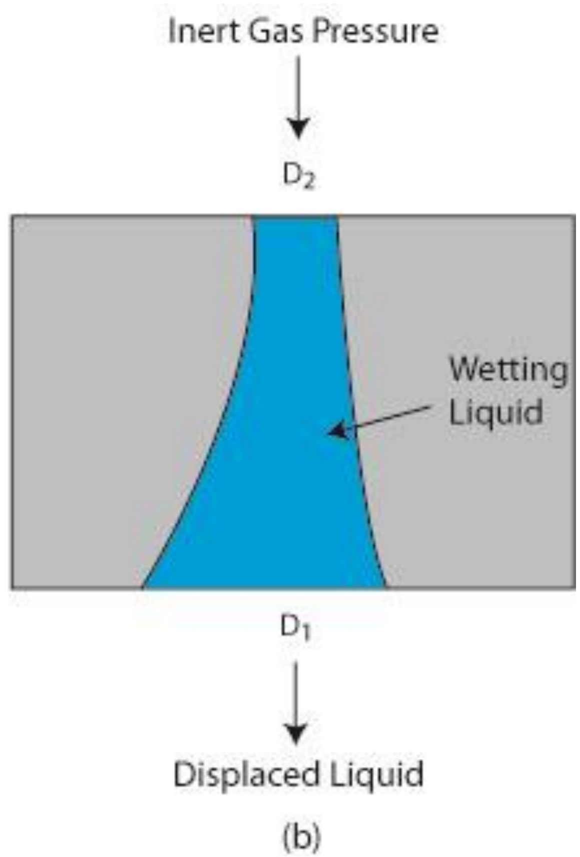


THROUGH PORE DIAMETER

Orientation of Converging and Diverging Pores

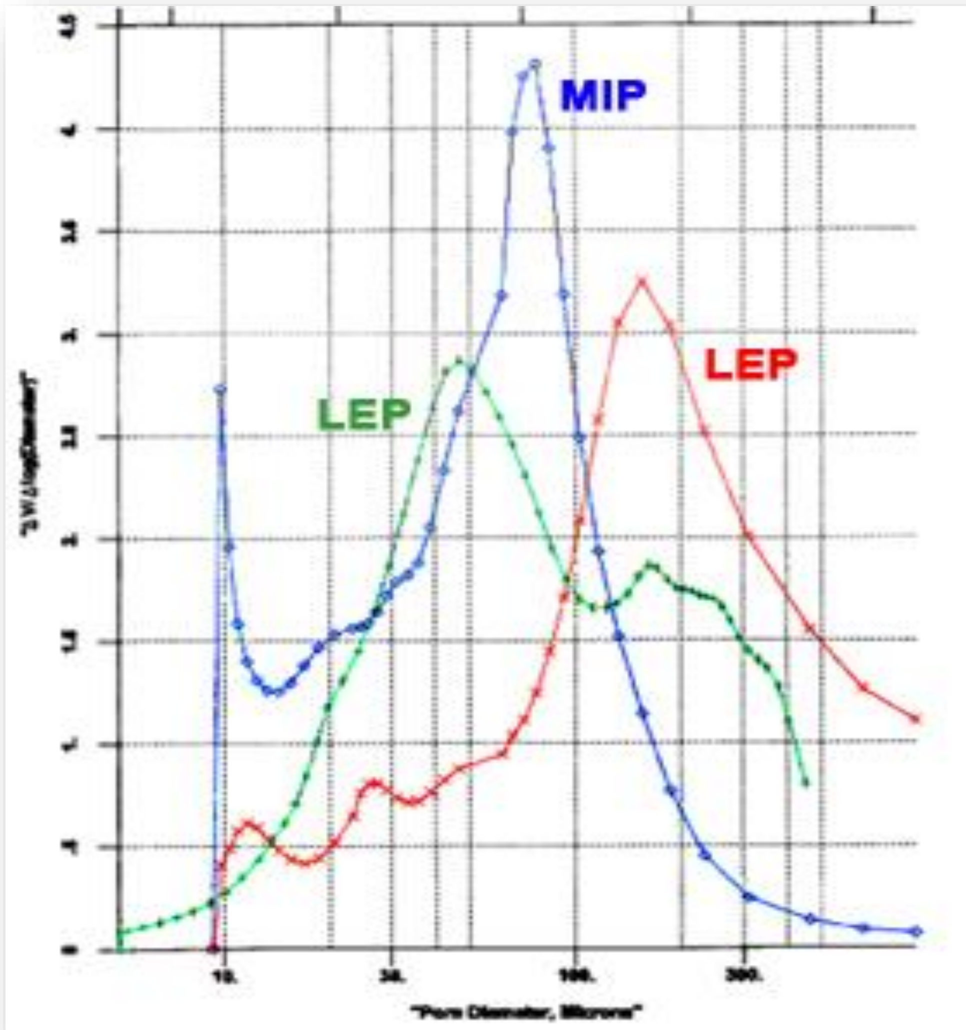
Measured Pore Diameters	D1 to D2	D2





THROUGH PORE DIAMETER

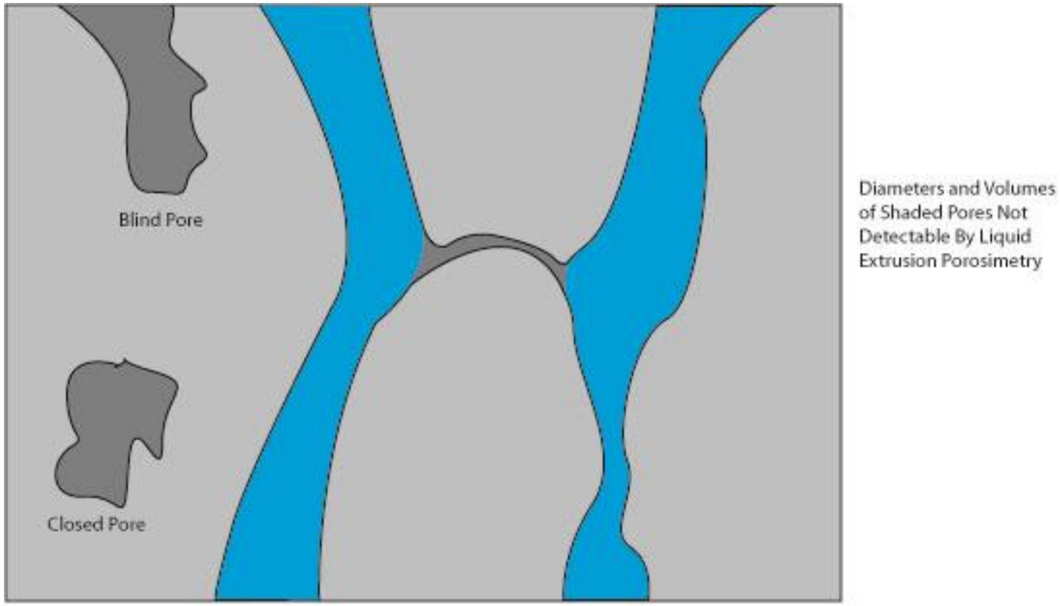
Converging & diverging pores in a textile



