The Journal of Supercritical Fluids 133 (2018) 38–41

Contents lists available at ScienceDirect

The Journal of Supercritical Fluids

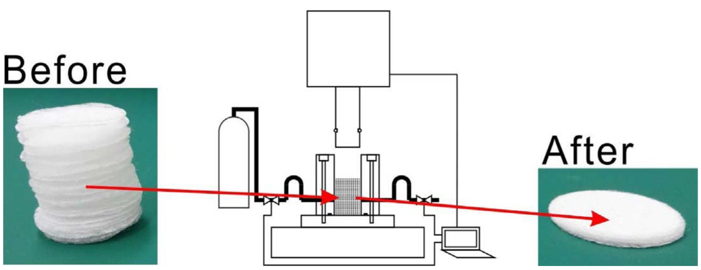
journal homepage: www.elsevier.com/locate/supflu

A new method for producing porous polymer materials using carbon dioxide and a piston

Takafumi Aizawa*

National Institute of Advanced Industrial Science and Technology, Research Institute for Chemical Process Technology, 4-2-1 Nigatake, Miyagino-ku, Sendai 983-8551, Japan

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:
Polymers
Carbon dioxide
Porous materials
Caulking

ABSTRACT

A new method for manufacturing a porous polymer material is developed. In this method, nonwoven fabric sheets are placed in a high-pressure vessel and carbon dioxide is introduced at its vapor pressure. Then, stacked nonwoven fabric sheets are pressed by a piston along with liquefying carbon dioxide to produce a porous polymer material. Through holes were generated in the manufactured polymer material and scanning electron micrographs revealed that the polymer fibers largely remained intact. Surface observation after peeling off the sheet indicates that physical bonding by caulking to be the possible bonding mechanism. According to this technology, stacking sheets with different mesh roughness could enable the fabrication of a filter with a gradient in the mesh. Pressing an enzyme or a catalyst within the stacked fabric sheets can create a reaction cartridge. Drug-containing porous materials that can control the drug-release rate are also expected.

1. Introduction

Processes using carbon dioxide as an environment-friendly solvent has attracted much attention [1–4]. In terms of material synthesis, processes controlling solubility through external factors such as the temperature and pressure of carbon dioxide, and those using high permeability (high diffusibility) of carbon dioxide have been proposed [5,6]. The safety of carbon dioxide is also evident from the existence of carbonated drinks and its practical application as an extraction solvent [7,8]. Many methods have also been proposed for processes focusing on carbon dioxide and polymers [9–15]. For example, polymerization processes in carbon dioxide [10,11], and fabrication of fine polymer particles [12,13] and foamed polymers [14,15] using carbon dioxide have been proposed. In most of these processes, supercritical carbon dioxide at a temperature higher than its critical temperature is used.

Porous materials with through holes could serve as filters by themselves or used for various industrial applications if they carry relevant objects therein. For example, they could be applied in the pharmaceutical field by loading a medicine and in a reaction cartridge to carry an enzyme or a catalyst. Application in the field of drug

* Corresponding author.
E-mail address: t.aizawa@aist.go.jp.

http://dx.doi.org/10.1016/j.supflu.2017.09.018
Received 31 July 2017; Received in revised form 14 September 2017; Accepted 14 September 2017
Available online 14 September 2017
0896-8446/

Fig. 1. Photograph of the equipment. Valves are located at the side of the servo-press unit.

delivery systems is expected if the porous material can facilitate a facile, controlled release of the loaded drugs.

This paper reports an easy method for producing a porous polymer material by caulking fibrous sheets in liquid carbon dioxide. The advantage of using liquid carbon dioxide instead of supercritical carbon dioxide is that the device configuration becomes simple. Specifically, because it is a room-temperature process, a temperature-control device is not necessary. Further, liquefaction with a piston eliminates the necessity of a high-pressure pump. As carbon dioxide is nontoxic, the process is suitable for food or medical processing. Moreover, since carbon dioxide is eliminated from the polymer under the atmosphere condition, it is an extremely safe process that does not cause contamination. This technology can be potentially applied to drugs that may be denatured by heat, because it is a room temperature process.

2. Experimental setup

Fig. 1 shows the photograph of the experimental setup. A piston and high-pressure vessel were designed, manufactured, and attached to servo-press unit JP-1504 manufactured by Janome Sewing Machine Co., Ltd. A sectional view of the piston and the press unit is shown schematically in Fig. 2. The vessel (inner diameter = 20 mm) and piston (outer diameter = 19.5 mm) were made of SUS 316 stainless steel. The vessel is divided into a body (B1) and base plate (B2) for retrieving the sample after processing. The body and base plate were sealed with an O ring and then connected with four hexagon socket cap screw bolts. An O-ring groove was provided on the piston halfway and the space inside the vessel was sealed with an O-ring after the piston goes down. Two 1/16-inch stainless steel tubes were connected to the body. One was for introducing carbon dioxide and the other for exhausting it. Swagelok ball valves with air actuators were installed on both sides of the servo-press unit and connected to the vessel body with 1/16-inch stainless steel tubes. The stainless steel tubes were equipped with slacks to render the vessel flexible for pulling out to the front when

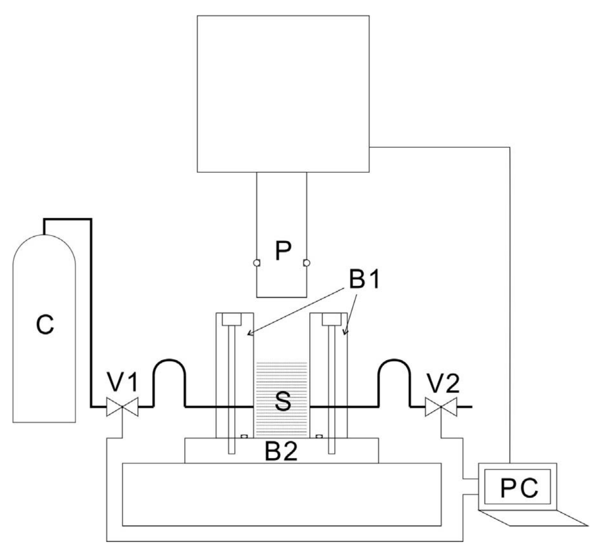


Fig. 2. Schematic illustration of the cross-section of the high-pressure vessel. B1: Body of the high-pressure vessel, B2: Base of the high-pressure vessel, C: Carbon dioxide cylinder, P: Piston, PC: Laptop PC, S: Sample, V1: Intake valve, and V2: Exhaust valve.