

11<sup>th</sup> International Congress on Engineering and Food (ICEF11)

## Moisture distribution in broccoli: measurements by MRI hot air drying experiments

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### Abstract

The internal moisture distribution that arise in food products during drying, is a key factor for the retention of quality attributes. To reveal the course of moisture content in a product, internal moisture profiles in broccoli florets are measured by MRI imaging during drying experiments with controlled air flow and temperature. The 3D images concern a matrix size of 64×64×64 elements. Signal intensity is converted to product moisture content with a linear relationship, while taking a minimum detectable moisture content of 0.3 kg water/ kg dry matter into account. Moisture content as a function of time is presented for a 2D cross sectional area in the middle of a broccoli sample. The average moisture contents for the cross sectional area obtained from the MRI imaging are compared with spatial model simulations for the moisture distribution. In that model the effective diffusion coefficient is based on the Free Volume Theory. This theory has the advantage that the changed mobility of water in the product during drying is taken into account and the theory also predicts the moisture transport in the porous broccoli floret. Key parameters for the Free Volume Theory are estimated by fitting to the experimental MRI results and the effective diffusion coefficient is given as a function of the product water content.

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Keywords: MRI; convective drying; moisture profiles

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### 1. Introduction

Drying is a main technology for food preservation. The low moisture content and water activity allow safe storage of food products over an extended period of time. Due to the heat load and dehydration, quality attributes of food products change during drying; for example structure, colour, healthy components and nutritional value. The changes in quality attributes depend on the local, time varying

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moisture content and temperatures in the product rather than the average moisture content. As a result, the internal moisture distribution and moisture diffusion are important in studying the influence of drying on quality attributes of the product.

Drying of food particles process is controlled by diffusion and is often described by Fick's law. However, food products behave more complex during drying. The product goes from a rubbery state to a glassy state, and the changes in water mobility, resulting in different diffusion properties, may not be ignored. To meet the changes in diffusivity, the free volume theory is applied to predict moisture transport during drying<sup>[1]</sup>. This theory is based on the physical properties of foods, and takes the mobility of the water molecules into account. Moreover, the free volume theory can be applied in porous media, like vegetables (for example the floret of broccoli).

Moisture transport and distribution in products are key factors for the development of the product quality. To control the quality development, knowledge of the moisture content as a function of place and time in the product is required. For that purpose spatially distributed moisture profiles can be measured with destructive methods by taking slices from the product or non-destructive methods such as  $\gamma$ -ray densitometry. Drawbacks of these methods are the requirements on the size of the sample, a limited resolution or they can only be applied in a one dimensional direction<sup>[2,3,4]</sup>.

As an alternative, magnetic resonance imaging (MRI) is a powerful method to study complex materials as food products. The pioneer technique allows the imaging of the products' interior non-destructively. With continuous and controlled drying conditions, moisture transport during drying can be recorded. MRI has been applied in food applications to study the rehydration of extruded pasta<sup>[5]</sup>, drying of potatoes<sup>[6]</sup>, drying of apple slabs<sup>[2]</sup>, and drying of food gels<sup>[7]</sup>.

In this work moisture profiles in broccoli during drying are obtained from MRI imaging data. Experimental results of the moisture profiles and the effective diffusion coefficients are compared with simulation of the Free Volume theory and key parameters are tuned.

## 2. Theory and Modeling

Fick's second law for diffusion controlled particle drying, is given as:

$$\frac{\partial W}{\partial t} = \frac{\partial}{\partial r} \left( D_{eff} \frac{\partial W}{\partial r} \right) \quad (1)$$

with  $D_{eff}$  is the effective diffusion coefficient ( $m^2 \cdot s^{-1}$ ),  $W$  the moisture content ( $kg_{water} \cdot kg_{dry\ matter}^{-1}$ ),  $r$  the position (m) and  $t$  the time (s).

