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Non-destructive, quantitative characterization of extruded starch-based products by magnetic resonance imaging and X-ray microtomography

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ABSTRACT

Magnetic resonance imaging (MRI) was used for non-destructive characterization of the pore structure of starch-based extruded products. The samples were obtained by extrusion cooking of corn meal in a twin screw extruder. The samples were extruded at varying mechanical energy input and, subsequently, the pore size distributions were analyzed. Two different sample preparation methods were introduced and discussed. Negative imaging was the favorable technique. In this method, the pores are filled with cyclohexane and the solvent is detected by MRI. The analysis method was validated by glass beads of known size as reference samples. The pore sizes detected by MRI were slightly higher than the real size of the glass beads. When extruded corn meal samples were analyzed, we were able to show, that despite an equal expansion index, samples can have different pore size distributions. Finally, the pore size distributions were compared to analysis via X-ray microtomography and advantages and limitations of both methods were discussed.

1. Introduction

Starch-based, extrusion cooked and expanded products, such as breakfast cereals or snacks have unique attributes during consumption. Apart from pure taste originated by flavours coated on the finished products, the sensorial quality is dominated by the texture which leads to the specific crunchiness, crispness, firmness and 'mouth feel' of these products (Attenburrow et al., 1989; Duizer, 2001; Luyten et al., 2004). These attributes are based on mechanical properties which are mainly influenced by bulk expansion and porosity (Meuser et al., 1982). Porosity is governed by pore size and shape and by cell wall thickness and mechanical properties of the matrix (Harper, 1986; Mercier and Feillet, 1975).

While many investigations focus on determining the overall expansion (Della Valle et al., 1997; Brummer et al., 2002; Alvarez-Martinez et al., 1988), less attention was paid to the characterization of the inner structure of extruded samples. Quantitative characterization of pores was performed by Warburton et al. (1990). The authors presented pore size distributions using 2-dimensional (2D) image analysis of cross sections of the samples. Images were obtained by a camera with a macro-lens, while 100 cells were evaluated for each pore size distribution. In a similar study, Sapirstein et al. (1994) estimated the pore size distribution of bread crumb grain by 2D images. However, it is not possible to analyze extruded starch based products such as snacks or breakfast cereals by conventional 2D imaging or scanning methods of the surface area without destroying the structure. Cutting them with a simple blade leads to cell wall breakage due to their brittle texture, and the pores are squeezed before image acquisition and analysis. In addition, the acquired 2D images provide only the cross section (area) of each pore. An extrapolation into the third dimension is required, which is often realized by assuming a certain shape of the pores.

In order to avoid these constraints non-destructive methods such as X-ray tomography (μCT) were applied for the characterization of cereal products (Trater et al., 2005; Agbisit et al., 2007). In spite of having a non-destructive method for characterizing the inner structure in three dimensions, the evaluation was based on 2D slices from the acquired data. Babin et al. (2007) investigated the pore diameter and cell wall thickness of extruded samples by image analysis of slices obtained by X-ray tomography. To evaluate the pore volume directly algorithms were developed for 3D data analysis (Baldwin et al., 1996). However, a proper validation of the data processing methods is challenging, and the necessary amount of samples or cells for a statistically relevant and representative result often remains unconsidered. In addition to μCT, magnetic resonance imaging (MRI) was used for real time visualization of moisture diffusion effects in hydration processes such as cooking of foods (Mohoric et al., 2004; Ramos-Cabrer et al., 2006).

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MRI as a method for non-invasive analysis of pore structure in an equilibrated porous system e.g. bread was used by Regier et al. (2007).

In this work, MRI is introduced as alternative method for the quantitative characterization of the pore size distribution of extruded, expanded products. Corn meal, which was extruded by a twin screw extruder, is used as a model system. An algorithm for 3-dimensional pore data analysis is applied. The accuracy and the reproducibility of MRI data are validated by using glass beads as a reference sample. The resulting pore size distributions are compared to data obtained from μCT. Both methods, MRI and μCT, are in principle non-destructive; the samples remain available for other examination techniques afterwards. In addition, the data processing algorithms used facilitated a quantitative evaluation of cell wall thickness in μCT data.

2. Material and methods

2.1. Raw material

Coarse corn meal, delivered by BÄKO, Germany, was used as raw material for sample preparation by extrusion. The median particle size was 350 μm. The moisture content of corn meal was determined to 12% wet basis prior to extrusion. Starch, protein and fat content were about 58%, 8% and 3.5%, according to manufacturer's data sheet, respectively.

For examination of the MRI and the pore size determination algorithm, glass beads with defined size were used. Therefore, they were dispersed in cyclohexane and examined via MRI. A first fraction of smaller glass beads had a volume of 0.008 up to 0.065 mm^3. The volume was calculated from the diameter which was measured by calliper on 25 particles. The second fraction of large glass beads had volumes of 30–35 mm^3.