THROUGH PORE DIAMETER

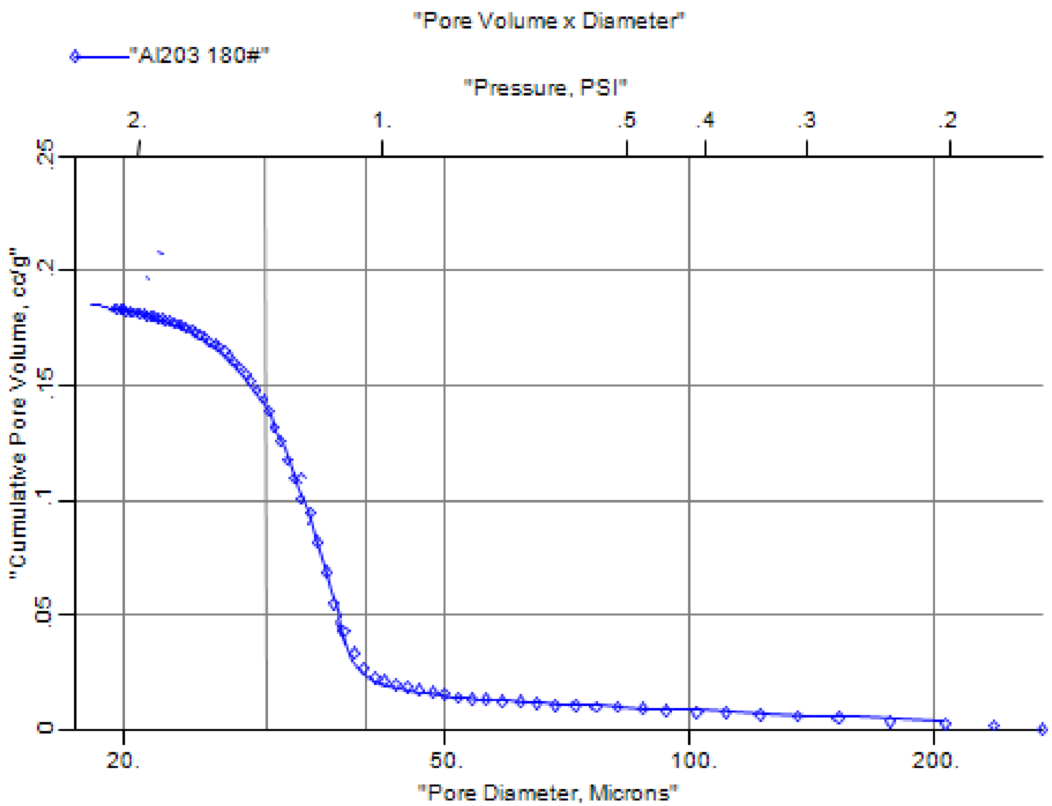
Diameters NOT measurable

- Blind pores
- Closed pores
- Narrow interconnecting pores between wider ones
- Some pores in complex Pore network



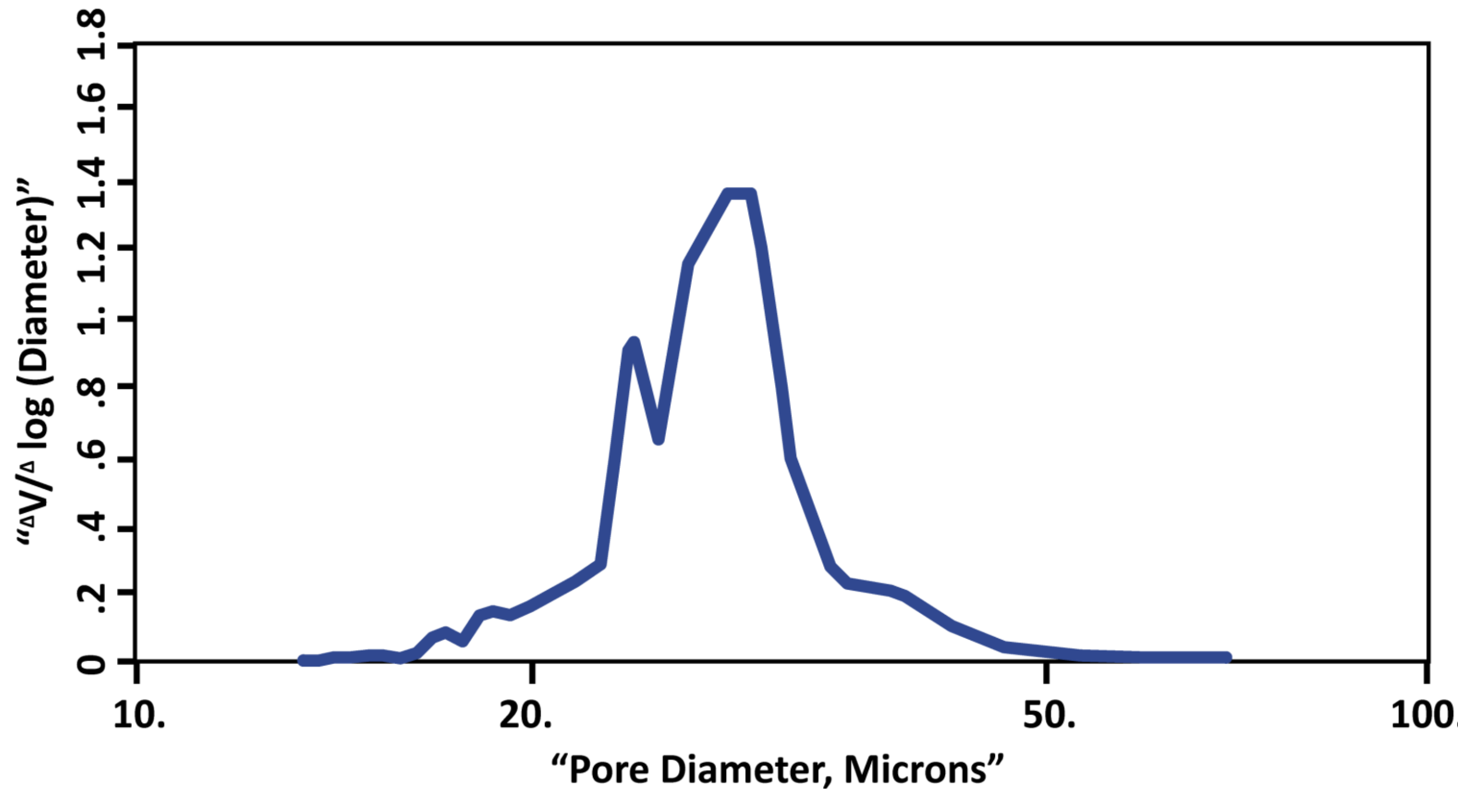
Through Pore Network

THROUGH PORE VOLUME



Volume of blind pores, close pores & some pores in complex pore networks are not measured

THROUGH PORE VOLUME DISTRIBUTION



THROUGH PORE VOLUME DISTRIBUTION

- Volume distribution function, f_v :

fv = - d V /d log D

- Pore volume in any range of pore diameters:

$$\int dV = -\int f(V)\, d(\log D)$$

= Area under the function

THROUGH PORE SURFACE AREA

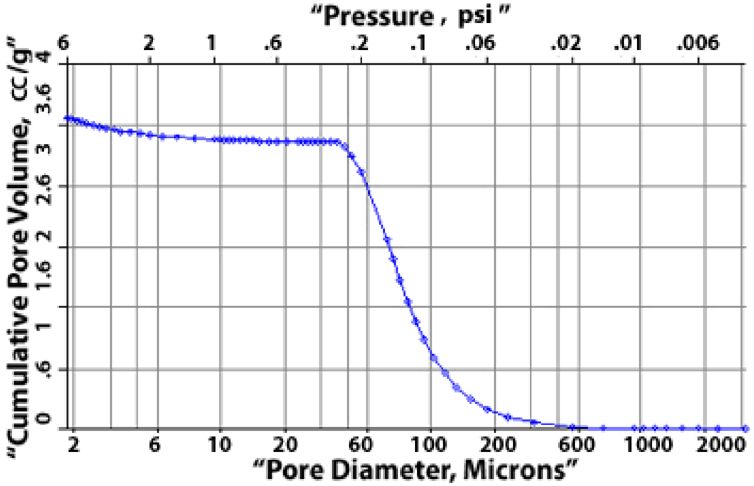
Computed from measured variation of volume with pressure or diameter

