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INVESTIGATION ON THE INFLUENCE OF PRE-TREATMENTS ON DRYING BEHAVIOUR OF BROCCOLI BY MRI EXPERIMENTS

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Abstract: Magnetic Resonance Imaging (MRI) allows the monitoring of internal moisture content of food products during drying non-destructively. In an experimental set-up with continuous and controlled hot air supply, the internal moisture distribution of broccoli with different pre-treatments are measured during drying. Moisture distribution, drying rate and shrinkage are compared and analyzed quantitatively. MRI results indicated that for fresh broccoli stalks the moisture content in the core of the sample increased after some hours of drying. With pre-treatments as peeling, blanching or freezing the moisture transport barrier in the skin of the broccoli sample was reduced. Shrinkage was uniform for most of the pre-treated samples and the moisture increment in the core did not occur. It was also found that with these pre-treatments progress of drying enhanced significantly. Therefore, from an drying efficiency and economic point of view, pre-treatments prior to drying offer important opportunities.

Keywords: MRI, hot air drying, broccoli stalk, increased moisture content, pre-treatments

INTRODUCTION

With the increasing consumers demands for food convenience, processed foods have a growing contribution to the daily food intake. As a result the need for high quality processed food increases. Consumers have become more critical on food quality; not only on appearance and taste but also on the nutritional value. Bioactive components like antioxidants have an important role because of their potential to reduce cancer. For example, lycopene in tomatoes contribute in the reduction of prostate cancer, lung cancer, breast cancer and a broad range of epithelial cancers (Shi et al., 2000, Goula et al., 2006, Chang et al., 2007, Dewanto et al., 2002). Glucosinolates, available in *brassicaceae* (cabbage, broccoli, cauliflower), have also significant anticarcinogenic properties, for example colorectal cancer (Verkerk et al., 2004, Verkerk et al., 2009, Volden et al., 2009, Cieslik et al, 2007).

To realize a long shelf life, vegetables used in processed foods are often dried. Freeze drying and convective drying are most used methods. Convective drying can be done in continuous operations, but due to the heat load the product attributes may change (texture, taste, bioactivity of nutritional components). Freeze drying has potential to retain the qualities the best, but is a batch process which is cost and labour intensive.

Convective drying of vegetables is mostly considered as diffusion controlled process (see for example Mulet et al., 1999). According to Fick's law, diffusion results in a moisture gradient with the highest moisture contents in the centre and the lowest at the edge of the product particles. To investigate the effect of the pre-treatments on the drying behaviour, it is essential to monitor the internal moisture distribution and shrinkage instead of the average values.

With traditional methods, it is difficult to achieve this goal. Till now, the internal properties have been examined with destructive methods (e.g. by taking slices from the sample) or non-destructive methods (e.g. γ ray densitometry). Various disadvantages arise from the limitation of the sample size, like a limited resolution or the results concern only one dimensional direction (McCarthy et al., 1991, Ruiz-Cabrera et al., 2005, Chen, 2007). Magnetic resonance imaging (MRI) offers an alternative to study the internal behaviour in food products.

Drying is not the only processing step for vegetables in convenience food. Pre-treatments precede the drying step and influence the drying pattern and product attributes as well (Tunde-Akintunde, 2010, Severini et al., 2005, Prajapati et al., 2011, Negi et al., 2000). For example, fresh vegetables are blanched to reduce the enzyme activity. Common indicators are peroxidase or lipoxygenase (Severini et al., 2005, Barrett et al., 2000, Orak, 2006, Nagy-

Gasztonyi et al., 2000, Sanjuan et al., 2000), or cysteinelyase (Ramirez et al., 1999). Freezing is applied to break the structure. Due to the changes in product elasticity and cell structure these pre-treatments affect the drying process and the final product significantly (Waldron et al, 2003, Okuzono et al., 2008).

This work concerns an investigation on the effect of pre-treatment methods prior to drying on the drying behaviour and shrinkage of broccoli stalks. The experiments are done in a MRI device with continuous and controlled hot air supply; moisture transport and shrinkage are recorded simultaneously during drying. The drying patterns of the samples under different pre-treatments are compared; drying rates and shrinkage are analysed quantitatively.

MATATERIALS AND METHODS

Materials

For all measurements broccoli was cut in pieces and parts of the stalk were used. The sizes of the samples were about 0.01 m in height, 0.01 m in radius. Figure 1 gives an example of a fresh broccoli sample.

