SCF Application #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_2 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_2 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_2 and cool to 5-10 °C. Flush liquid CO_2 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_2 to maintain a pressure slightly above the critical pressure of CO_2 (around 1200 psi). The system is held at these conditions for a length of time depending on the thickness of the gels.

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Finally, the CO_2 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 ºC
Valve temperature:	130 ºC
CO_2 Flow Rate:	10 LPM (gas)
Drying time:	2 hours
Depressurization:	2 Bar/min

Conclusion

Supercritical CO_2 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.

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