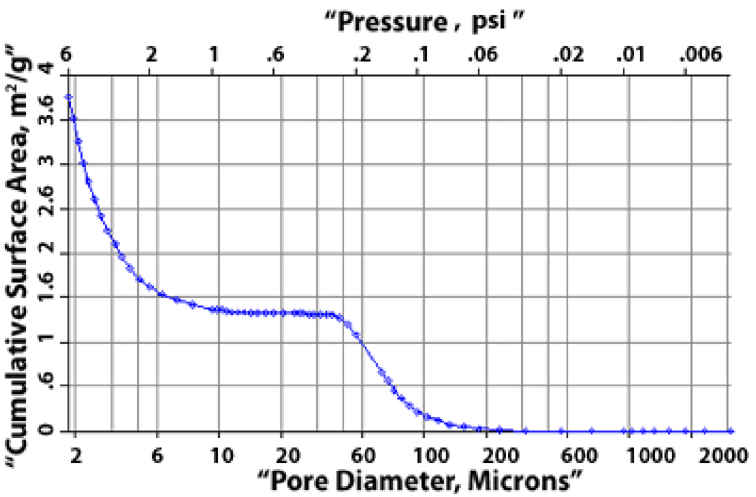
$$dS/dV = 4/D$$

$$S_{s/g} = \int \frac{4}{D} dV$$

$$= \int \left(\frac{p \quad dV}{\gamma_{l/g} \cdot \cos\theta} \right)$$

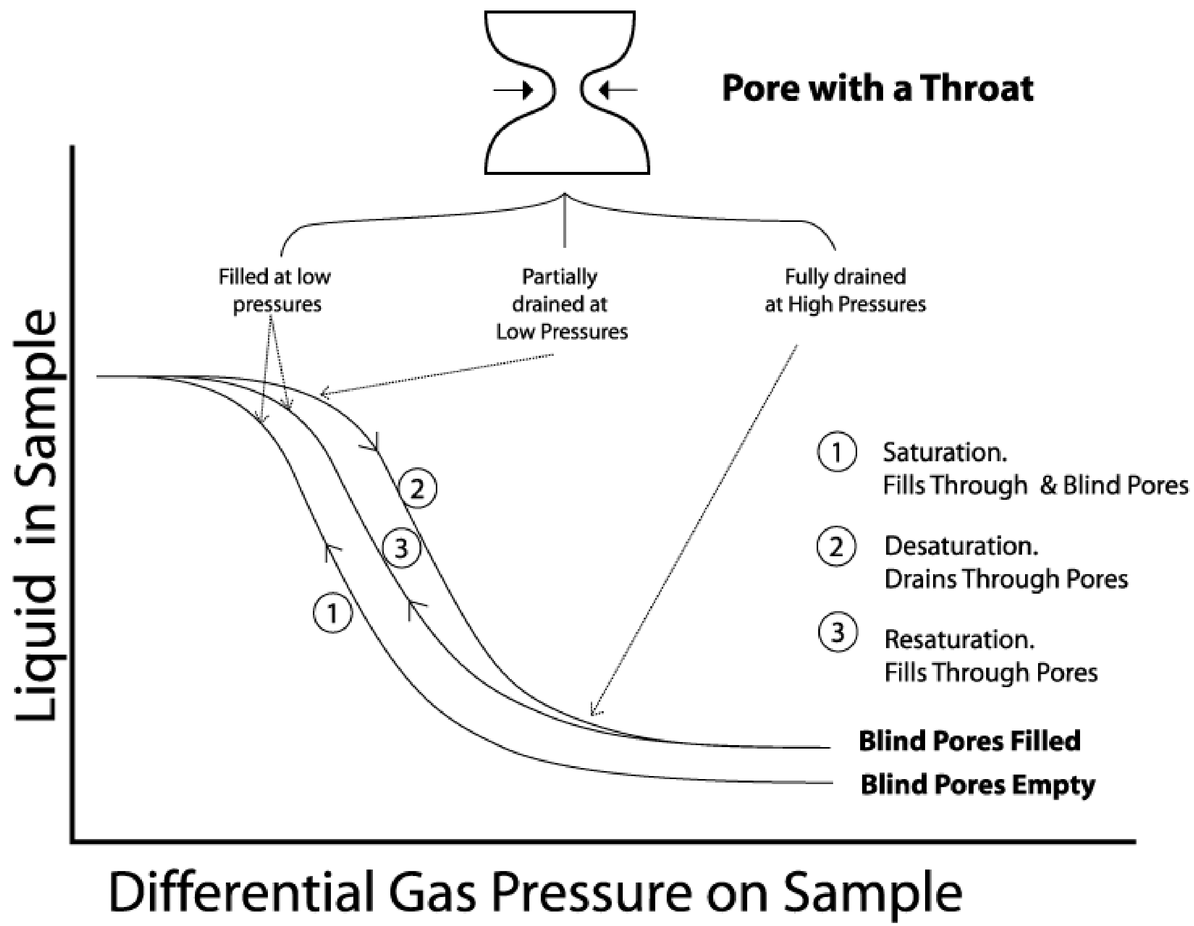
THROUGH PORE SURFACE AREA





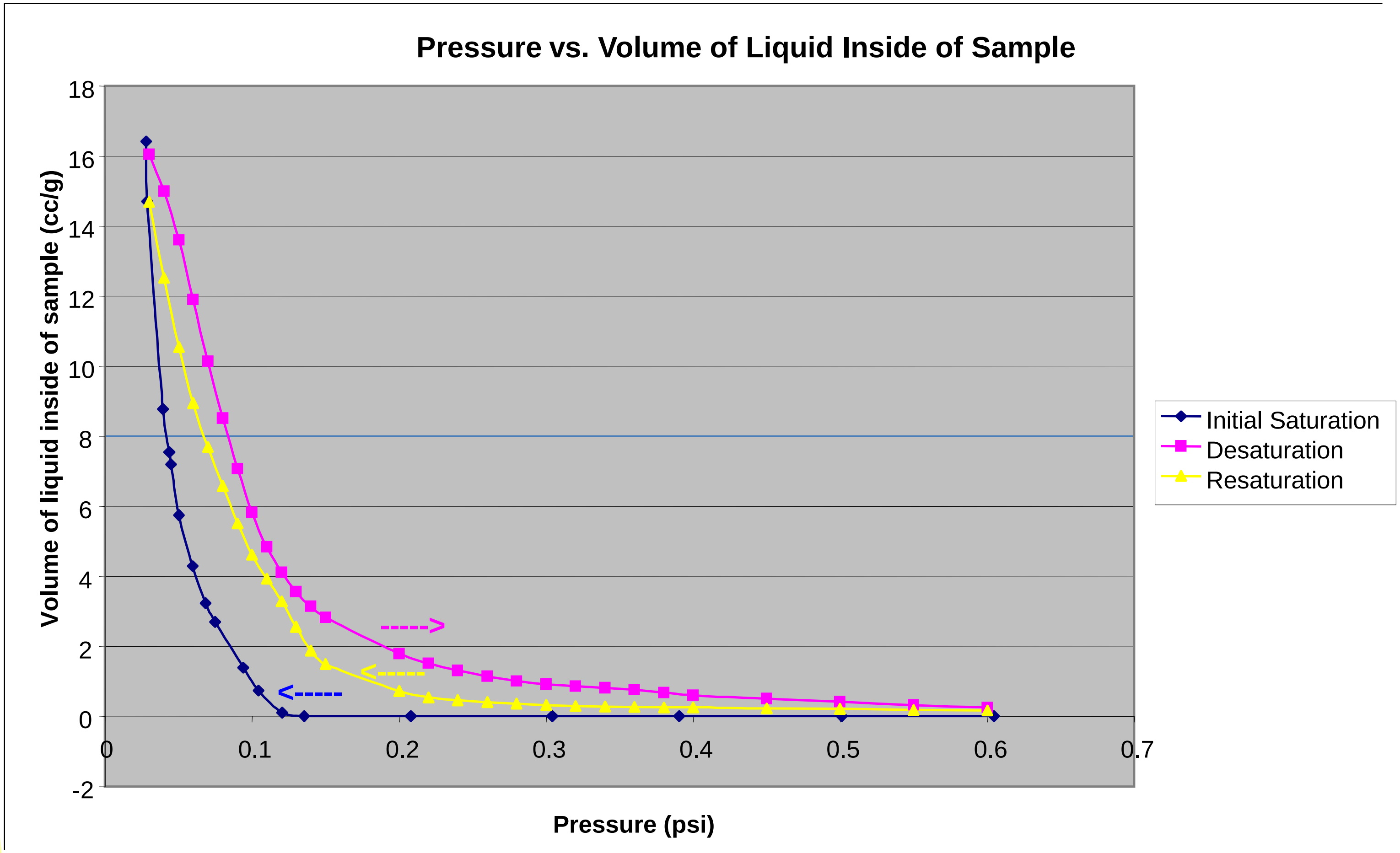
SATURATION – DESATURATION – RESATURATION VOLUME

