



Thermodynamic and hydrodynamic study of a gas-liquid flow in a cyclone separator downstream supercritical drying

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ARTICLE INFO

Article history:

Received 16 March 2016

Received in revised form 21 July 2016

Accepted 21 July 2016

Available online 22 July 2016

Keywords:

Aerogel

Supercritical CO₂

Organogel

Cyclone separator

Thermodynamic modeling

CFD

ABSTRACT

Aerogels may be synthesized by extracting the liquid solvent from an organogel using supercritical CO₂. The mixture (CO₂-solvent) leaving the autoclave should be separated and all the solvent in the sample should be recovered at the end of the extraction. This paper deals with a pilot-scale unit where CO₂ and the solvent, here toluene, are separated using a cascade of cyclone separators. During the separation, the toluene recovery rate during is below 65%. In order to determine the reasons behind this malfunction, a thermodynamic study and a hydrodynamic study (Computational Fluid Dynamics, CFD) of the mixture separation in the cyclones were carried out. According to CFD, cyclones allow an efficient mechanical separation of the liquid solvent and the gaseous CO₂. On the other hand, the thermodynamic model revealed that under the actual operating conditions, a large fraction of the solvent remains in gaseous state and is carried away through the cyclone gas outlet, hence the poor overall separation performance.

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1. Introduction

Aerogels are ultra-light solid materials derived from gels. They are composed of up to 99.98% volume of air. Thereby, they are almost weightless and are very efficient heat insulators. These unique properties make aerogels attractive for a variety of applications in the chemical, aerospace and car industries. In particular, aerogels could be used as super-insulators in building walls [1]. They can also be integrated into transparent walls, particularly in double glazing [2,3]. Therefore, during the last 30 years, aerogels have been the subject of an increasing number of research works [4]. Different techniques, have been developed to obtain aerogels from gels containing a solvent such as the processes of evaporative drying [5], freeze drying [6,7] and supercritical drying.

Supercritical drying of gels has been so far the option with the most satisfying results [4]. This process consists in extracting the solvent from the gel using supercritical CO₂ (SC CO₂). In these

conditions, the density difference between the two phases disappears, nullifying the surface tension [8]. The resulting aerogel shows good thermo-mechanical properties.

The first aerogels produced were silica aerogels in the early 1930s [9]. They were obtained by drying silica gel. The solvent, to be extracted, was generally an alcohol (ethanol [10–14], methanol [15], liquid isopropanol [16], or 1-butanol [17]). Extensive research has been carried out on supercritical drying of silica gel. A drying mechanistic model showed that the drying process was conducted by the dissolution phenomena of CO₂ into ethanol, the solvent, rather than convective evaporation. It indicated that the production of crack-free transparent aerogels depends on pressure, temperature, gel thickness, and CO₂ velocity [12]. Other studies provided mathematical models and simulations of alumina/silica gels drying [14,17]. It was also shown that the aerogels properties depend on the nature of the gel (organic or inorganic) and the drying process duration [13].

The gel considered in this paper is an organogel, which is a physical gel composed of gelator molecules organized in a three-dimensional supramolecular network able to trap solvent molecules and preventing them from flowing [18]. The organogelation phenomenon involves some low molecular weight molecules

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(<2000 g mol⁻¹), like *n*-alkanes (hexatriacontane [19]), crown ether [20], calixarene [21] and retains the interest of some scientists.

These small molecules generally lead to thermoreversible gels, very attractive for numerous industrial applications [22–32]. Organogels are generally dried by an extraction process with supercritical CO₂ to obtain aerogels [33]. These aerogels, besides the properties given above have another interesting characteristics: their hydrophobicity. In particular in the field of thermal insulation, hydrophobicity is a major advantage in the choice of material [34]. The Laboratory of Macromolecular Physical Chemistry (LCPM, Nancy, France) showed that it was possible to obtain gels from amino acid derivatives and appropriate organic solvents yielding the organogels employed in this study [35,36].

Once the solvent is extracted from the gel, the CO₂-solvent mixture needs to be separated. Several techniques may be used for gas-liquid separation, among which active centrifuges (i.e. rotational devices) and passive inertial equipment, e.g. cyclonic separation devices [37,38].

Cyclones are mechanical separators used to separate liquid-liquid, solid-liquid, gas-solid or gas-liquid mixtures. They consist of a vertical pipe fed with the mixture through a tangential inlet, and of two outlets, one at the bottom and one at the top of the device. The tangential feed provides swirl motion to the incoming mixture which enhances the separation efficiency compared to gravity separators. Indeed, denser components undergo higher centrifugal forces and tend to accumulate near the wall and to fall down, being discharged from the bottom outlet, while lighter components exit from the top outlet. Cyclones do not require much maintenance (no moving parts) and their operating costs are low since they do not need much energy to operate [39,40].

