



3D Microstructure of supercritical fluid extrudates. II: Cell anisotropy and the mechanical properties

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ARTICLE INFO

Article history:

Received 2 February 2008

Accepted 21 December 2008

Keywords:

Supercritical fluid extrusion

3D microstructure

Anisotropy

ABSTRACT

The mechanical properties of biopolymeric cellular foams are often governed by their microstructure. 2D and 3D microstructural data of supercritical fluid extrudates were obtained with X-ray microtomography and correlated with the mechanical properties determined using compression and three-point bending tests. Cell size from transverse cross-sections of SCFX extrudates decreased with radial distance from the center. In the longitudinal direction, the cell shapes were more elliptical than spherical and were aligned along the extrusion direction. These findings indicated the presence of a certain degree of anisotropy in SCFX extrudates in both directions. Both piece density and the ratio of cell wall thickness to cell diameter were observed to be good predictors of compressive and flexural mechanical properties, including jaggedness parameters. Compressive modulus data suggested that cell shape anisotropy due to cell elongation in the longitudinal direction actually affected the mechanical properties of SCFX extrudates. X-ray microtomography was found to be useful to investigate 2D and 3D morphology of SCFX extrudates, including cell shape and cell size anisotropy.

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1. Introduction

In our previous paper, the effects of supercritical fluid extrusion (SCFX) process conditions and formulations on the melt rheology, as well as the expansion characteristics and 3D microstructure formation of SCFX extrudates were described (Cho & Rizvi, 2009). In this study, the mechanical properties of SCFX extrudates are presented in relation to their microstructure because the quality attributes of expanded foods are often judged from their texture (Peleg, 1997).

The mechanical properties of cellular foams are governed jointly by their cell wall material property and the cellular structure that can be characterized as the ratio of open to closed cell, average cell size, cell size distribution, cell wall thickness, cell shape, uniformity of the structure, and presence of skin (Peleg, 1997). However, regardless of their microstructure, the product density (i.e., either piece or bulk density) has been widely used as a predictor of their mechanical properties due to the simplicity of measurement and the reasonable correlation with the textural properties. In general, more expanded products with lower product density exhibit low breaking strength and compressive modulus (Sokhey, Rizvi, & Mulvaney, 1996). Fang and Hanna (2000) proposed a simple power law relationship between the mechanical properties and the density of starch-based extruded foams. The cellular solid approach of Gibson and Ashby's model was found to be useful to predict the mechanical

properties of solid food foams, such as starch-based extrudates and bread (Liu & Scanlon, 2003). However, discrepancies with regard to the exponent value of Gibson and Ashby's model and a lack of fit of experimental data to the power law for cellular food products are often reported (Liu & Scanlon, 2003; Warburton, Donald, & Smith, 1990). There are several potential reasons for this. First, it might be due to the fact that Gibson and Ashby's model utilizes the mechanical properties of the solid matrix, which are difficult to determine experimentally with precision (Hutchinson & Mantle, 1989). Second, the microstructure attributes of cellular solid foams with the same density can vary. Destrumaux, Bouvier, and Burri (1998) have proposed that corn grits extrudates with the same density, two cellular structures (coarse or fine) can be obtained depending on extrusion conditions. Barrett and Peleg (1992) also showed that the breaking and plateau stresses of corn grits extrudates were negatively correlated with the average cell size. Finally, many cellular solids, including starch-based extrudates and bread crumb, are anisotropic in nature. Anisotropy can be observed either in cell shape or in cell size distribution. Several researchers (Li, Gao, & Sabhash, 2006; Gibson and Ashby (1997), Warburton, Donald, & Smith, 1992) have reported that structural anisotropy affects the resultant mechanical properties of solid foams. Therefore, the correlation between the mechanical properties and the density of extrudates might be improved when the microstructural attributes are taken into account. Bureau and Gendron (2003) proposed that researchers should utilize the ratio of foam density to average cell diameter as a single parameter for relating to the mechanical properties of polyolefin foams, rather than relative density.

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The extrusion process has been widely utilized in expanded cellular solid foods (Cho & Rizvi, 2008). Expanded products using conventional steam-based extrusion usually exhibit non-uniform cellular structure. Typically, steam-based extrudates have an average cell size of 1–3 mm, whereas individual pore diameters can be as large as 6–8 mm (Barrett and Peleg 1992). The polydispersity index, which indicates the uniformity of cell size distribution, of steam-based extrudates is approximately 0.29, suggesting heterogeneous cell distributions (Alavi, Gogoi, Khan, Bowman, & Rizvi, 1999). A novel technology, supercritical fluid extrusion (SCFX) utilizes supercritical CO₂ (SC-CO₂) as a blowing agent instead of steam and allows researchers to produce expanded products with a polydispersity index as high as 0.96 (Alavi et al., 1999). It is also possible to control the macro- and microstructure of SCFX extrudates, such as piece density, cell size, and cell size distribution, by manipulating process conditions, such as pressure drop rate in the die, the ratio of SC-CO₂ to the feed, and SC-CO₂ residence time (Cho & Rizvi, 2008). Although SCFX extrudates have a relatively uniform cell size distribution, they are expected to show a certain degree of anisotropy in microstructure because SCFX extrudates expand more in the cross-sectional direction compared to the longitudinal direction (Sokhey et al., 1996). Research leading to improved knowledge about the microstructure-mechanical property relationship would be beneficial for tailoring textural properties of SC-CO₂ expanded products.