According the Maxwell-Eucken relationship, the diffusion coefficient for water in porous products is a combination of the diffusion coefficient of water in the continuous phase ( $D_c$ ; product) and in the dispersed phase ( $D_d$ ; air):

$$D_{eff} = D_c \left( \frac{D_d + 2D_c + 2(1 - \varepsilon)[(D_d - D_c)]}{D_d + 2D_c - (1 - \varepsilon)[(D_d - D_c)]} \right) \quad (2)$$

with  $D_d$  ( $m^2 \cdot s^{-1}$ ) is the water diffusion coefficient in air which is given by Olek (2003)<sup>[15]</sup>:

$$D_d = 23 \times 10^{-6} \frac{98100}{P} \left( \frac{T}{273.15} \right)^{1.75} \quad (3)$$

where  $P$  is the pressure (Pa), and  $T$  the temperature (K).

$D_c$  ( $\text{m}^2.\text{s}^{-1}$ ) is the water diffusion coefficient in the solid matrix, and follows from the Darken relation<sup>[8]</sup>:

$$D_c = Q(\phi D_w + (1 - \phi) D_s) \quad (4)$$

$$Q = 1 - 2\chi\phi(1 - \phi) \quad (5)$$

$D_w$  ( $\text{m}^2.\text{s}^{-1}$ ) is the self-diffusion coefficient of water molecules,  $D_s$  ( $\text{m}^2.\text{s}^{-1}$ ) the self-diffusion coefficient of solid particles,  $\phi$ (-) the volume fraction of the solid phase,  $Q$ (-) the thermodynamic factor, and  $\chi$  (-) the interaction parameter

The self-diffusion coefficient of water molecules  $D_w$  ( $\text{m}^2.\text{s}^{-1}$ ), is given by the Free Volume theory:

$$\ln\left(\frac{D_w}{D_0}\right) = \frac{\Delta E}{RT} - \frac{\gamma_1 V_1^* + \xi \gamma_2 V_2^*}{\gamma_1 \left(\frac{K_{21}}{\gamma}\right)(K_{21} - T_{g1} + T) + \gamma_2 \left(\frac{K_{12}}{\gamma}\right)(K_{22} - T_{g2} + T)} \quad (6)$$

The free volume parameters for water and product are given in Table 1:

Table 1. Free volume parameters

Water properties	Value	Product properties	Value
$V_1^*$ ( $\text{ml.g}^{-1}$ )	0.91	$V_2^*$ ( $\text{ml.g}^{-1}$ )	0.59
$T_{g1}$ (K)	136	$T_{g2}$ (K)	360
$D_0$ ( $\text{m}^2.\text{s}^{-1}$ )	$1.39 \times 10^{-7}$	$K_{22}$ (K)	69.21
$\Delta E$ ( $\text{J.mol}^{-1}$ )	$1.98 \times 10^3$	$k$ ( $\text{J.K}^{-1}$ )	$1.38 \times 10^{-23}$
$K_{21}$ (K)	-19.73	$a$ (m)	$1 \times 10^9$
$K_{11}/\gamma$ ( $\text{m.L.g}^{-1}.\text{K}^{-1}$ )	$1.945 \times 10^{-3}$		

$D_s$  is the self-diffusion coefficient of solid particles, which follows from the Stokes-Einstein theory<sup>[9]</sup>:

$$D_s = \frac{kT}{6\pi a \eta_{eff}} \quad (7)$$

To solve equation 1, symmetry in the product is assumed, at the centre of the product there is no mass transfer and at the surface where  $r=R$ , the boundary condition for mass transfer is:

$$k_c(C_{surface} - C_{air}) = D_{eff} \rho_p \frac{\partial W_{r=R}}{\partial r} \quad (8)$$

$$k_c(C_{surface} - C_{air}) = D_{eff} \rho_p \frac{\partial W_{r=R}}{\partial r} \quad (9)$$

With  $k_c$  the mass transfer coefficient ( $\text{m.s}^{-1}$ ),  $C_{surface}$  and  $C_{air}$  are respectively the vapour concentration at the product surface and air ( $\text{kg.m}^{-3}$ ) and  $\rho_p$  the product density ( $\text{kg.m}^{-3}$ )

### 3. Materials and Methods

#### 3.1. Materials

Broccoli was bought from a supermarket. For each measurement, a piece of floret was taken from the stalk. The size of this part was about 0.01 m in height, 0.01 m in radius, the floret had a stalk part of 0.005 m in height and 0.005 m in radius.

#### 3.2. 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%.

#### 3.3. MRI imaging

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.