Figure 1 Cross section of a broccoli stalk sample

Pre-treatments

In total six different pre-treatments were applied. An overview of the samples and pre-treatments is given in Table 1. After all pre-treatments, the free water at the sample surfaces was removed at room temperature with tissue paper before further processing

Drying chamber

The sample was fixed by a stick on a sample supporter and inserted into a drying chamber in the MRI measurement device. The size of the drying chamber was 0.032 m in diameter and 0.2 m in length. A continuous flow and temperature controlled air was supplied. The air temperature was respectively 30°C and 50°C, the air velocity 1.0m/s and the relative humidity 10%.

MRI imaging equipment

All measurements were performed on a 3 T (128 MHz for protons) MRI system (Bruker, Karlsruhe, Germany), consisting of an Avance console, a superconducting magnet with a 0.5 m vertical free bore (Magnex, Oxford, UK), a 1 T/m gradient coil, and a birdcage RF coil with an inner diameter of 0.04 m

Table 1 Overview of pre-treatments and experiments

Pre-treatment	procedure
Peeling	1.0mm-1.5mm skin was removed
Blanching	90°C water, 3 minutes
Freezing	-25°C, 48 hours
Experiment	pre-treatment
1.	Non treated
2.	Peeled
3.	Non-peeled, blanched
4.	Peeled, blanched
5.	Non-peeled, frozen
6.	Peeled, frozen

MRI imaging

3D images were obtained using a Turbo Spin Echo (TSE) MRI sequence (Scheenen et al., 2000), a repetition time TR of 2 s, an effective spin echo time TE of 3.35 ms and a spectral bandwidth SW of 50 kHz. Only 16 echoes were acquired in the TSE train to avoid T2-weighting. Odd and even echoes were separately phase encoded forming two different images to avoid Nyquist ghost's artifacts, so the turbo factor was 8. Two acquisitions were averaged to improve image quality. The Field-Of-View (FOV) was $35 \times 35 \times 35$ mm3 with a matrix size of $64 \times 64 \times 64$ resulting in a spatial resolution of $0.55 \times 0.55 \times 0.55$ μm3. The interval time between measurements was 34 minutes.

T2 mapping was done using a Multi Spin Echo (MSE) imaging sequence (Edzes et al., 1998), a TR of 2 s, a TE of 3.59 ms and a SW of 50 kHz. Per echo train 128 echoes were acquired; 16 acquisitions were averaged to improve image quality. The FOV was 35×35 mm2 with a matrix size of 64×64 resulting in an in-plane resolution of 0.55×0.55 μm2. The slice thickness was 3 mm. The interval time between measurements was 34 minutes.

Numerical methods and data analysis

MRI-measurement data handling and analysis was performed with home-built software written in IDL (RSI, Boulder, CO).

A linear relationship was applied to interpret the MRI signal intensity to moisture content. The final moisture contents were determined by the oven method (105°C, 24 hours).

For shrinkage calculations pixels with an intensity value higher above 0.75 were counted and for each

pixel a volume of 0.16mm3 was assigned. By summing up the volume of all counted pixels the sample volume was calculated.

RESULTS AND DISCUSSION

Drying pattern of fresh broccoli stalks

Drying was continued until the moisture content of the samples was constant. Depending on the material properties, the experimental time for the fresh broccoli stalks ranged from 12 to 48 hours. Figure 2 presents the T2 measurements for the central cross section of fresh broccoli samples dried at 30°C and 50°C. Differences in brightness indicate the distribution of moisture throughout the sample; the brighter the colour, the higher the moisture content. The bar in the figures provides a relative scale for the moisture content. The boundaries of each sample image in Figure 2 give the information on the current sample size and from this information shrinkage is derived. Drying at 50°C is faster than drying at 30° and therefore the top figure contains more images compared to the bottom one

Because of the low moisture content in the dry stage, the intensity of the sample images is weak and the image is difficult to interpret. However, the intensity was still monitored and gives also sufficient information about the progress of drying in the late stage. The MRI signal for T2 and 3D intensity for wet products is linear with signal intensity, but below a certain moisture content, the intensity and T2 signal is non-linear (McCarthy et al., 1991, Reis et al., 2003, Chen, 2006).

In diffusion driven drying, moisture diffuses from the center of the product to the surface. First, a moisture gradient is developed from the center to the surface and later the moisture concentration in the center decreases gradually. Figure 2, however, shows a different behavior; after some hours of drying the moisture in the center increases (compare for 30°C drying the brightness of row 1 and row 2 with row 3, and for 50°C the last samples of row 1). The observed redistribution of moisture shows that drying of broccoli stalks is not a standard diffusion process. Johnson et al. (1998) reported the similar phenomena but without further explanation.

