Water-free processing of novel porous materials via cryoextraction and supercritical drying

Pavel Gurikov[1,2] and Raman S.P. [1,2]

Laboratory for Development and Modelling of Novel Nanoporous Materials, Hamburg University of Technology, Eissendorfer Str. 38, 21073 Hamburg, Germany
Aerogelex UG, Barlachstr. 2, 21073 Hamburg, Germany
e-mail: pavel.gurikov@tuhh.de

Low-temperature supercritical drying (SCD) with CO_2 remains the golden standard for processing of wet gels into corresponding aerogels [1]. In a typical protocol, a gel is first prepared from nano-scaled building blocks, usually, in water followed by a solvent exchange to ethanol and extraction process. The described pathway preserves a considerable amount of the space between the building blocks created at the gelation step and results in highly porous materials (aerogels). This pathway is however fundamentally limited by the ability of the precursor to form a gel [2]. The aim of this work to pave the way for the processing of any kind of starting materials into aerogels.

The core idea of the novel approach is to dissolve or disperse the starting material in a pure or mixed solvent with a moderate melting point (0 – 25 °C). The mixture is then frozen and subjected to cryoextraction with an organic solvent as extracting agent (e.g., acetone) or directly with liquid/sc-CO_2. The cryoextraction temperature is determined from corresponding solid-liquid equilibrium data. Finally, the extracting agent recovered in the SCD process leaving behind a highly porous matrix.

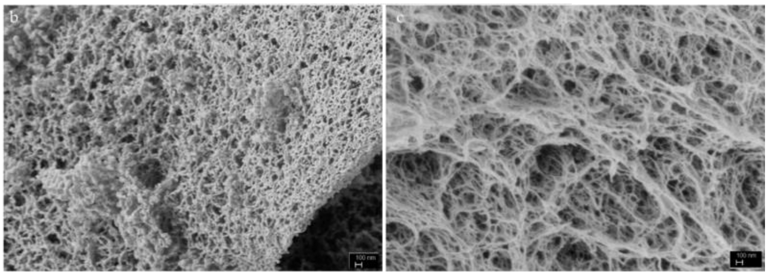
Figure 1: Porous structures from the novel freezing/sc-drying approach.

We exemplify the suggested approach by processing industrial polymers and some low-molecular weight compounds into novel nanoporous materials. In many cases such materials can be processed in a fully water-free manner what remain a challenge in conventional approaches. We discuss how the pore size and morphology can be controlled by the solvent composition and how the process parameters can be deduced from phase equilibria data.

References:

1. Smirnova, I., Gurikov, P.; *Annual Review of Chemical and Biomolecular Engineering* **2017,** *8,* 307 – 334.
2. Gurikov, P., Smirnova, I.; *Gels* **2018,** *4*, 14.