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Recent advances in the pore size analysis of nanoporous solids: Sorption, phase and hysteresis behavior of pure fluids confined to novel mesoporous molecular sieves and disordered porous networks

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The analysis of gas adsorption/desorption isotherms is widely used for the characterization of porous materials with regard to their surface area, pore size, pore size distribution, and porosity. However, in order to test the validity of appropriate theoretical models/methods on a quantitative level, model adsorbents are needed. Accordingly, recent advances in the synthesis of nanoporous materials of uniform pore structure and morphology have attracted much interest. Systematic, experimental and theoretical sorption studies of pure fluids on so-called mesoporous molecular sieves (as for instance MCM-41, MCM-48 and SBA silica's) have indeed served as a basis for a much better understanding of pore condensation and hysteresis phenomena. Aspects of these recent advances in gas adsorption and appropriate consequences for pore size analysis will be reviewed in this seminar. We focus in particular on the applicability of novel, microscopic methods (e.g. Density Functional Theory) for a combined micro/mesopore analysis. However, despite the above-mentioned progress in the understanding of the adsorption behavior of fluids in nanoporous materials, the influence of textural parameters (pore shape, degree of disorder etc.) on phenomena like pore condensation and hysteresis, is not completely understood and therefore still under investigation. In order to address these problems, we carried out an extensive, systematic, experimental study on the temperature and pore size dependence of the pore condensation and hysteresis behavior of pure fluids (nitrogen, argon, krypton) in mesoporous, silica molecular sieves (e.g., MCM-41, MCM-48, SBA-15,) and more disordered porous materials like silica gel, controlled-pore glasses as well as in novel mesoporous TiO₂ materials. Our sorption data reflect strongly the interrelation between the shape of sorption isotherms with: (i) pore size and temperature, i.e., differences in the thermodynamic states of the pore fluid and the bulk fluid, (ii) the geometrical heterogeneity (i.e., the degree of disorder) of the porous material. These factors have to be taken into account in order to obtain a comprehensive surface and pore size characteristics of the porous material. In addition we refer to related work, where the sorption, phase and hysteresis behavior of a nonwetting fluids (mercury) in mesoporous materials was studied. Appropriate consequences for the application of mercury porosimetry for pore structural characterization will be also briefly discussed.

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