Expected behavior of sample

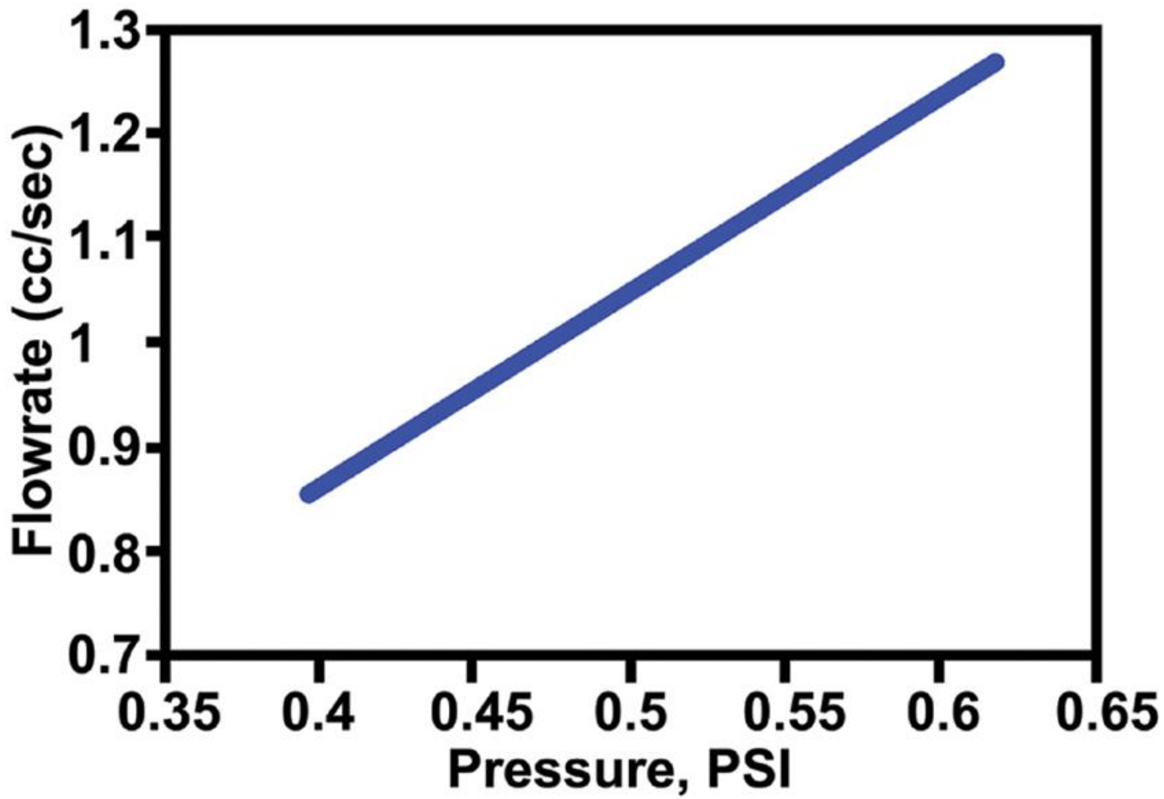


SATURATION – DESATURATION – RESATURATION VOLUME

Saturation & Desaturation Behavior of Fibrous Pad



LIQUID PERMEABILITY



Permeability computed using the Equation

LIQUID PERMEABILITY

$$\text{Darcy's Law:} \quad v = -\frac{k}{\mu} \cdot \frac{dp}{dx}$$

v = linear flow rate
k = ***permeability***
μ = viscosity of the fluid
p = pressure
x = displacement in the direction of flow

In terms of volume flow rate:

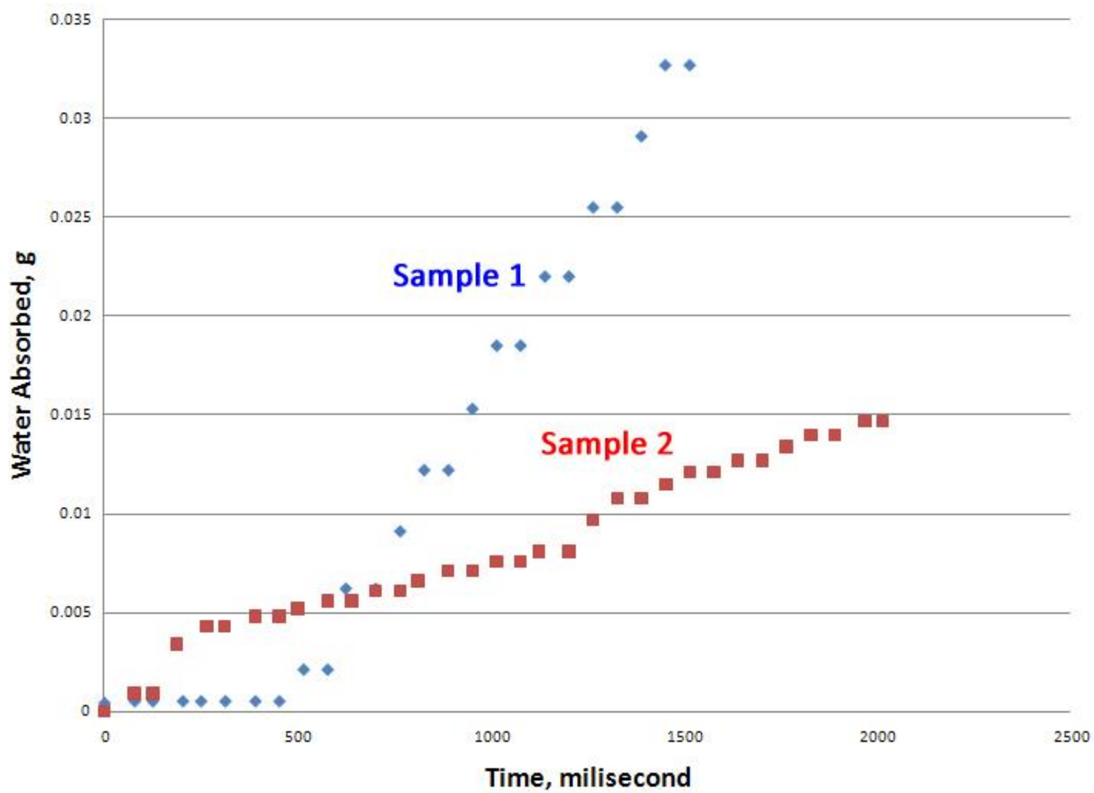
$$\underline{F} = k \cdot \left(\frac{A}{\mu \cdot \ell} \right) \cdot (P_i - P_o)$$

F = flow rate in volume at the pressure, **p**, and the measurement temperature, T per unit time
l = thickness of the porous material
P_i = inlet pressure
P_o= outlet pressure
A = cross-sectional area of the porous material

LIQUID ABSORPTION

Many Applications

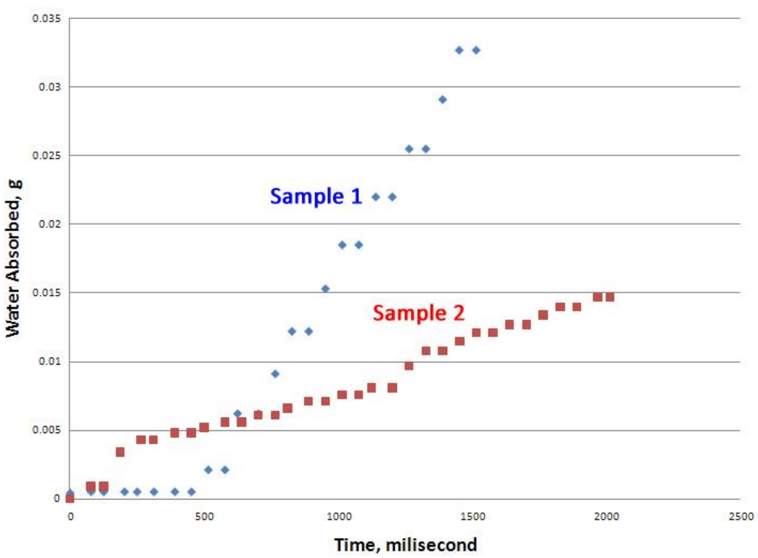
- Absorption of body fluids during surgery
- Household cleaning products
- Personal hygiene products
- Diapers



LIQUID ABSORPTION

Liquid Absorption Test Procedure

- Membrane removed from the sample chamber
- Liquid raised just above the support screen
- Instrument starts recording weight of the liquid in the cup about *20 times a second*
- Sample placed on the support screen
- Absorption rate is computed from the rate of weight change

