

First evidence of solvent spillage under subcritical conditions in aerogel production

Alberto Bueno^{*a}, Ilka Selmer^a, Raman S.P.^a, Pavel Gurikov^a, Wibke Lölsberg^b, Dirk Weinrich^b,
Marc Fricke^b, Irina Smirnova^a

^a Hamburg University of Technology, Institute of Thermal Separation Processes, Eißendorfer Straße 38, 21073 Hamburg, Germany.

^bBASF Polyurethanes GmbH, Elastogranstr 60, 49448 Lemfoerde, Germany

* alberto.bueno@tuhh.de

Production of aerogels starting from almost all precursors requires a supercritical drying step to preserve the mesoporous structure of the gel. Such systems include most of organic gels derived from synthetic polymers and almost all biopolymers. Supercritical drying, which requires high pressures, increases the initial capital expenditure for the production facility.

A common practice in the industry and research facilities is to divide the supercritical drying process in three main steps: pressurization, extraction and depressurization. The pressurization and depressurization are considered dead times since the common thought is that no solvent can be extracted during these stages. The rush through the pressurization step requires however significant compression capabilities which translate to considerable capital costs. Meanwhile the rate of depressurization step depends mainly on the nature of the material.

Integrating the pressurization and the extraction into one efficient extraction step would require smaller compression capabilities reducing the capital expenses and thus making it more attractive to the industry. This can be achieved by taking advantage of the solvent expansion phenomena that takes place at subcritical conditions and avoids the gas/liquid interface to propagate into the pore structure. Here, we present the first evidence of solvent spillage under subcritical conditions during aerogel production.

The main objective was to understand the underlying phenomena controlling the solvent removal kinetics during autoclave pressurization. Alginate, silica and polyurethane as gels and ethanol, MEK and ethanol/water as solvents were investigated. During solvent expansion at subcritical conditions, CO₂ gas diffuses into the solvent contained in the gel pores causing the solvent mixture to increase its volume and flow out of the gel. Low temperatures during this phase allows to keep a large mass transfer driving force between the gas/gel interface and a sooner solvent spillage due to a higher gas solubility and a lower compressibility of the mixture.