Despite the increasing moisture content in the centre of the product, the drying curves for the full samples which are given in Figure 3a show monotonic decreasing moisture content.

The drying behaviour of broccoli stalks is consequence of the structure of broccoli stalks which is shown in Figure 1. Figure 1 shows that the internal structure of broccoli contain two parts: the core and skin. The moisture content of the core and skin are different; the moisture content is 11.2 kg water/ kg dry matter while the core contains around 16.5 kg water/ kg dry matter. The surface with a low initial moisture content dries faster and reaches the glassy state after a relative short time and the rigid glassy structure forms a barrier to moisture transport (Kumar et al., 2001, Hu et al. 2006, Leeratanarak et al., 2006 and Hiranuarachat et al., 2011). In the meantime shrinkage of the particle increases the internal stress, which results in stress driven moisture transport towards the centre of the product.
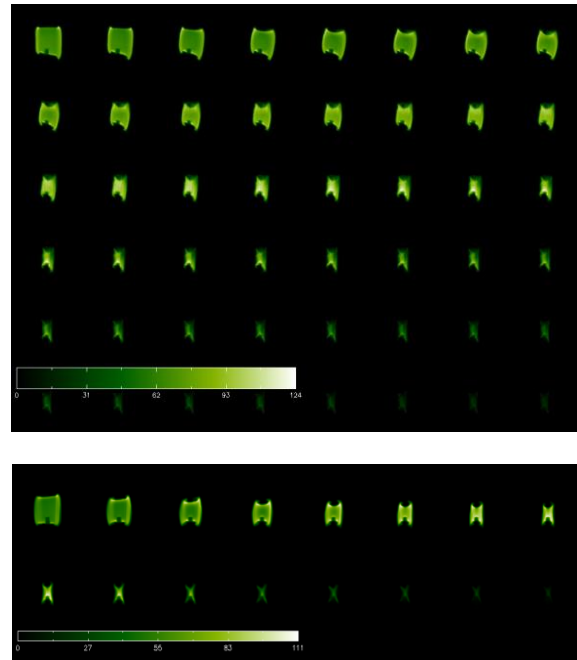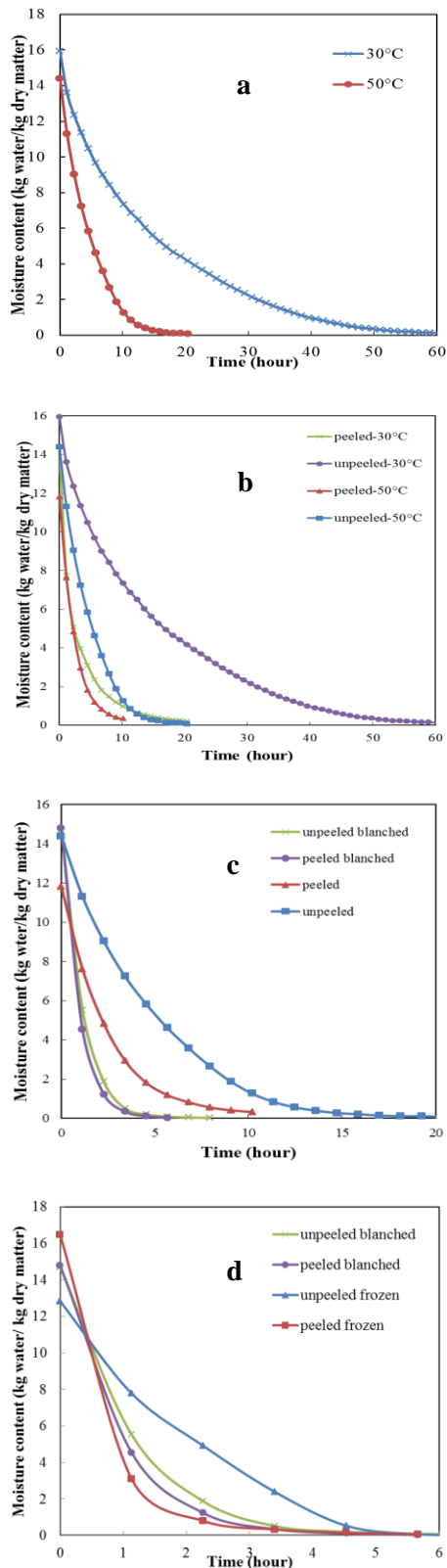

Figure 2 Series of MRI intensity of the middle slice of fresh broccoli samples in time. Top: drying at 30 °C; Bottom: drying at 50 °C.