A study of the CO₂/solvent separation is required after the supercritical extraction. For some processes, this separation was carried out by simple cooling of the mixture, a very economical technique [10,14,15]. Meanwhile, depressurization technique was also adopted by using cyclone separators [41,42]. The coupling of extraction/separation led some authors to study and model the two steps together [43]. However, few researchers have used or modeled cyclones to separate CO₂ gas from a liquid, regardless of the extraction step as it is used in this work [44,45].

In this study, drying of organogels made of amino acids and toluene solvent are studied. Drying experiments are presented in Section 2. In these experiments, SC CO₂ is blown around the organogel in order to extract the toluene. The CO₂-toluene mixture is then separated using cyclones. But poor separation efficiency is achieved: a large amount of solvent is not recovered in the liquid outlets.

In order to determine the reasons behind this malfunction, a thermodynamic study and a hydrodynamic (Computational Fluid Dynamics, CFD) study of the cyclones are performed. CFD simulations were carried out, using the commercial code ANSYS Fluent, Section 3. They aimed at determining the efficiency of the mechanical separation in the cyclone. In Section 4, thermodynamic modeling is performed using PRO/II software, with the purpose of calculating the composition and state of the mixture at the cyclone outlet.

2. Experimental study

2.1. Products

First, an amino acid organogelator is synthesized (see [35] for details). The obtained organogelator is then dissolved into the toluene solvent (C₇H₈) under heating in a flask fitted with a reflux condenser. After cooling, the mix is poured into a cylindrical mold (30 mm diameter and 15 mm height) and stored at 4 °C.



Fig. 1. Simplified scheme of supercritical drying.

2.2. Experimental set-up

As it is shown schematically in Fig. 1, a cylindrical organogel of 30 mm of diameter and a thickness varying between 5 and 15 mm is initially submerged in toluene, in an extractor (autoclave) of 210 mL. It is then dried by pumping supercritical CO₂ through the extractor at a flow rate ranging from 300 to 750 g/h. Temperature and pressure of supercritical drying are set to 45 °C and 90 bar respectively, so that the toluene-CO₂ mixture is supercritical [46]. The experiment duration varied from 90 min to 250 min depending on the run. Downstream of the drying unit, the mixture of SC CO₂ and solvent (toluene) is depressurized in order to liquefy the toluene. The mixture is then transferred to a cascade of three cyclone separators, so as to mechanically separate the liquid toluene and the CO₂ gas phases: hence, the solvent is recovered and CO₂ is discharged outside of the unit.

The drying process is composed of three main systems detailed in Fig. 2.

In the feed line **A**, gaseous CO₂ is initially fed from the cylinder at a pressure of 50–60 bar using valve 1. The gas is then cooled in the cold exchanger (I) down to a temperature of 4 °C to be liquefied and flows through the LEWA pump (EK1). Next, the CO₂ enters the damper (III), which reduces pressure fluctuations so as to provide steady operating conditions for drying. The CO₂ flow then goes through a pressure regulator (IV) and a heat exchanger (V) so that it reaches the operating conditions of 90 bar and a temperature of 15 °C (during step 1, Fig. 3) or 45 °C (during step 2, Fig. 3). The second stream returns upstream the pump through the CO₂ tank (II). At the outlet of the heat exchanger, the supercritical CO₂ leaves the feed zone at 15 °C or 45 °C (Fig. 3) and 90 bar. A Coriolis mass flowmeter Micromotion (VI) measures the cumulative mass of CO₂ (±1g.) and the instantaneous CO₂ flow rate.

In the autoclave **B**, the organogel is dried using SC-CO₂. Firstly, the organogel sample is introduced into the autoclave (VII), at an initial temperature of 15 °C. In order to prevent premature evaporation of the solvent contained in the gel at the process starting up, it is submerged with a pure solvent volume (quantity of solvent added = 3.5 g). Valve 4 is opened while valve 5 is kept closed in order to fill the autoclave with liquid CO₂ (15 °C, 90 bar), which comes into contact with the organogel and the free solvent. A Bourdon manometer (P3, Fig. 2) provides a reading of the pressure in the autoclave. The system remains in these conditions for 35 min (Fig. 3: step 1 and 2). The temperature is increased in less than 10 min up to 45 °C (Fig. 3, step 3). These conditions are maintained

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