Surface and Pore Characterization Laboratory (YGL)

Surface and pore characterizations of materials are important in research and industrial applications. The YGL of R&D Training Center has instruments like Surface Characterization Unit, Mercury Porosimeter, Helium Pycnometer and Contact Angle - Surface Tension Measurement Instrument to serve this purpose.

Surface Characterization Unit : In order to compensate for the imbalance of electronic forces of the atoms on their surfaces, solids (adsorbents) try to attract the gas or liquid (adsorbate) atoms around. This causes the occurrence of a process called adsorption.

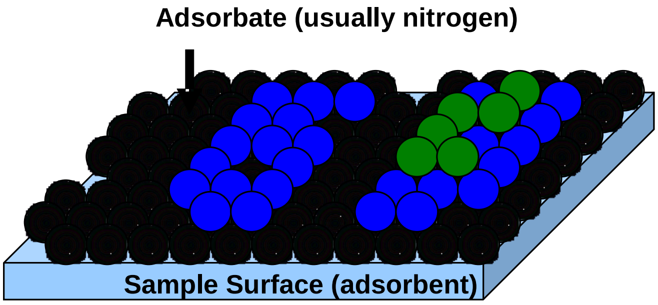


Figure-1. Adsorption Process

The samples are taken to degasser unit and heated under vacuum (up to 350°C) for purification and dehydration prior to experiments. After this process, the samples are subjected to analysis with nitrogen gas at the temperature of liquid nitrogen. Adsorption isotherm, which shows how much nitrogen is adsorbed on the material as a function of pressure, is obtained in the end of these tests. (Figure-2).

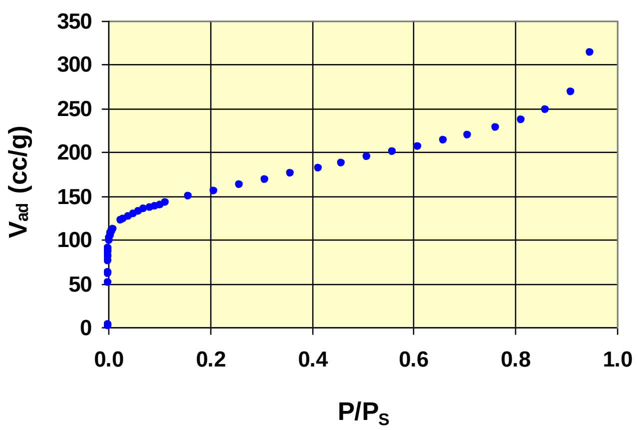


Figure-2. Sample Adsorption Isotherm

It is possible to determine these properties of solids once the adsorption isotherm is obtained:

- BET Surface Area (Single or Multi-point)
- Micropore size Distribution (0.5 nm – 2 nm)
- Mesopore Size Distribution (2 nm – 50 nm)
- Total Pore Volume
- Mean Pore Diameter

Although the amount of the sample required varies according to the properties of the material, usually 1 g of sample is considered to be sufficient for analysis.

Mercury Porosimeter : The operation of mercury porosimeters depends on the physical principle that a non-reactive, non-wetting liquid will not penetrate fine pores until sufficient pressure is applied to force its entry.

The porosimeter has two sample holders, namely low pressure (until 50 psi) and high pressure (until 60,000 psi. Only solid materials are taken into analysis in the instrument, and it is possible to measure the macro-meso pore sizes of samples between 200-0.0036 microns.

After the determination of the amount of mercury entering into the sample in the end of the experiment, these properties of the material can be obtained (Figure-3):

- Porosity,
- Pore volume distribution,
- Pore size distribution,
- Bulk density and apparent density

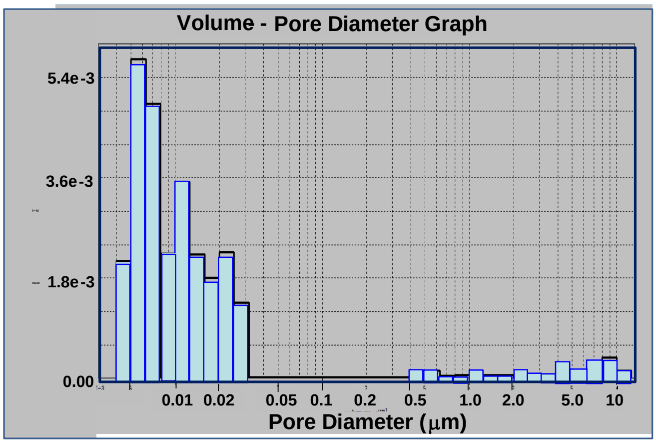


Figure-3. Sample Pore Size Distribution

Samples to be anlayzed should be in form of chunks and pellets. Materials in powder form should be supplied to the laboratory as pellets. The sample to be supplied for measurements should be enough to fill a 1 cm long and 0.7 cm diameter cell.