Drying patterns after pre-treatments

Peeling

To verify the role of the skin in the increasing moisture content of the product centre, broccoli stalks were peeled prior to drying. The results for two drying temperatures are shown in the images of Figure 4. Compared to Figure 2 the increased moisture content hardly occurs which confirms the transport barrier of the skin. The first images also show an increased moisture content at the surface, which is result of moisture release at the cutting surfaces.

The drying curves for the peeled samples are compared with the drying curves for the no-peeled samples in Figure 3b. The removal of the mass transfer barrier enhanced drying both at 30 and 50°C drying (from 48 to 20 hours and from 15 to 10 hours respectively).

Figure 3 Drying curves of fresh broccoli stalks at different drying temperatures. a: fresh samples at 30 and 50°C drying; b: peeled fresh samples at 30 and 50°C drying; c: peeled blanched samples and unpeeled blanched samples dried at 50°C; d: peeled frozen samples and unpeeled frozen samples dried at 50°C.
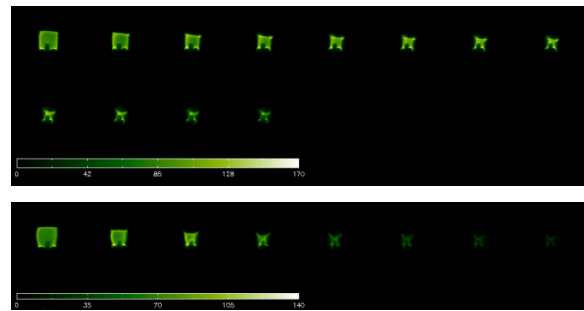
Figure 4 Series of MRI intensity of the middle slice of peeled broccoli stalks in time. Top: drying at 30 °C; Bottom: drying at 50 °C.

Blanching

Blanching results in tissue softening and can level out the differences in mechanical properties between core and the skin (Hiranuarachat et al., 2011). For the blanched samples only a small difference in drying behaviour between the peeled and unpeeled sample is found in Figure 3c and Figure 5. So, the barrier for mass transport by the skin is removed by blanching. Not only the structure of the skin is softened, but also the internal matrix is softened by blanching. Blanching enhances therefore also drying of the internal mass. The drying time is reduced to about 5 hours.
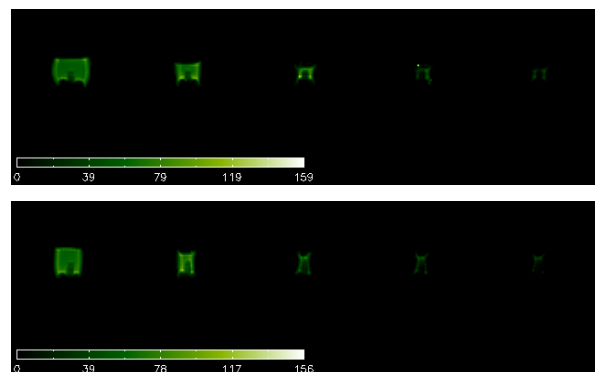
Figure 5 Series of MRI intensity of the middle slice of blanched broccoli stalks in time. Samples are dried at 50°C. Top: fresh unpeeled sample is blanched before drying Bottom: sample first peeled then blanched before drying

Freezing

During freezing ice crystals are formed in the tissue. Upon thawing individual ice crystals merge into large complexes which both break the structure and increase the pore size. Results for peeled and non-peeled samples are given in Figure 3d and Figure 6 . For both peeled and non-peeled samples, the change in the structure due the treatment advances drying significantly and drying is completed within 6 hours.

Although the surface water was removed before drying, the highest moisture content was detected at

the surface of the non-peeled sample. The skin remains a mass transfer barrier and causes moisture accumulation in the skin. Because of the mass transfer barrier, moisture removal occurs in longitudinal direction. The barrier for mass transport is absent for the peeled sample and therefore the moisture content in the core and boundary layer does not increase.

