261g Structural Characterisation of Adsorbed Films in Periodic Mesoporous Silica by in-Situ Small-Angle X- Ray Diffraction

Susanne Jaehnert, Gerald Zickler, Oskar Paris, and Gerhard H. Findenegg

Mesoporous silica materials with pores of uniform size and shape, such as MCM-41 and SBA-15, can be used to study phase transitions of fluids in strongly confined systems. Due to the periodic arrangement of the pores in these materials it is also possible to apply diffraction techniques to extract structural information about materials incorporated in the pores. We have recently developed a technique which allows in-situ structural studies of physisorbed films in MCM-41 type materials by small-angle X-ray diffraction (SAXD). The method is based on an analysis of the intensities of the Bragg peaks resulting from the 2D hexagonal array of the pores. For physisorbed films this dependence can be modeled by the form factor of a core-shell cylinder, with the adsorbed film forming a shell of thickness d. As d increases along the adsorption isotherm, the minima of the form factor are shifted to higher q, thus causing systematic changes of the intensity of the individual Bragg peaks. This novel technique has been used to study the physisorption and pore condensation of organic vapors in a SBA-15 silica material of 8 nm pore diameter. We derive information about the individual stages of pore filling, i.e., the micropore adsorption into the corona of the matrix around the mesopores, the formation of a multilayer adsorbed film at the walls of the cylindrical mesopores, and the thickness of that film at the pore condensation pressure. In this way we can test existing models of the pore filling and of the thickness of physisorbed films at highly curved interfaces. In addition, the effect of the strength of fluid-wall interactions was assessed by comparing the measured film thickness in the native and surface modified SBA-15 material.