SFE529: Aerogel Drying using Supercritical CO_2

Introduction

Gel

A gel is typically composed of a polymer network that is filled throughout its whole volume by a fluid. If the fluid is water the gel is known as a hydrogel, if the fluid is an alcohol the gel is termed an alcogel, and if the fluid is a gas the gel is classified as an aerogel.

Aerogels

Aerogels are porous materials in which the liquid component of the gel has been replaced with a gas, without the collapse of the gel structure. Aerogels are produced by extracting the liquid component of a gel with supercritical CO2. This technique allows the liquid to be extracted without causing the solid matrix in the gel to collapse from capillary forces caused in conventional evaporation. This technique results in a solid with extremely low density and extremely low thermal conductivity.

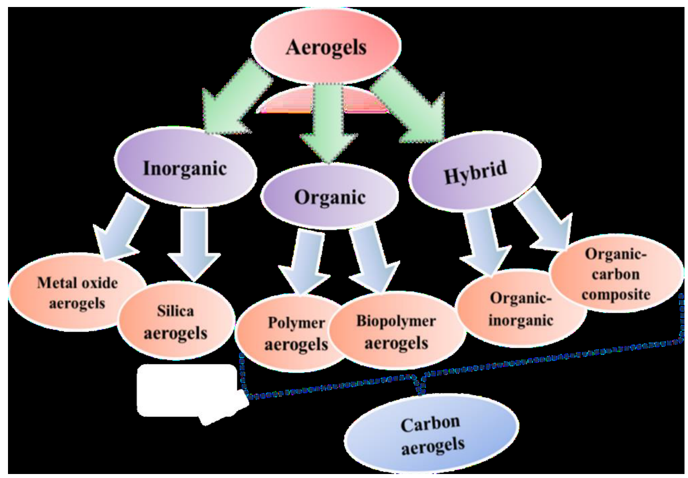
Classification of aerogels

Different forms, microstructures and composition of aerogels can offer greater functionality of aerogels for new applications.

Aerogel Forms- monoliths, granules, powders, and films
Aerogel Microstructure : microporous, mesoporous, and microporous.

Aerogel Chemical Composition

- organic, inorganic, and hybrid.
Aerogels can be divided into three compositional categories: inorganic (metal and metal oxides), organic (synthetic polymers and biopolymers), and organic–inorganic hybrid materials **.** Carbon aerogels are another class of aerogels with unique physical properties that are a result of pyrolysis of organic and hybrid composite aerogels.



Equipment

Aerogels may be dried in Applied Separations Helix, SFE-2 , and basic models. In addition, Applied Separations offers specialized Helix and Sirocco systems that include automatic depressurization software for aerogel drying.

Applied Separations – Aerogel Dryer

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Materials

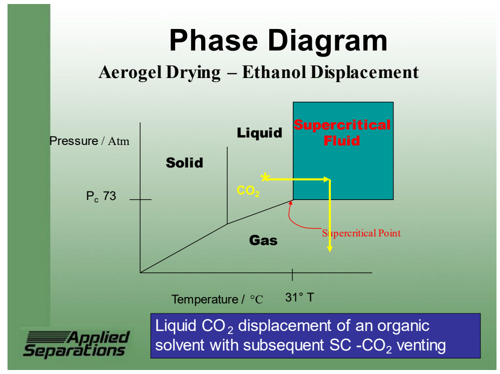
Ethanol
Alcogel
Autoclave
Carbon dioxide (liquid CO2 cylinder)

General Supercritical Drying Method

(See the attached references describing the use of Applied Separations equipment for detailed procedures)

Place the alcogel in an autoclave filled with a measured amount of ethanol. Pressurize the vessel to 100 bar with liquid CO2. Flush liquid CO2 through the vessel until all the ethanol has been removed from the vessel and the gel. Once the gels are free of ethanol, heat the vessel to 40°C and convert the liquid CO2 into supercritical CO2. Continue to extract the gel with a dynamic flow of supercritical CO2 for a specified time to ensure all residual alcohol or liquid solvent is removed.

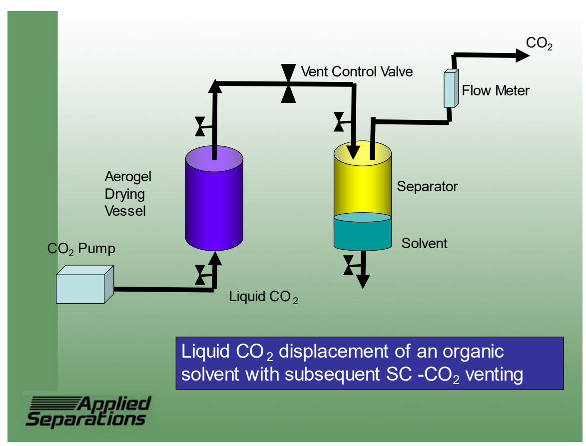
The system is held at these conditions for a length of time depending on the thickness of the gels. When the extraction of all the alcohol is complete , depressurize the vessel to atmosphere while maintaining the temperature above the critical temperature of CO2 (31 C). In this way supercritical CO2 will depressurize directly to a gas and bypass the liquid phase and avoid any capillary forces that may collapse the aerogel. (see Phase Diagram)



Drying Conditions

Extractor

Vessel: 1000 mL
Pressure: 100 bar, 1500 psi
Temperature: 40 ºC for supercritical state
Valve temperature: 130 ºC
CO2 Flow Rate: 1 to10 LPM (gas)
Drying time: 1 to 5 hours
Depressurization: 2- 5 Bar/min
(Finally, the CO2 is slowly depressurized to ambient pressure while maintaining the vessel temperature above 31 °C.)



Applied Separations – Helix Aerogel Dryer Schematic

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Applications

Aerogels are used in diverse applications including:

Energy – thermal insulation, phase change materials for energy storage, methane storage in aerogels

Environmental – water and air treatment adsorbents, valorization of wastes and bio-mass products

Biomedical - Carriers for poorly water-soluble drugs including proteins and polypeptide-based drugs, synthetic bone grafts for regenerative medicine , dressings for wound healing

Aerospace- silica aerogel polymer nanocomposites for cryogenic propellant tank storage ,space suit thermal insulation and fire resistance.

Textiles-coatings for water, fire, and chemical resistant fabrics, alginate aerogel synthesized in PET nonwoven fabric

Optics/ Electronics - opto-fluidic waveguides, organic light emitting diodes (OLEDs), and chemical sensing technologies

Conclusion

Supercritical CO_2 technology can produce dried aerogels quickly and economically.

Capillary forces that collapse aerogels are avoided by depressurizing from the supercritical CO_2 phase directly to the gaseous CO_2 phase at low temperatures and pressures.

The safety concern generated using flammable supercritical ethanol or other flammable supercritical organic solvents is eliminated by using CO_2. There is no need to reach the critical temperature of ethanol at 241 C.

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