In addition, 3D image analyses like X-ray microtomography provide a better understanding on the relationship between the mechanical properties and the microstructure of expanded cellular foams than 2D image analyses, such as scanning electron microscopy and light microscopy (Trater, Alavi, & Rizvi, 2005). The 2D images are destructive in nature and do not provide accurate information on cell size distribution because cells are generally sliced off-center. Further, conventional 2D images do not allow imaging of the same specimen at different depths. Drawbacks of the conventional imaging technologies can be overcome by using non-invasive techniques that generate 3D maps of the internal structure of small samples with micrometer resolution (Trater et al., 2005). X-ray microtomography was found to be useful for determining the correlation between 3D microstructure and the mechanical properties of cellular solid foods (Agbisit, Alavi, Cheng, Herald, & Trater, 2007).

The purpose of this work was to (1) determine the 2D and 3D microstructures of starch-based SCFX extrudates focusing on cell anisotropy with X-ray microtomography and (2) investigate the effects of the microstructural attributes on the mechanical properties of SCFX extrudates.

2. Materials and methods

2.1. Sample preparation

Full experimental details for SCFX extrudates production were described in our previous paper (Cho & Rizvi, 2009). Whey protein added starch-based SCFX extrudates were produced using a Wenger TX-57 twin screw extruder (Wenger Manufacturing, Sabetha, KS) for 2D and 3D image analysis, and mechanical property measurement. The ratios of pregelatinized corn starch to whey protein isolate (WPI) were 0, 3, 6, 12, and 18 wt%, whereas the ratios of SC-CO₂ to feed were 0.0, 0.25, 0.5, and 0.75 wt%. Emerging extrudates from the die were collected on metal trays and dried at 85 °C in a convection oven to obtain 5–6 wt% moisture content for further analysis.

2.2. Piece density measurement

Piece density (PD) (kg/m³), defined as the ratio of the mass of the sample to its total volume including the voids, was measured

using the geometrical method (Cho & Rizvi, 2008). The volume of the extrudate was calculated by multiplying the cross-sectional area by the length, assuming the extrudate is a straight cylinder. The procedure was repeated 10 times for each set of samples.

2.3. X-ray microtomography

As described in our previous paper, SCFX extrudates were scanned using a desktop X-ray microtomography imaging system (Model 1072, 20–100 kV/0–250 u, SkyScan, Belgium) set at 40 kV/100 μA (Cho & Rizvi, 2009). A microtomographic scan was performed by rotating the specimen at small angular increments. The radiographs were then reconstructed into 3D images using Cone-Rec software (Skyscan, Belgium). Longitudinal images were obtained using Dataview software (Skyscan, Belgium) from reconstructed cross-sectional images. Both cross-sectional and longitudinal images of SCFX extrudates were used for further 2D and 3D image analysis.

2.4. 2D image analysis

Sigma Pro version 5.0 software (SPSS, Inc.) was utilized for 2D image analysis. Prior to the analysis, the thresholding process was conducted with a value of 45. The 2D cell area was equivalent to the sum of calibrated pixels units. In this study, the 2D cell diameter was obtained from the 2D cell area, assuming that each cell is circular, and the average values in cross-sectional (CD_{cross}) and longitudinal (CD_{long}) directions were calculated.

The 2D radial variation of microstructural characteristics was evaluated by dividing each 2D cross-section into eight shells. The 2D cell diameter was obtained from cells in each shell. The average value was presented as a function of the distance from the center. The cell number density in each shell was determined by counting the cell number and expressed as the cell number per shell unit area. Cell elongation in longitudinal directions was characterized by the anisotropy ratio (AR) that was defined as the average cell diameter ratio of cross-sectional to longitudinal directions.

2.5. 3D image analysis

3D microstructure analysis was conducted using Skyscan software (CT-Analyser, version 1.5.0.2, Skyscan, Belgium). A volume of interest (VOI) function, for which the regions of interest (ROI) were interpolated across slices, was used and then segmented into white and black regions. The lower and upper grey threshold settings were 45 and 255, respectively. First, the total volume of SCFX extrudates sample (TV), total volume of cells (objectives, TCV), and total surface area of cells (TCS) were determined and utilized for further analysis. The void fraction (Vf) was calculated by dividing total cell volume (TCV) by total volume (TV). The 3D average cell diameter, 3D cell wall thickness, and 3D cell number density were obtained using three different approaches and compared with the data obtained by manual 3D morphology analysis from our previous paper (Cho & Rizvi, 2009).

2.5.1. Trabecular bone analysis analogy

First, cell diameter (CD_{trabecular}) and cell wall thickness (CWT_{trabecular}) were measured directly by means of their analogues in trabecular bone analysis using CT-Analyser software. Bellido, Scanlon, Page, and Hallgrímsson (2006) successfully utilized trabecular parameters, such as trabecular thickness and trabecular separation, to determine bubble diameter and bubble separation in dough, respectively. In this study, structure thickness and structure separation in trabecular bone analysis were utilized as cell diameter (CD_{trabecular}) and cell wall thickness (CWT_{trabecular}).

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