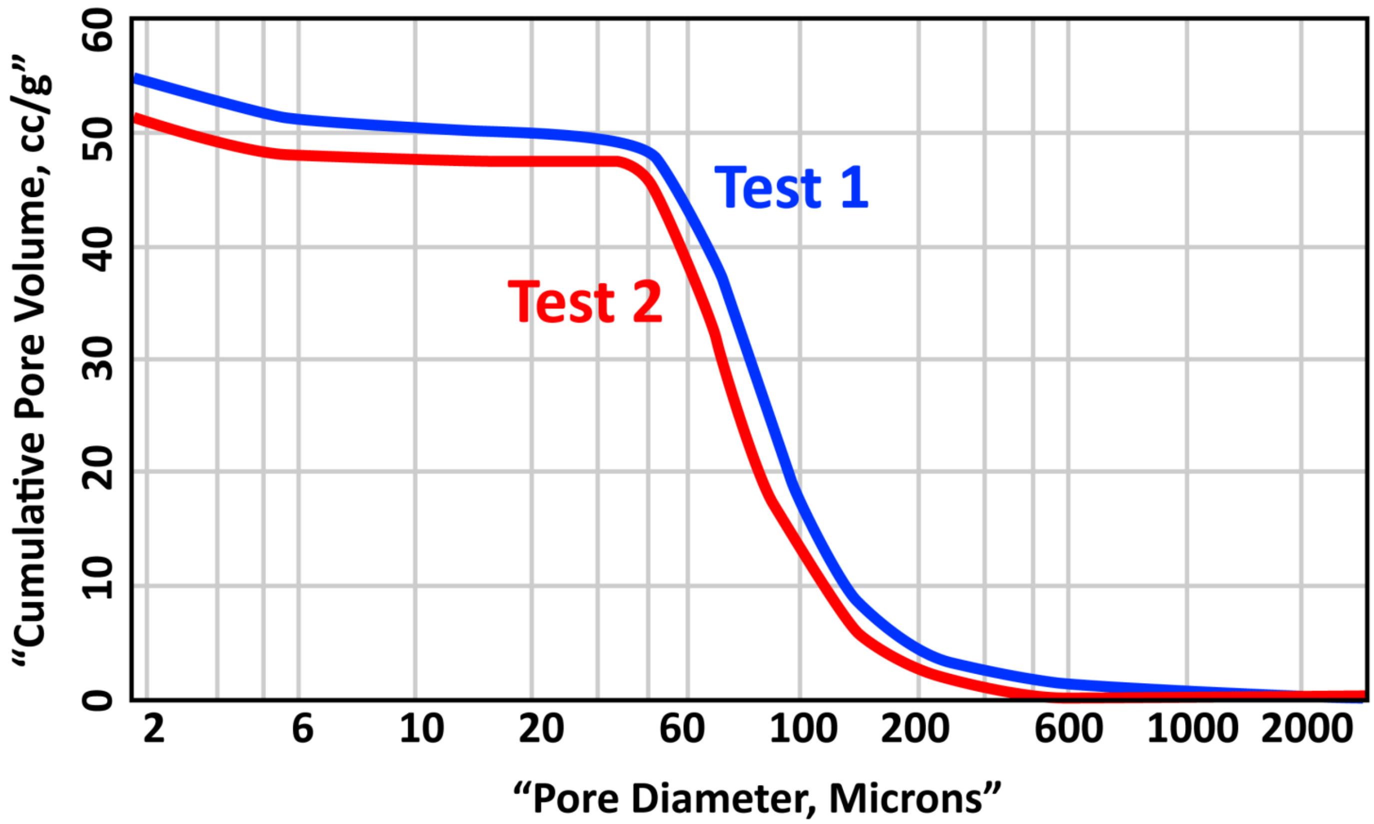


ACCURACY & REPRODUCIBILITY

SAMPLE	Weight of Wet Sample, g		Weight of Wetting Liquid, g	
	Before Test	After Test	Extruded from Sample	In Balance
Foam	5.4809	2.5172	2.9637	3.0922
Laser Sintered Metal	2.6254	2.1807	0.4447	0.4447
Sintered Metal	2.4490	1.9807	0.4683	0.4760

