Nitrogen- Sorption

Materials Science & Technology

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Introduction

Gas sorption can be used to determine the specific surface of a powder.
Furthermore the analysis of the empty pore system based on Kelvin condensation of the adsorbate at low temperatures is possible.

Measurement Principle

Figure 1: N_2 sorption equipment

The gas adsorption technique is performed by the addition of a known volume of gas (adsorbate), typically nitrogen, to a solid material in a sample vessel at cryogenic temperatures.
At cryogenic temperatures, weak molecular attractive forces will cause the gas molecules to adsorb onto (attach to the surface of) a solid material. An adsorbate (gas) is added to the sample in a series of controlled doses, the pressure in the sample vessel is measured after each dosing. There is a direct relationship between the pressure and the volume of gas in the sample vessel. By measuring the reduced pressure due to adsorption, the ideal gas law can then be used to determine the volume of gas adsorbed by the sample. The resulting relationship of volume of gas adsorbed vs. relative pressure at constant temperature is known as an adsorption Isotherm. From the analysis, and the cross-sectional area of the adsorbate gas molecule, the surface area and pore size distribution of the sample can be derived.

Relevance for Our Field

The analysis of the specific surface of a powder is possible applying N_2-sorption (BET-surface).

The specific surface of cement and additives plays an important role when composing a mixture of cementitious material and influences reactivity, packing density and rheology.

Furthermore, the inner surface and even the pore size distribution of a cement-based porous system may be analysed. This is important when studying gas or liquid transport, a well as hydration mechanisms.

Example

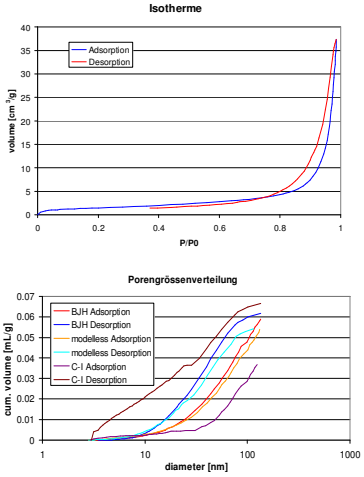
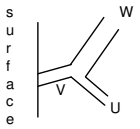


Figure 2:Full adsorption/desorption isotherm of a cement paste and the derived calculations of pore size with different models

The gas adsorption can be used to determine pore size distributions from sorption isotherms. Many different models considering adsorption on a surface and the condensation of the adsorbate in the small pores into account have been developed. Geometry considerations hereby play a crucial role. Furthermore one has to take connectivity aspects into account: Assuming that filling time is not important and equilibrium is achieved in the experiment, filling (adsorption) will be from the finer to the larger pores, meanwhile in a real pore system the desorption process is rather complicated and metastable Nitrogen very common.



Adsorption:
U → V → W

Desorption:
V + W → U

Applications & Potentials

- measurement of the specific surface of a powder
- measurement of the inner surface of a porous material
- determination of the micro-pore (< 2nm) content
- determination of the pore size distribution of a mesopourous pore system (2-100nm) from adsorption isotherms

Furthermore this method also may be executed using other gases like CO_2, H_2O etc.

Limitations

- pore size distribution is only possible on dried samples, drying level influences the result
- pores larger than about 100 nm in diameter are not analyzed.
- no standard theory is available as assumptions on the adsorption mechanism and pore geometry are necessary.
- The size of the used molecule (N_2, H_2O etc.) significantly influence the determined specific surface area, this may be explained by interlayer effects.