

Protein Aerogels: Insights into solvent exchange and shrinkage phenomena using 1-D Raman Spectroscopy

Martin Peter Dirauf,^{a,*} Pavel Gurikov,^b Andreas Siegfried Bräuer^a

^a*Institute of Thermal-, Environmental-, and Resources' Process Engineering (ITUN),
Technische Universität Bergakademie Freiberg, Germany*

^b*Laboratory for Development and Modelling of Novel Nanoporous Materials, Hamburg
University of Technology, Germany*

*Martin.Dirauf@tu-freiberg.de

During various steps of bio-aerogels synthesis such as solvent exchange or supercritical drying, unwanted shrinkage of the gel matrix can take place, thereby deteriorating the unique properties of the final aerogels. Here, we experimentally investigate into the shrinkage of whey protein hydrogels during solvent exchange in pure ethanol using the combination of three different techniques: (i) spatially resolved Raman spectroscopy for *in situ* analysis of the radial concentration profiles of water, ethanol and proteins together with a (ii) conventional camera for real time monitoring of the diameter change of the gel. Furthermore, (iii) the compressive strength of the gel cylinders during solvent exchange is evaluated using a universal testing machine. For gel cylinders with a diameter of 10 mm, we report most of the shrinkage takes place in the first 90 min of solvent exchange. Up to this point, solvent exchange is detected only in the outermost layers of the gel. Parallel measurements of compressive strength of the entire gel show an increase by a factor of three during this period, which likely stops further shrinkage. After completion of solvent exchange, we report a non-homogeneous density distribution of the protein matrix: the gel shows a compactification of its matrix that is especially pronounced in its center.

Acknowledgement

The authors gratefully acknowledge financial support for parts of this work through the projects BR 3766/19-1 and GU 1842/3-1 by the German Research Foundation (Deutsche Forschungsgemeinschaft – DFG).