#### 3.4. MR Imaging

3D images were obtained using a Turbo Spin Echo (TSE) MRI sequence<sup>[10]</sup>, 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 artefacts, so the turbo factor was 8. Two acquisitions were averaged to improve image quality. The Field-Of-View (FOV) was 35×35×35 mm<sup>3</sup> with a matrix size of 64×64×64 resulting in a spatial resolution of 0.55×0.55×0.55 μm<sup>3</sup>. The interval time between measurements was 34 minutes.

T2 mapping was done using a Multi Spin Echo (MSE) imaging sequence<sup>[11]</sup>, 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 mm<sup>2</sup> with a matrix size of 64×64 resulting in an in-plane resolution of 0.55×0.55 μm<sup>2</sup>. The slice thickness was 3 mm. The interval time between measurements was 34 minutes.

#### 3.5. Numerical methods and data analysis

MRI-measurement data handling and analysis was performed with home-built software written in IDL (RSI, Boulder, CO). To solve Equation 1-8, the structure of broccoli is configured in COMSOL and the equations are defined for each part of the structure. Parameter estimation was done with Matlab in combination with COMSOL.

### 4. Results and Discussion

#### 4.1. Moisture profiles from MRI imaging

Figure 1 gives 3D image measurements for the cross section at the centre of the broccoli sample. The intensity of the colour indicates the moisture content distribution within the sample; the brighter the

colour, the higher local moisture content. The outer surface of the floret has a dark colour, whereas the stalk has a bright colour, which corresponds to the faster drying at the surface and slower drying in the centre. Shrinkage also has been recorded simultaneously. After some time, the signal for the surface is too weak to interpret progress of drying by visual observation. In the high concentration range, the signal intensity is proportional to the moisture content, but in the dry stage, the intensity values may deviate from a linear relationship<sup>[2,12,13]</sup>.

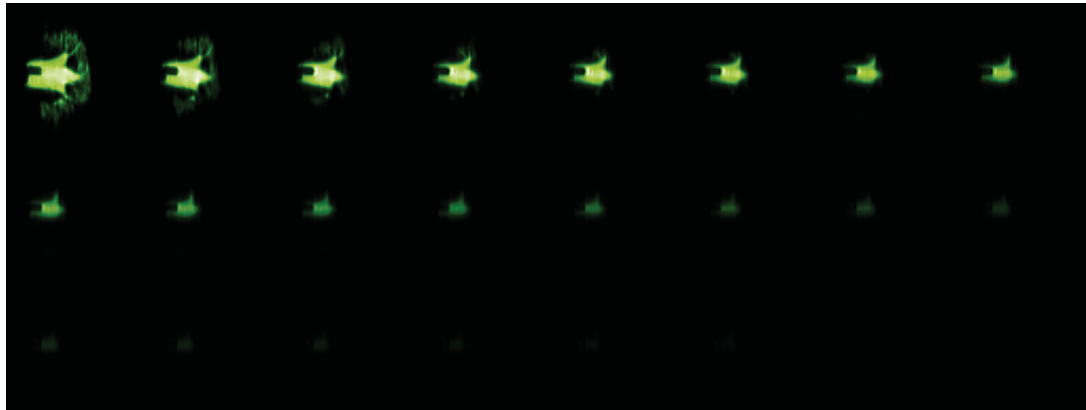


Fig. 1. Series of MRI intensity of the middle slice in time. Progress during drying is given in the pictures at the succeeding rows. The interval time between the samples given in this figure is 68 minutes

#### 4.2. Moisture diffusivities

Major advantage of the Free Volume theory is that moisture diffusion is predicted from general physical properties. In previous work the physical properties of simple sugars like sucrose were used to predict  $Deff$  by the Free Volume theory. However, this assumption may result in a not fully accurate result. In equation 6 universal constants and water properties are used, but parameters as  $\zeta$  (the ratio between molar volume of solvent versus solute, can be up to 0.9),  $V_2^*$  (the critical volume of a component, up to 0.9) and  $\epsilon$  (the porosity, which may range between 0.6-0.7) can be tuned towards the experimental data.

To convert the intensity to moisture content either an exponential relationship or a linear relationship can be used[2,6,14]. Here a linear relationship is applied, while keeping in mind that the minimum detectable moisture content equals to 0.3 kg water/kgdry matter[6]. The average moisture content in the cross section as shown in the images was used for data fitting on a spatial model simulated in COMSOL[1]. The dimensions and form used in the COMSOL model were close to the sample used in MRI experiments.