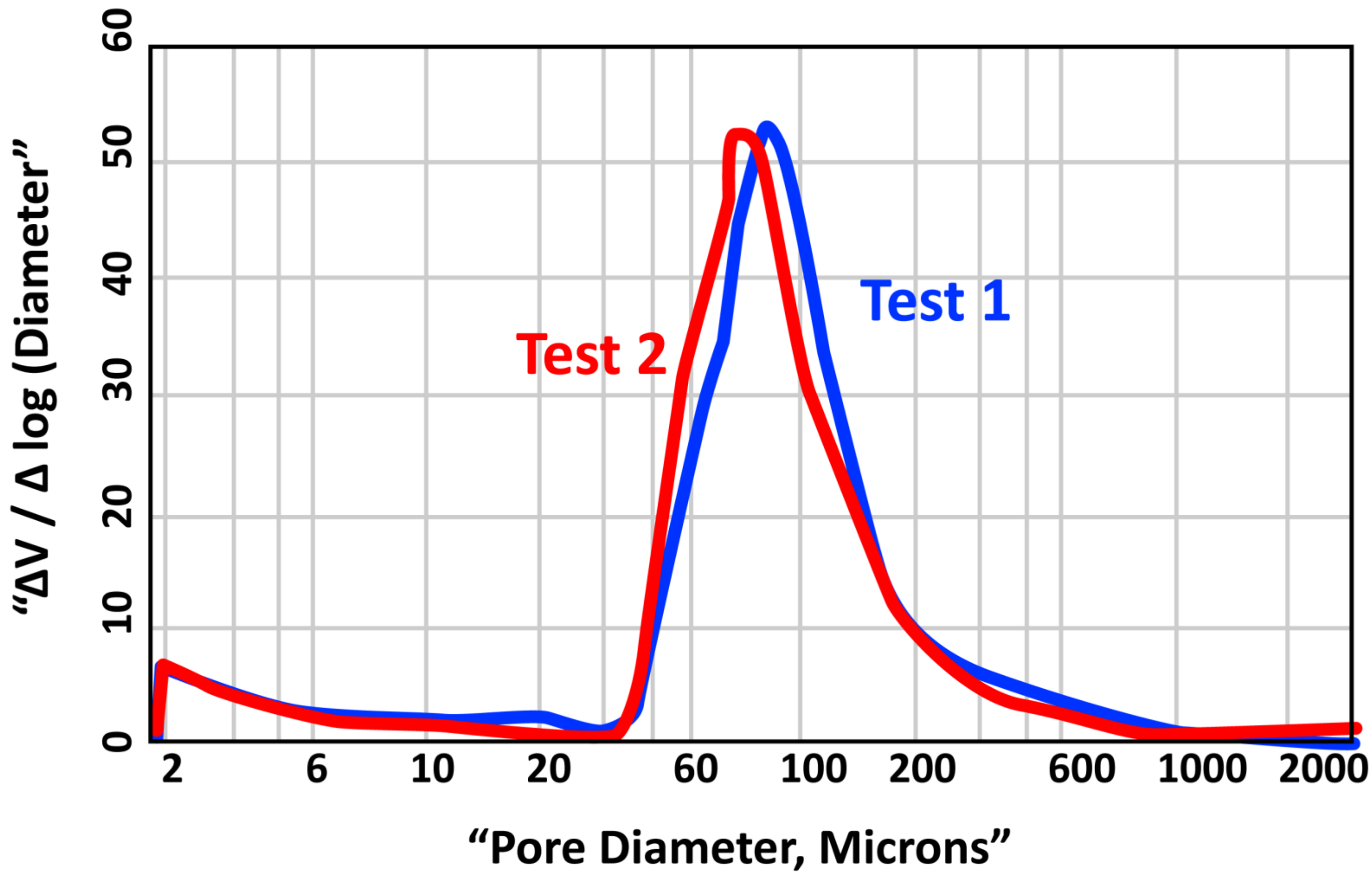
ACCURACY & REPRODUCIBILITY

REPEAT TESTS



ACCURACY & REPRODUCIBILITY

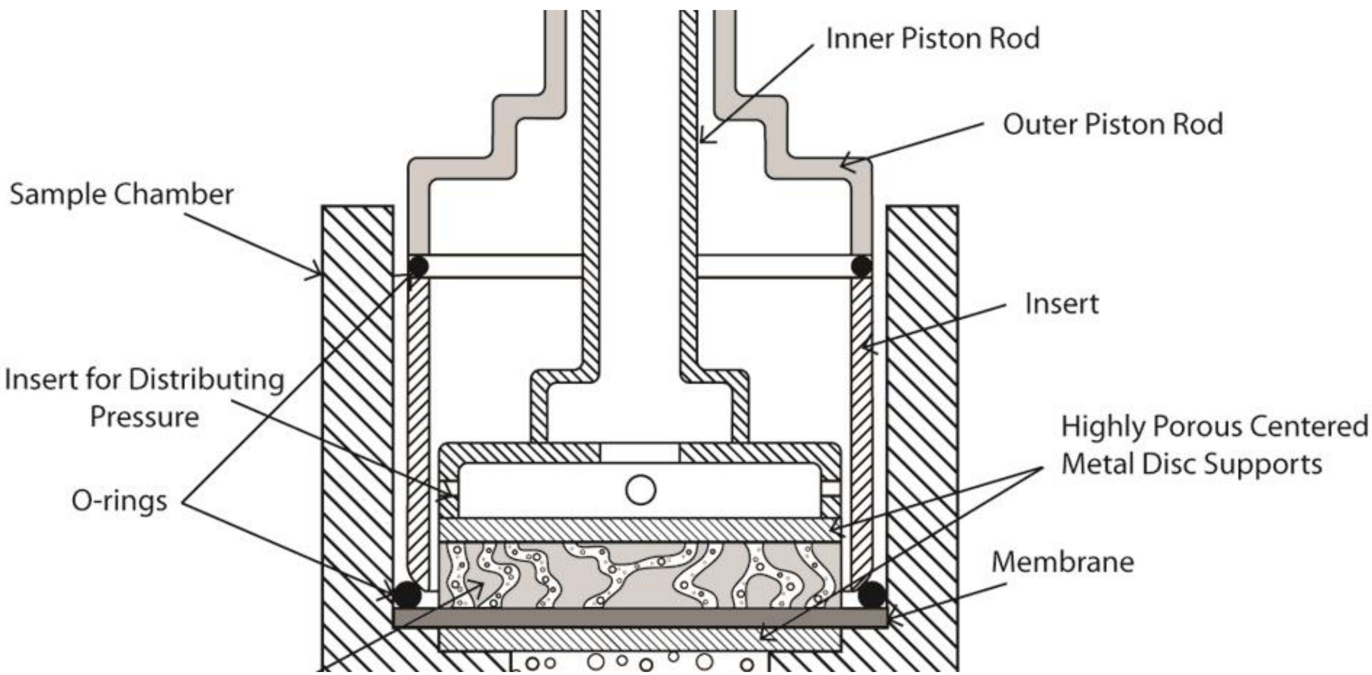
REPEAT TESTS



TESTS UNDER APPLICATION ENVIRONMENTS

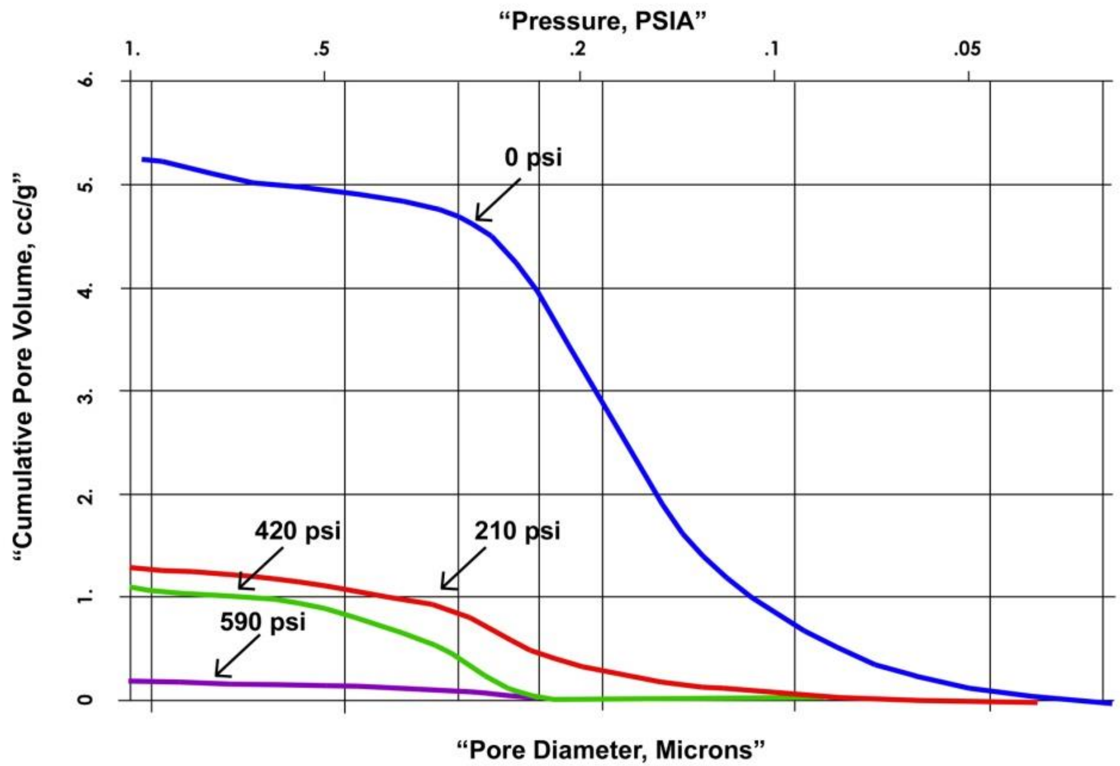
SAMPLE UNDER COMPRESSIVE STRESS

- One piston for compression of sample
- Second piston for compression of O-rings



TESTS UNDER APPLICATION ENVIRONMENTS

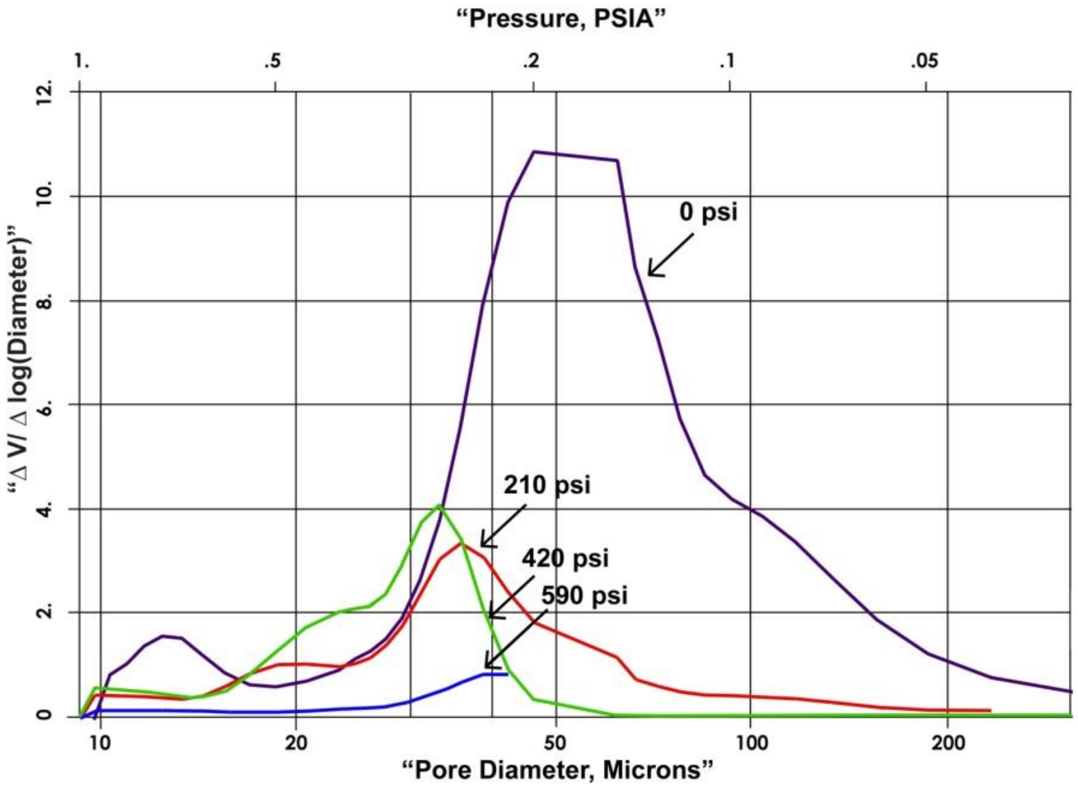
SAMPLE UNDER COMPRESSIVE STRESS



Decreasing influence of compressive stress on pore volume

TESTS UNDER APPLICATION ENVIRONMENTS

SAMPLE UNDER COMPRESSIVE STRESS



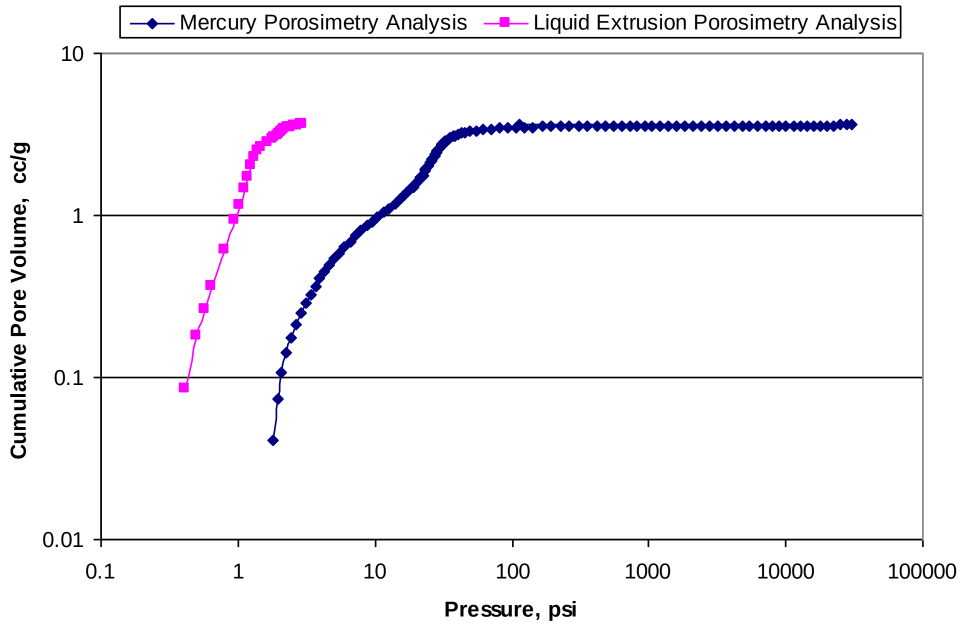
Effects of compressive stress on pore volume distribution of a felt

