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The impact of freeze-drying on microstructure and rehydration properties of carrot

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ABSTRACT

The impact of freeze-drying, blanching and freezing rate pre-treatments on the microstructure and on the rehydration properties of winter carrots were studied by μCT, SEM, MRI and NMR techniques. The freezing rate determines the size of ice crystals being formed that leave pores upon drying. Their average size (determined by μCT) can be predicted in a quantitative manner by considering dendritic growth and freezing rates. Blanching as a pre-treatment, however, did not affect pore size distribution induced by freeze-drying. Upon rehydration of the freeze-dried carrots, PFG NMR and MRI show that cellular compartments were not restored and instead a porous network with permeable barriers is formed. Blanching pre-treatment introduced a less connected and more anisotropic porous network if followed by fast freezing, indicating that more of the native cell wall morphology is preserved.

1. Introduction

Consumers have a high appreciation for fruits and vegetables, which are an important dietary source of vitamins, phytochemicals, fibers and minerals (Hoffmann, Boeing, Volatier, & Becker, 2003). The intake of fruits and vegetables has been associated with a wide range of beneficial health effects (Pomerleau, Lock, & Mckee, 2006). A main hurdle for consumers to raise their daily intake is the lack of convenience in preparing meals. The food industry has addressed this by offering the consumer dried fruits and vegetables, which are rehydrated shortly before consumption. A major obstacle for further growth in this area is the relative poor quality of the rehydrated fruits and vegetables in the product after preparation. Another bottleneck is the poor compromise between convenience in meal preparation and textural quality (Jangam, 2011; Prothon, Ahrne, & Sjoholm, 2003).

Most dehydrated fruits and vegetables are produced by air drying. A disadvantage of this method is a substantial degradation in quality, including appearance (shrinkage, drying-up, darkening), nutrients, flavor, and the low rate of rehydration (Devahastin & Niamnuy, 2010; Ratti, 2001). Higher quality products can be obtained using more expensive freeze-drying methods (Mujumdar & Law, 2010). Freeze-drying involves crystallization of water in ice crystals, which subsequently sublimate, thus leaving a porous dried product. This may lead to loss in texture and an increase in friability (Brown, 1976; Chassagne-Berces et al., 2009; Ratti, 2001; Van Buggenhout et al., 2006). Improvements in the freeze-drying process of foods have been driven by engineering, where technologies are being optimized to balance rehydration rate and final texture (Mujumdar, 2011; Sagar & Kumar, 2010). Considering the underlying microstructure and its role in rehydration may enhance the efficiency and rate of process innovation (Mebatsion, 2008). A major barrier to embark on such an approach has been the lack of adequate quantitative measurement technologies that enable decision making based on sound microstructural data. Hence we embarked on an approach where we quantitatively assessed microstructural features of freeze-dried carrots as a model system.

In this work the impact of thermal pre-treatments and freeze-drying on the microstructure of the cortical tissue of winter carrots was investigated. The purpose of this investigation was to quantitatively describe the features of dry and rehydrated microstructures by means of dedicated image analysis and NMR parameters. To achieve this, a suite

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of imaging techniques in combination with NMR relaxometry and diffusometry tools was employed. In order to visualize the microstructure of the dried carrots at the μm–mm level X-ray computerized tomography (μCT) and scanning electron microscopy (SEM) were used. μCT allows for high-resolution 3D visualization and characterization of the dried and hydrated material (van Dalen, Notenboom, van Vliet, Voortman, & Esveld, 2007). μCT can probe the microstructure of samples non-invasively with an axial and lateral resolution down to a few micrometers and a field of view of up to a few mm under environmental conditions. Time-domain NMR and MRI were used to assess mobility of water in rehydrated samples in a non-invasive manner. Compartment integrity and tissue permeability in plant materials have been studied by 2D relaxometry and MRI. Time-domain NMR relaxometry (van Duynhoven, Voda, Witek, & As, 2010) has already been used to study freeze-drying of carrots (Hills & Nott, 1999). Relaxometry could detect sublimation of the frozen core and removal of non-frozen water during freeze-drying. A similar approach was used to study osmotic dehydration of apple (Cornillon, 2000).

To obtain an indication if one has good control over the microstructure via process conditions of the freezing step during freeze-drying, the size of the pores of the freeze-dried samples, as obtained by image analysis, was compared with scaling rules for ice crystal size induced by dendritic growth. The process conditions were characterized by the heat transfer coefficient of the coolant and the freezing (coolant) temperature, which can be reformulated in terms of freezing rate. In our comparison we also used literature data of ice crystal size growth in comparable materials at different freezing rates.