The drying curves for the blanched and frozen samples are compared in Figure 3d. The frozen peeled sample dried the fastest and the non-peeled frozen sample is the slowest. The blanched samples are in between these curves. The change of structure in the skin for the blanched sample is more effective than that for the frozen sample. The results for the peeled samples show that although blanching destroys the internal cell walls moisture transport is more enhanced by the growing pore size during freezing.
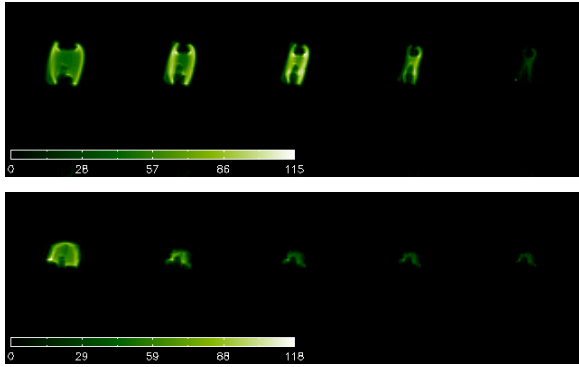
Figure 6 Series of MRI intensity of the middle slice of frozen broccoli stalks in time. Samples are dried at 50°C. Top: fresh sample frozen before drying Bottom: sample first peeled then frozen before drying

Shrinkage patterns for fresh and pre-treated products

Removal of moisture creates in a first instance voids in the product. For products above the glass transition temperature, the internal stress result in collapse of voids and subsequently in shrinkage of the product. The degree of shrinkage is defined as the ratio between the lost volume compared to the initial value:

$$S = \frac{V_0 - V_i}{V_0} \qquad (1)$$

The fraction of moisture removed from the initial product is defined as:

$$X = \frac{M_0 - M_i}{M_0} \qquad (2)$$

The MRI images in Figure 2, Figure 4, Figure 5 and Figure 6 all show the decreasing size of the samples due to drying. Shrinkage data obtained from these figures is given in Figure 7 and plotted as a function of the fraction of removed moisture.

Figure 7a shows for the fresh samples that below X=0.85 shrinkage is linear with the fraction of removed moisture. In the last phase of drying (X>0.85) shrinkage is no longer linear with the fraction of removed moisture. The product is close to the glassy state and therefore it was expected that shrinkage would be retarded (i.e. the degree of shrinkage changes hardly while removing moisture). The results show the opposite which is result of the accuracy of the MRI-signal in this phase of drying. We work on an extended calibration procedure to increase the accuracy in this phase.

The line in Figure 7 corresponds the situation where shrinkage is equal to moisture removal. Data above the line indicate that shrinkage is faster, and below the line shrinkage is slower than moisture removal. In Figure 7a the data points for drying at 50°C are above that for drying at 30° which indicates that shrinkage at 50° is faster than at 30°C. The samples dried at 50° are more elastic as they are less close to the glass transition temperature than the samples at 30°C.

MRI images in Figure 2 and Figure 4 show different forms for shrinkage during drying. The skin of the broccoli is stiff and close to the glassy state and therefore deformation of the skin is retarded. As soon as the skin forms a significant and stiff barrier for moisture transport, moisture removal takes place from the top and bottom of the sample. It results in an early stage of drying in a "butterfly" shape. The peeled samples keep their original form for a long time, but towards the end of drying when the edge of the product approaches the glassy state, these samples also end with the "butterfly" shape.

Figure 7b presents the degree of shrinkage as a function of the fraction of removed moisture for the peeled samples at 30 and 50°C. For a fraction of removed moisture below 0.85 shrinkage is linear to the fraction of removed moisture. Despite the differences in moisture removal rate (see Figure 3b) for the two drying temperatures shrinkage is nearly equal for the two drying temperatures and the differences are less than for the non-peeled products in Figure 7a.