COMPARISON WITH MERCURY INTRUSION POROSIMETRY

Nano fiber Mats: Compressible, Fibrous, contains primarily through pores

Pore Volume:

- Mercury intrusion: Measures through & blind pores
- Liquid Extrusion: Measures through pores
- Pore volume identical



TESTS UNDER APPLICATION ENVIRONMENTS

CHEMICAL & THERMAL ENVIRONMENT

- A variety of test liquids:
  - Oils
  - Water
  - Sugar solutions
  - Fat
  - Many other application liquids
- Hydrogel testing using water
- Test gas with controlled humidity
- Temperature up to 200˚C

COMPARISON WITH MERCURY INTRUSION POROSIMETRY

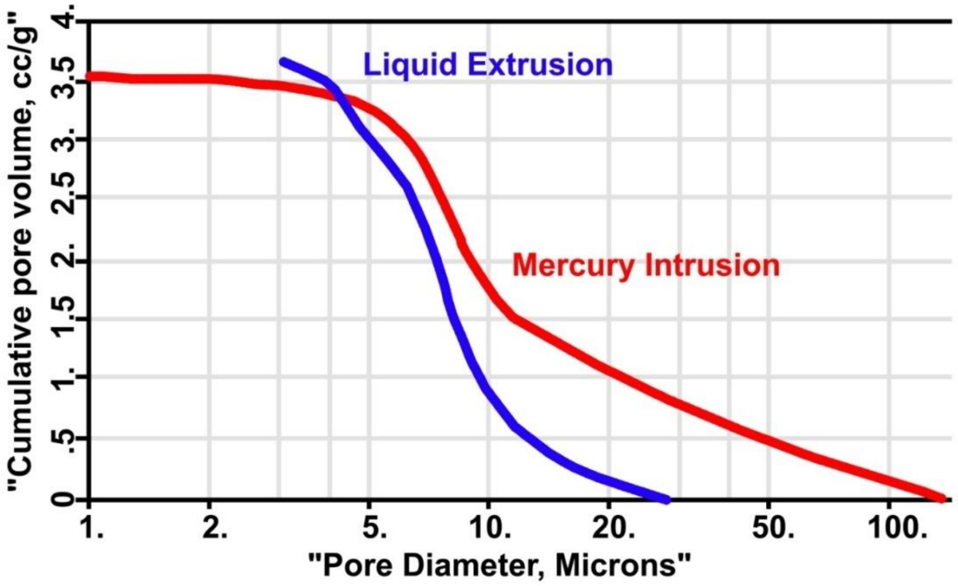
Identical Pore Volume

	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%

Much higher test pressure by Mercury Intrusion

Comparison with Mercury Intrusion Porosimetry

Different Pore Volume Distribution

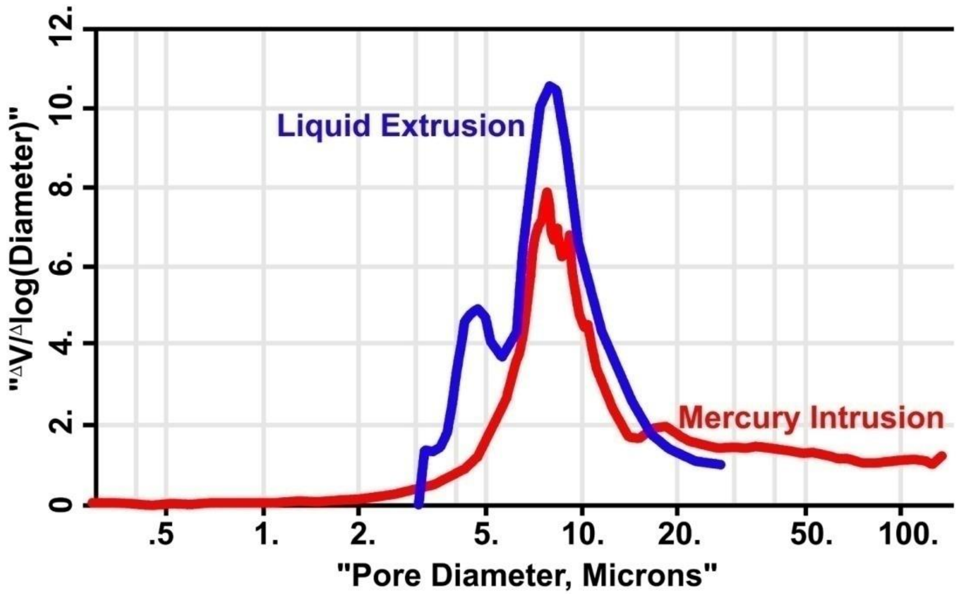


Mercury Intrusion:

Higher volume at large diameters due to compression at high pressure

COMPARISON WITH MERCURY INTRUSION POROSIMETRY

Different Pore Volume Distribution



Liquid Extrusion:

- Higher resolution for small pores
- Two Peaks

COMPARISON WITH MERCURY INTRUSION POROSIMETRY

FUEL CELL COMPONENT:

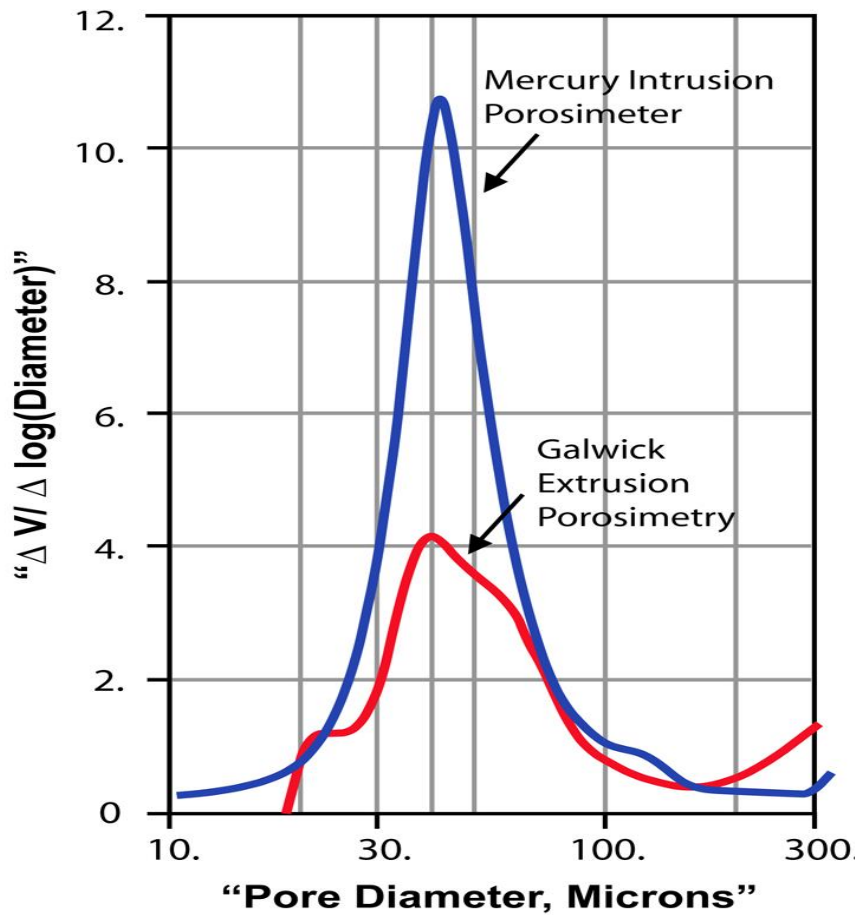
Insensitive to pressure

Contains blind pores

- Identical distribution
- Pore Volume

  LEP: 2.05 cc/g

  MIP: 3.66 cc/g
- 44 % uniformly distributed blind pores



COMPARISON WITH MERCURY INTRUSION POROSIMETRY

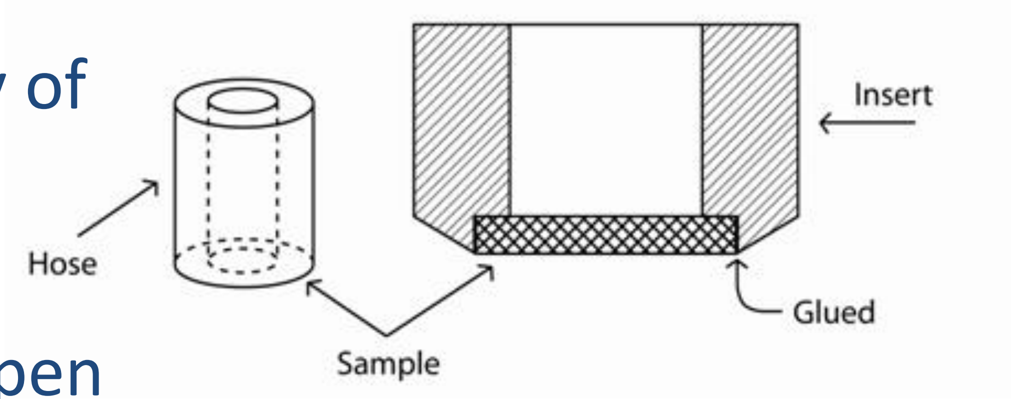
OPERATIONAL CHARACTERISTICS

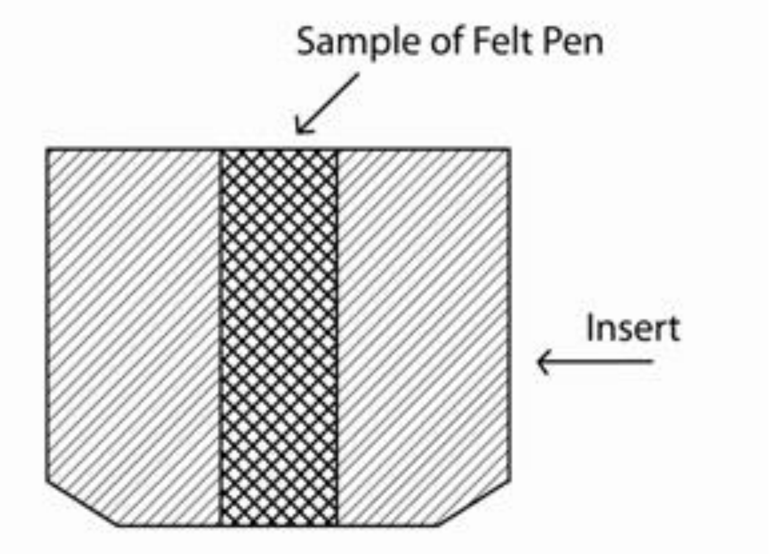
Liquid Extrusion Porosimetry	Mercury Intrusion Porosimetry
1. No toxic substance used	1. Mercury used
2. Low test pressure Negligible structural distortion	2. An order of magnitude higher test pressure Appreciable structural distortion
3. Sample reusable	3. Sample discarded
4. Liquid permeability measurable	4. Permeability NOT measurable
5. Only through pores measurable	5. Through & Blind pores measurable
6. Samples with pore diameter 2000 μm measurable	6. Samples with pores greater than 200 μm difficult to measure

LIQUID EXTRUSION POROSIMETRY

WIDE APPLICABILITY

- Liquid permeability of hose
- Pore volume distribution of felt pen





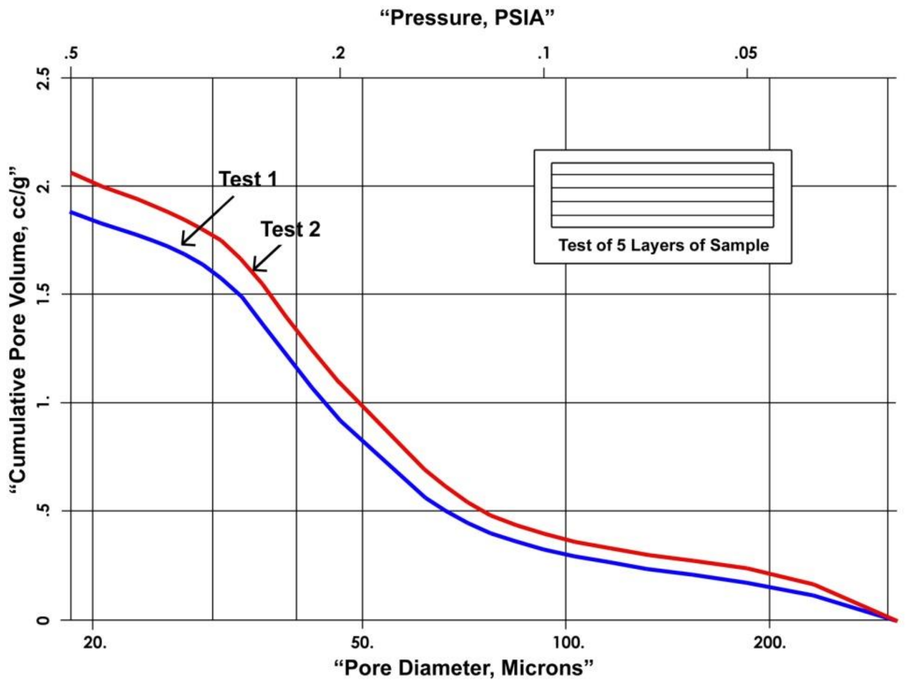
LIQUID EXTRUSION POROSIMETRY

WIDE APPLICABILITY

- Non-rigid pressure sensitive materials like felts, hydrogels, sponges and foams
- Pre-filter materials whose pore volume is important for dirt-holding capacity
- Geotextiles, coated fabrics, sponges and foams containing large pores
- Patches, bandages and implants for healthcare applications & drug holding capacity
- Rocks and soil for oil and water holding capacity
- Nonwovens, fuel cell components, and textiles for liquid and chemical holding capacity

LIQUID EXTRUSION POROSIMETRY

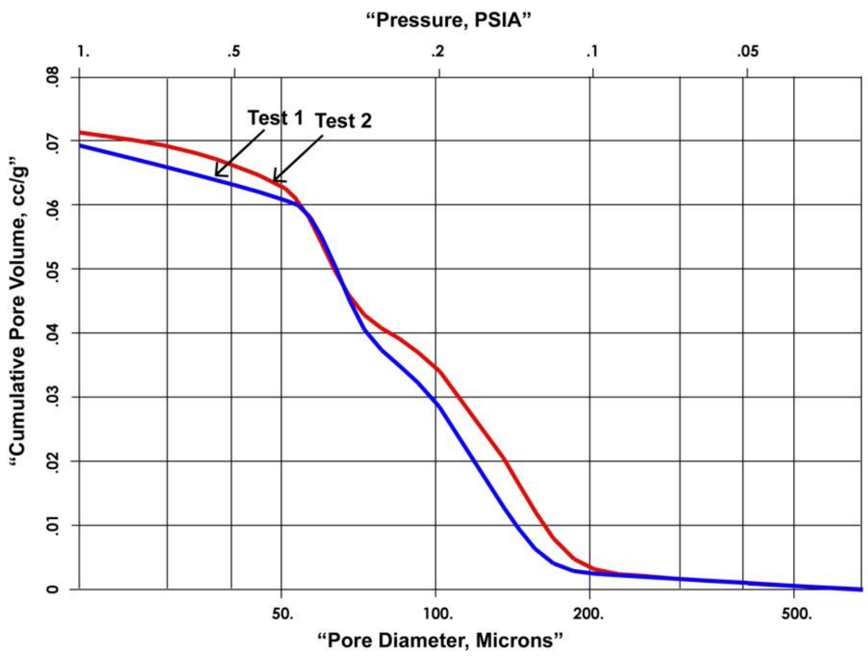
WIDE APPLICABILITY



Pore volume of thin fuel cell component using 5 layers

LIQUID EXTRUSION POROSIMETRY

WIDE APPLICABILITY



Pore volume of large pores in sintered metal implant using mineral oil

SUMMARY & CONCLUSIONS

- We have considered the basic principles of wetting liquid extrusion porosimetry.
- We have analyzed the technique of liquid extrusion porosimetry.
- We have discussed the measurement and of pore structure characteristics and their significance.
  - Through pore volume,
  - Pore diameter,
  - Volume distribution,
  - Surface area,
  - Liquid permeability.

SUMMARY & CONCLUSIONS

We have compared the techniques, LEP & MIP, highlighting the differences in their operational features.

Thank You!

PMI

POROSITY & PERMEABILITY TESTING