2.2. Sample preparation

The samples were prepared by extrusion of the corn meal in a co-rotating twin screw extruder ZSK 26 Mc by Coperion Werner & Pfleiderer, Germany. The process parameters mass flow rate and screw speed were varied to change the specific mechanical energy input (SME) and subsequently obtain samples of different sectional expansion index (SEI) and longitudinal expansion index (LEI) (Table 1). The SME was calculated according to the description of Meuser et al. (1982) and is indicated in W h/kg. SEI is defined as the ratio of extrudate cross-sectional area and the area of the die orifice. LEI is the ratio of extrudate velocity after leaving the die and melt velocity in the die (Alvarez-Martinez et al., 1988).

After extrusion the starch-based products had moisture contents below 11%. They were prepared for MRI analysis applying the two different methods of positive and negative imaging. As these products are hard materials with short transverse relaxation times and low moisture content, conventional MRI methods fail. With the aim of high spatial resolution in 3D, also solid state MRI was not applicable properly. In order to detect the pore walls directly (positive image) despite these facts, the samples were saturated with moisture under defined atmospheric conditions. An example of the magnetic resonance image obtained is shown on the left side in Fig. 1. The brighter regions are illustrating the pore walls. According to the water sorption isotherms for corn meal reported by Horvat et al. (2013) moisture contents between 11% and 24% were achieved by storing the samples at relative humidity between 50% and 97% at a temperature of 30 °C for 10 days. The second analysis method, the negative imaging, aimed for detecting the voids within a pore. Therefore, pore walls had to be kept dry and to make them undetectable by conventional MRI. An example is shown on the right side of Fig. 1. The dark areas depict the pore walls in this case. In our study, the void area within the pores was then detected by filling them with cyclohexane by Carl Roth GmbH & Co. KG in a vacuum chamber. In contrast to air, cyclohexane can easily be detected by MRI. To increase the imaging quality within a given measurement time, 4Hydroxy-TEMPO, Sigma–Aldrich Chemie GmbH, was added to cyclohexane. TEMPO is a paramagnetic molecule with an unpaired electron spin and therefore is able to enhance the proton relaxation rate R_1. The examination of the method and data processing was also performed with the cyclohexane-TEMPO-mixture. Therefore, a sample container was filled with glass beads and cyclohexane-TEMPO-mixture.

2.3. 3D-data generation by MRI

The experiments were performed on an MRI tomograph (Bruker Avance 200 SWB by Bruker BioSpin GmbH). The superconducting magnet has a magnetic-flux density of 4.7 T and a 150 mm vertical bore. The Bruker gradient system micro2.5 was used. The ^{1}H NMR bird-cage (25 mm inner diameter) was equipped with a polytetrafluorethylene sample holder. Nuclear magnetic resonance (NMR) pulse sequences were used as provided by Bruker within Paravision 4.0. The radiofrequency pulse for a rectangular 90° flip angle was 45 μs at 0 dB. The shaped pulses, used in the imaging sequences, were sinc-pulses with 1 ms for excitation (14 dB attenuation). For refocusing, a bandwidth matched sinc pulse with 0.749 ms (5.5 dB attenuation) was applied. The 3D proton density distribution was measured by a RARE sequence, applying an echo time of 6.4 ms and a recycle delay of 1.5 s. The quantitative analysis of the images requires a good signal to noise, leading to the acquisition of 256 scans. The field of view had a typical volume of 20 × 20 × 20 mm^3, which was divided into 128^3 volume elements (voxels). Thus, every voxel included the signal amplitude for a volume of 3.815×10^{-3} mm^3 (156 μm × 156 μm × 156 μm). The measurement time for this resolution was 8–13 h per sample. A higher resolution is applicable, but would drastically increase the measurement time.

2.4. 3D-data generation by X-ray microtomography

The samples were measured with a commercially available cabinet cone-beam microCT, (μCT 50, SCANCO Medical AG, Brüttisellen, Switzerland). It operates with a cone beam originating from

Table 1
Process parameters screw speed, mass flow rate and specific mechanical energy SME varied during extrusion processing of samples and resulting expansion indices SEI and LEI with their standard deviation.

sample	screw speed (rpm)	mass flow rate (kg/h)	SME (W h/kg)	SEI		LEI	
1	800	40	163	18.63	±1.69	0.69	±0.03
2	1800	40	197	12.90	±0.99	1.58	±0.14
3	1200	30	199	12.29	±1.77	1.43	±0.02
4	1800	30	247	12.01	±1.62	2.51	±0.11
5	1400	60	158	16.31	±0.93	0.99	±0.04
6	1800	60	170	12.43	±0.98	1.12	±0.18