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Plant tissues as hierarchical templates for inorganic nanomaterials

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The modification or transformation of plants has been used since hundreds of years to obtain a broad variety of useful materials, such as for instance paper or activated carbons. In particular the rich structural hierarchy of plant tissues makes them ideal as scaffolds or casting moulds for inorganic materials such as carbon or ceramics. The cellular structure and the nanocomposite character as well as unique directional mechanical properties of the biological templates can be expected to yield entirely new materials with advanced properties. Besides potential applications as lightweight nano-composites for structural applications, such materials are typically porous at several length scales, making them interesting candidates for applications as catalysts and filters. One of the major challenges in the synthesis process of such materials is to preserve or to replicate the hierarchical plant structure at all levels down to the nanometer scale while retaining the mechanical integrity.
The structural characterization of such materials requires the combination of several experimental techniques covering multiple length scales. Besides electron microscopy, in particular X-ray diffraction (XRD) and small angle X-ray scattering (SAXS) are very useful techniques to study structural features of such weakly ordered systems at the molecular and at the nanometer scale. In the present contribution we show, how SAXS and XRD can help to understand, to quantify and finally to tailor the structural details of inorganic nanomaterials derived from plants by different synthesis approaches.
In recent years we have been successful in transforming wood into cellular carbon materials, and have investigated their nanostructure and preferred orientation with SAXS/XRD and their mechanical properties with nanoindentation. Moreover, we started to investigate silica-rich plants as hierarchical templates for the synthesis of silicon carbide or SiC/C composites by simple pyrolysis. Besides these direct conversion processes, we used wood tissue as a casting mould for the synthesis of mesoporous oxide ceramics with directional porosity on the micrometer and the nanometre scale. This was done by infiltrating nanoparticle sols into delignified wood, followed by drying and calcination. In particular, we could show with the help of SAXS/XRD that the entire hierarchical structure of the wood tissues including the microfibrillar orientation of the cellulose fibrils can be transformed into a mesoporous $Ce_{0.5}Zr_{0.5}O_2$ ceramic.

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Physisorbed films in periodic mesoporous silica studied by *in-situ* synchrotron small-angle diffraction

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Mesoporous materials are attracting great attention due to commercial interest in their applications in chemical separations and heterogeneous catalysis, as well as scientific interest in the challenges posed by their synthesis, processing, and characterisation. It is well known that the filling of nanometre-sized pores proceeds via the formation of an adsorbed film. The film is growing in thickness with increasing vapour pressure, until a sharp increase of the adsorbed mass occurs due to capillary condensation of the vapour. Capillary condensation represents a first-order phase transition in a confined geometry and commonly exhibits a pronounced hysteresis with respect to adsorption and desorption. In the present work, the ordered mesoporous silica material SBA-15 [1] with cylindrical pores of uniform size was used to study sorption of an organic liquid (perfluoropentane, C_5F_{12}) *in situ*. Due to the periodic arrangement of the pores in SBA-15 it is possible to apply diffraction techniques to extract detailed structural information about pore walls, adsorbed films and changes upon capillary condensation. A dedicated cell was designed and fabricated for *in-situ* sorption at room temperature. Small-angle X-ray diffraction patterns were collected during continuous adsorption and desorption at the beamline A2 at HASYLAB (Hamburg, Germany). The transmission of the samples was determined *in situ* during sorption by using an ionisation chamber in front of the sample cell to monitor the primary X-ray flux and a photodiode mounted in the beamstop to measure the transmitted photons. Seven diffraction peaks from the two-dimensional hexagonal pore lattice were clearly detected. The diffraction peaks changed their intensities with varying vapour pressure and after exceeding the pore condensation point a strong decrease of peak intensities was observed. Data evaluation was based on the analysis of the integrated intensities of the diffraction peaks with a structure model for the electron densities in the pores including the bulk silica matrix, a microporous corona of uniform density and a liquid film. The model was used to fit the experimental intensities, providing quantitative information about the structure of the degassed sample as well as about the growing liquid film [2]. In the present contribution we present the results of this *in-situ* investigation of sorption in ordered mesoporous materials and we discuss their scientific and practical relevance.

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