Helium Pycnometer : Helium pycnometry employs Archimedes' principle of fluid displacement and Boyle's Law to determine the volume and true density.
The displaced fluid should be an inert gas that can penetrate all but the finest pores, thereby assuring maximum accuracy. or this reason, helium is recommended due to its small atomic dimensions.
Helyum'un ideal gaz olarak davranışları da tercih nedenidir. Its behavior as an ideal gas is also desirable. Other gases such as nitrogen can be used, often with no measurable difference. Helium pycnometer can be used on solids for this purpose:

- Measurements of true volume and true density
- Determination of percentage of solids in a slurry

Optical Contact Angle and Surface Tension Measurement apparatus (Goniometer): This instrument is used in order to investigate the wetting properties of solids by liquids.

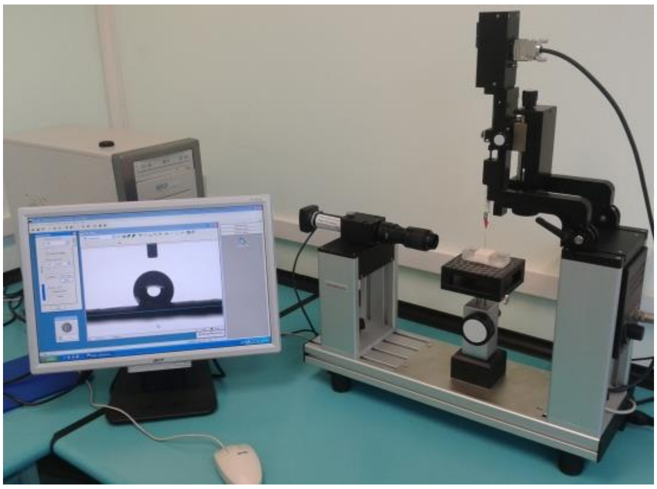
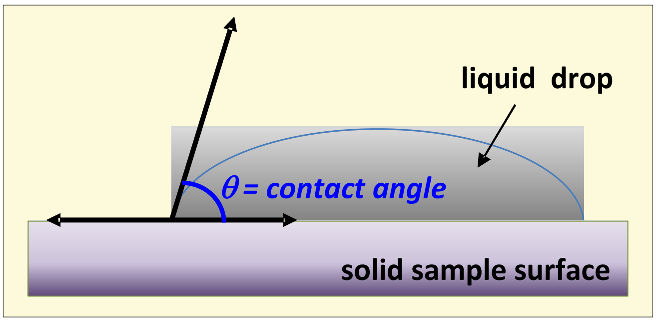


Figure-4. Optical Contact Angle and Surface Tension Measurement Apparatus

Contact angle (θ) is the quantitave measurement of a solid surface by a liquid. It is defined as the angle which is formed by the liquid drop at the three-phase border that gas, liquid and solid phases intersect. Contact angle is the angle between the solid and the liquid at the intersection point (Figure-5). θ values smaller than 90° indicates that the liquid spreads on the surface, or wets it.



Şekil-5. Temas açısı

Surface Free Energy is described as the force on the liquid surface which holds the unit length under tension. Surface Free Energy is calculated in terms of dyne/cm from the contact angles (θ) of at least two liquids.

The work necessary to increase the liquid surface by unit area is described as **Surface/Interfacial Tension**. The contact surfaces of a gas and liquid, or two immiscible liquids resemble an elastic membrane under tension. This tension is called as surface tension if it belongs to the free surface of the liquid; it is called as interfacial tension if it belongs to the interface of two liquids.

It is possible to determine these material properties with this instrument:

- Contact angle and Surace Free Energy in solids
- Surface/Interfacial Tension in liquids

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