Average moisture content from simulations were compared with the average moisture content derived from the MRI signal intensity. The parameters  $\zeta$ ,  $V_2^*$ ,  $\epsilon$  and  $kc$  were obtained by minimizing the error between model and experimental results. Results are given in Figure 2. The obtained parameter values were  $\zeta = 0.9$ ,  $V_2^* = 0.9$ ,  $\epsilon = 0.6$ , the mass transfer coefficient  $kc$  was 0.023 at 50°C and 0.013 at 30°C. The standard error for the measurements at 50°C was 0.284 and at 30°C 0.272.  $R^2$  was respectively 0.982 and 0.989.

Figure 3, shows the resulting effective diffusion coefficient as a function of moisture content. The graph shows that the effective diffusion coefficient has a maximum value and decreases for the lower moisture contents. Here the product approaches the glass transition temperature which results in a lower mobility of the water molecules.

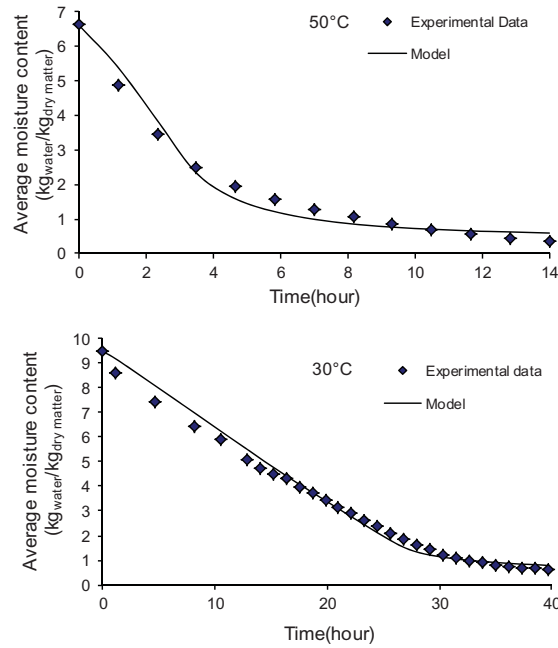


Fig. 2. Measured and estimated drying profiles: Top: at 50°C, bottom at 30°C

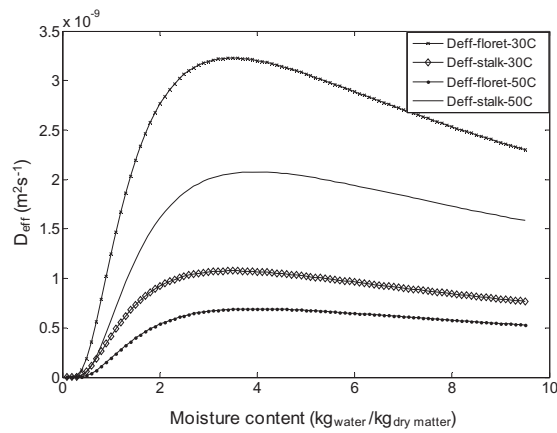


Fig. 3. Effective diffusion coefficients for broccoli florets and broccoli stalks at 30°C and 50°C

### 5. Conclusions

MRI imaging is successfully applied to monitor the moisture content and its spatial distribution in broccoli during drying. Moisture is proportionally to the intensity signal, but in the region of low moisture contents the resolution of the equipment limits the interpretation of the signal.

The MRI imaging results in a cross section allowed the estimation of the effective diffusion coefficient from the Free Volume theory. The results show that the effective diffusion coefficient varies during drying, which is a consequence of the changed mobility of water in the product.

MRI imaging makes it possible to study the internal properties of food products during processing. With the resulting information on the effective diffusion coefficients, moisture profiles during drying can be calculated which are essential to find processing conditions that retain heat sensitive healthy and nutritional components. Further process optimization work can be done based on this work as well.

## Acknowledgment

This work is supported by the Energy Research Program EOS (EOS LT07043) of the Dutch Ministry of Economics.

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Presented at ICEF11 (May 22-26, 2011 – Athens, Greece) as paper EPF360.