The cell structure of skin and core of the blanched samples is significantly softened and results in uniform mechanical properties. As a result the form of the peeled an non-peeled samples in Figure 5 are nearly equal. The drying rate of both samples is also nearly equal (see Figure 3c), but the shrinkage of the peeled blanched sample is below that of the non-peeled sample (Figure 7c). Again, for a fraction of removed moisture below 0.85 shrinkage is nearly linear to the fraction of removed moisture.
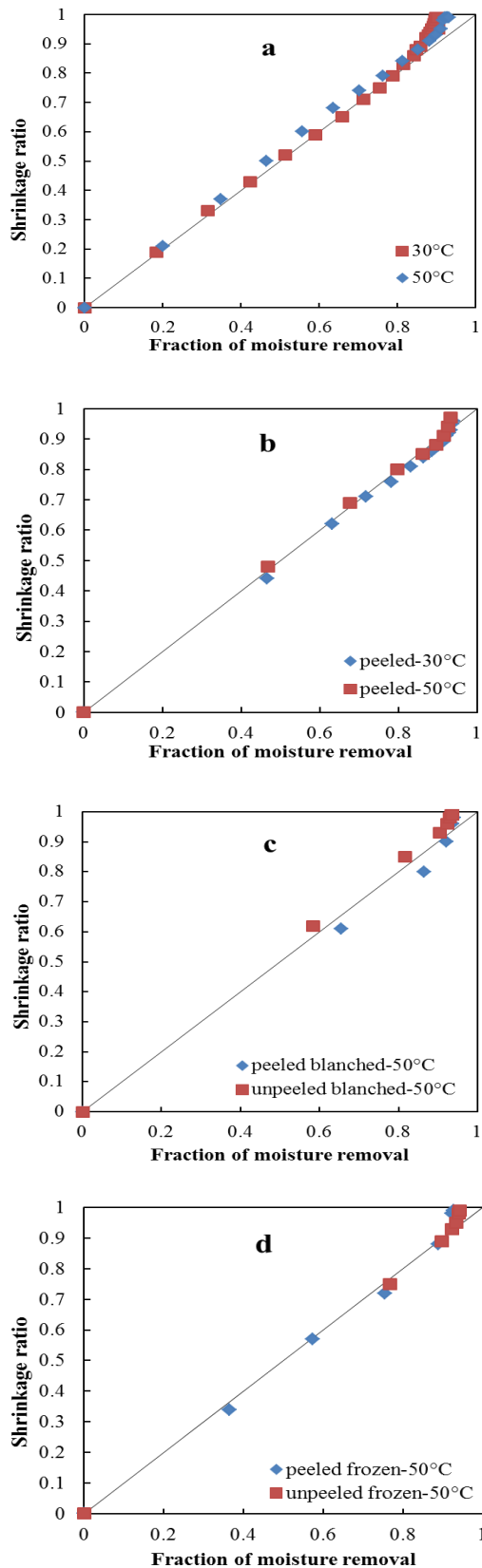
Figure 7 Shrinkage as a function of the fraction of moisture removal. a: fresh samples at 30 and 50°C drying; b: peeled fresh samples at 30 and 50°C drying; c: peeled blanched samples and unpeeled blanched samples dried at 50°C; d: peeled frozen samples and unpeeled frozen samples dried at 50°C.

Ice crystals formed during freezing form larger ice crystal complexes at thawing. The structure of the product is damaged and creates also large pores. Due to the internal stress the product shrinks easily during drying. The images in Figure 6 show a strong "butterfly" form for the non-peeled frozen sample. Drying occurs mainly along the top and bottom of the sample; the skin is still a significant barrier. The peeled frozen sample dries uniformly and the shape remains during drying. For both samples shrinkage was equal to the volume of lost moisture (see Figure 7d)

CONCLUSION

MRI is used to measure the internal moisture distribution, the drying curves and shrinkage of broccoli stalks during drying. Drying results of fresh products and pre-treated products (peeled, blanched and frozen) are presented.

Images from the internal moisture distribution show that fresh broccoli stalks do not dry uniformly. The skin of fresh product forms a significant barrier for moisture transport. Drying of fresh products does not follow the Fick's law for diffusion. It was even observed that the moisture in the centre of the product increased. Therefore, we believe that drying models for fresh products should be extended by additional terms for moisture transport caused by stress diffusion.

Blanching as a pre-treatment softens the skin and core of the product and creates uniform properties throughout the material to be dried. In this case drying can, indeed, be considered as a diffusion driven process. Freezing and subsequently thawing as pre-treatment of fresh broccoli does not change the non-uniformity of the product. Drying is not a diffusion driven process. Removing the skin of the broccoli stalks by peeling results in a uniform product with drying behaviour that is close to diffusion driven drying. Pre-treatments as blanching, freezing and peeling all enhance the drying rate significantly.

The MRI data allowed to monitor shrinkage expressed as the relative change of volume and change of moisture of the product during drying. The data showed that shrinkage is linear to the amount of moisture removed from the product up to about 85% removed moisture, after which it is nonlinear. The data also indicated that the higher drying temperature resulted in faster shrinkage.

MRI images and data were essential to reveal the internal patterns during drying of broccoli. In our work we didn't consider the effects of pre-treatments for other vegetables. MRI is a very good method to

reveal the drying patterns in other vegetables and products.

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