2. Materials and methods

2.1. Materials

The carrots used in the current study were of the winter carrot type purchased in a local supermarket, having sizes of 3 to 6 cm in diameter and a length of 20 to 30 cm. The carrot root consists of two distinct layers: a central stele and a peripheral cortex. The anatomy of the carrot root is greatly affected by the growth of the stele, hence four different regions can be distinguished as shown in Fig. 1: three inner regions belonging to the stele (vascular tissue) and the outer region which is the cortex. Cylindrical samples with a diameter of 8 mm and a length of 10 mm were cut as it is shown in Fig. 1. The selected tissue consists of cells with a diameter of 20 to 100 μm, depending on the age of the carrot.

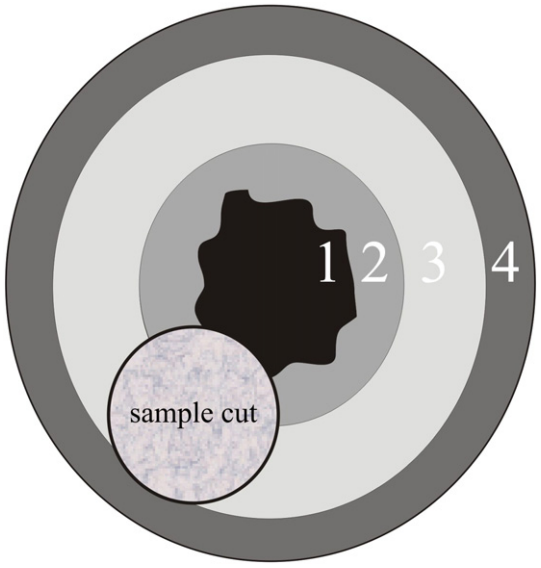

Fig 1. Schematic representation of the four distinct tissue regions of a winter carrot: 1—parenchyma cell type, 2—parenchyma cells and xylem vessels, 3—outer cells of the vascular tissue and phloem vessels, and 4—cortex tissue. The disk indicates the sampling spot for the cylindrical samples employed in this work.

2.2. Pretreatments and freeze-drying of carrots

Certain thermal pretreatments were applied to the carrot samples before freeze-drying. The first pretreatment was blanching for one minute in boiling water. Nonblanched samples were also selected for the next step. Secondly, samples were frozen at four different temperatures: −28, −80, −150 and −196 °C. The freeze-drying protocol consisted of time-incremented temperature steps from −30 °C up to 25 °C at a constant low pressure of 0.4 mbar. The time needed to complete this process was about 27 h, see Table S1 in the supporting information for full details.

2.3. Scanning electron microscopy (SEM) of freeze-dried carrots

A piece of dried carrot was cut into two halves in such a way that a cross-section was obtained. A very thin slice was cut off from the surface with a razor blade to obtain a high quality cross-sectional surface of the remaining piece of dry tissue. This surface was sputter coated with platinum for better SEM imaging quality. The Pt coated sample was inserted into a Jeol 6490 LA scanning electron microscope and both the peripheral and central areas were imaged at magnifications ranging from 10× to 1000×.

2.4. X-ray microscopic computerized tomography (μCT) of freeze-dried carrots

The internal porous structures of the samples were visualized using a SkyScan 1172 desktop X-ray micro-tomography system (Belgium, http://www.skyscan.be). The X-ray tube was operated at 39 kV and 248 μA. μCT is a non-invasive method for the reconstruction of a three-dimensional volumetric image based on a large number of two-dimensional projections of a sample under different angles. The sample was imaged in a plastic cylindrical sample holder with an inner diameter of 11 mm. A stack of 2481 flat cross sections (4000×4000 pixels) was obtained after tomographic reconstruction of projection images (4000×2096 pixels). These projections were acquired under different rotations over a 180 degree interval with a step size of 0.20° (frame averaging = 2). The acquisition time for one projection was 589 ms (exposure) resulting in a total acquisition and read-out time per scan of about 50 min (1019 projection images/scan). For tomographic reconstruction the following settings were used: no smoothing, ring artifact correction = 20 and beam hardening correction = 40%. A pixel size of 4.0 μm was selected. The samples were scanned using two scans connected in the vertical direction to increase the axial field-of-view (oversized scan) and subsequently merged together during reconstruction.

2.5. Nuclear magnetic resonance (NMR) of rehydrated carrots

Carrot samples were rehydrated in water at 95 °C for 15 min. After rehydration, the samples were gently rolled on an absorbent paper tissue to remove the water dripping over the surface, and wrapped in cling-film to prevent moisture loss during measurements. Time-domain NMR relaxation experiments were carried out at room temperature (22 °C) on a Maran (Resonance Instruments, Oxford, UK) spectrometer operating at a proton frequency of 30 MHz. Transverse relaxation times were measured using a Carr–Purcell–Meiboom–Gill (CPMG) pulse sequence with echo-times (TE) of 400 μs and a repetition time (TR) of 5 s. Data were averaged over 64 acquisitions. T_2 distributions were calculated as a continuous distribution of exponentials by means of the CONTIN software (Provencher, 1982), as well as a discrete analysis assuming a sum