

## SCF 529

### Drying of Aerogels with Supercritical Carbon Dioxide

#### Introduction

An aerogel is a nanoporous material that has a high surface area, low density, and low thermal conductivity. They can be made of organic or inorganic material, but the most common forms are made from inorganic-metallic materials by the sol-gel polymerization of selected silica and carbon.

Aerogels can be produced as monoliths with extraordinarily large surface areas and high porosities. Due to their unique composition, aerogels have a wide variety of current applications, including the production of insulators, sound absorbers, catalyst supports, adsorbents, and supercapacitors. Silica aerogels, for example, are often used as thermal insulators as they can withstand temperatures up to 1000°C and can be produced in tiles or plates.

Typically, silica aerogels are prepared by a two-step process using ethoxysilanes. This process produces a silica gel skeleton in ethanol. The ethanol is removed by placing the wet gel in an autoclave and heating the autoclave to between 250-300°C. This method is very time-consuming due to the high temperatures needed to heat the autoclave. Another problem with traditional methods of drying is that gels often crack under the stress created by interfacial tension at the meniscus of liquid and vapor.

Supercritical CO<sub>2</sub> is an alternative aerogel drying technology that significantly reduces the

time, solvent consumption, and temperatures needed to make monolith silica aerogels.

In addition, since there is no distinction between liquid and vapor phases at the supercritical conditions, the supercritical drying process eliminates interfacial tension to produce crack-free aerogels.

This application describes a method for drying silica aerogel rods with supercritical CO<sub>2</sub> and ethanol.

#### Equipment

- ✓ Applied Separations' Helix Supercritical System



#### Materials

- ✓ Ethanol-HPLC grade
- ✓ Silica gel rods
- ✓ Autoclave
- ✓ Carbon dioxide –supercritical grade

#### Supercritical Drying Method

Place silica gel rods in an autoclave filled with ethanol. Pressurize the vessel to 750-850 psi with CO<sub>2</sub> and cool to 5-10 °C. Flush liquid CO<sub>2</sub> through the vessel until all the ethanol has been removed from within the gels and vessel. Once

the gels are free of ethanol, heat the vessel to 35°C. Release CO<sub>2</sub> to maintain a pressure slightly above the critical pressure of CO<sub>2</sub> (around 1200 psi). The system is held at these conditions for a length of time depending on the thickness of the gels.

Finally, the CO<sub>2</sub> is slowly released to ambient pressure while maintaining the vessel temperature above 31 °C.

## Drying Conditions

### *Extractor*

Vessel:	500 mL
Pressure:	1200 psi
Temperature:	35 °C
Valve temperature:	130 °C
CO <sub>2</sub> Flow Rate:	10 LPM (gas)
Drying time:	2 hours
Depressurization:	2 Bar/min

## Conclusion

Supercritical CO<sub>2</sub> technology can produce dry, crack-free aerogel rods quickly and economically. In addition, solvent use is minimized and the high temperatures required by conventional drying methods are reduced.

## References

van BOMMEL, M.; de HAAN, A. "Drying of silica gels with supercritical carbon dioxide." *Journal of Materials Science* **29** (1994) 943-948.