Characterization of solid and powders

The measurement of specific surface area and pore size distribution allows the study of the chemical-physical characteristics of porous substances.
SINTEF Materials and Chemistry has a range of instrumentation and techniques that allow determination of these parameters.

- Density
- Envelope Density
- Specific Surface Area
- Pore Volume and Pore Size Distribution
- Porosimetry

Fisions Pascal 140 and 240 Mercury Porosimetetry

Density

Solid density is measured by a Helium Pycnometer, Micromeritics AccuPyc 1330 He-pycnometer.

Envelope Density

Envelope density is the mass of an object divided by its total volume, including the volume of its pores and small cavities. The envelope volume of solid samples is measured by a powder pycnometer, Micromeritics GeoPyc 1360. When the absolute density of the solid material is provided, the pore volume and the porosity of the sample can be calculated.

Specific surface area

The standard method for measuring specific surface area is based on the physical adsorption of nitrogen on the solid surface, using the BET method **[S. Brunauer, H.P. Emmet and E.Teller:**Adsorption of gasses in Multimolecular Layers. J.Am. Chem.Soc 60, 309 (1938)]. The adsorbed amount of nitrogen can be measured either volumetrically or gravimetrically.

The gravimetric method is used to measure surface areas, in the range of 1 –30 m^2/g, while the volumetric method is used to measure surface areas greater than 30 m^2/g.

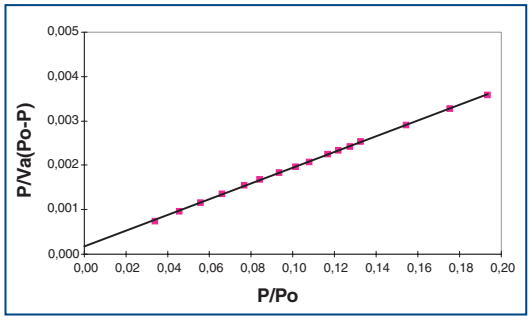

Figure right:
A BET isoterm obtained from nitrogen adsorption to a slate sample. From this plot the surface area is calculated – (16.4 m^2/g).

Equipment	Method
Sartorius S3D-V Supermicrobalance	Gravimetric adsorption measurements
Carlo Erba Sorptomatic Model 1900 C.Erba Sorptomatic MS 190 Multisampler	Volumetric adsorption measurements and nitrogen desorption
C. Erba Porosimeter Macropore Unit 120 C. Erba Porosimeter Mod. 2000	Mercury Porosimetry
Fisions Pascal 140 Low Pressure Porosim. Fisions Pascal 240 High Pressure Porosim.	Mercury Porosimetry
Micromeritics AccuPyc 1330	Solid density
Micromeritics GeoPyc 1360	Envelope density

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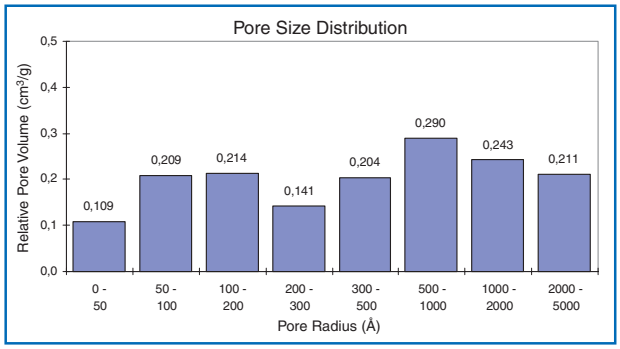
Pore Size Distribution

The most important methods to determine pore volume and pore size distribution are the mercury penetration method and the nitrogen adsorption / desorption method. Pores with pore radius in the range of 50 Å – 55 µm are measured by mercury intrusion, while the nitrogen desorption isoterm is used for calculation of pores with pore radius in the range of 10 –300 Å.

Mercury porosimetry

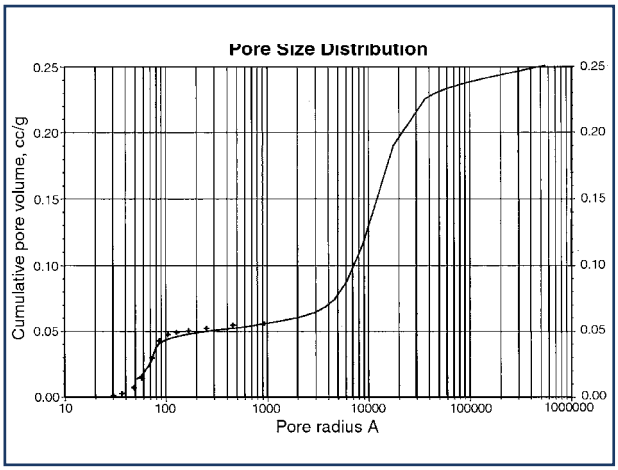
Mercury porosimetry is based on the fact that mercury has a high surface tension and that it is a strongly non-wetting liquid on most substrates. The pore volume and the pore size distribution are measured by determining the amount of mercury forced into the pores as a function of the pressure.



Pore size distribution of a porous polymer particle obtained from mercury intrusion measurements.

Nitrogen desorption

At the starting point for nitrogen desorption the pores are completely filled with liquid nitrogen. Gradually lowering of the pressure will result in desorption of measurable quantities of nitrogen. From these data, the pore volume and the pore size distribution are obtained.



Pore size distribution of a catalyst. Data from nitrogen desorption measurements (+) are combined with mercury intrusion data (solid-drawn curve). The figure illustrates the good agreement between the two methods in the overlapping region.

Applications

Polymer particles	Pharmaceuticals	Soils, minerals
Catalysts	Building materials	Ceramics
Active carbons	Cements	Drill cores
Charcoals	Pigments	Glass

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