



## Effect of pulsed electric field treatment on water distribution of freeze-dried apple tissue evaluated with DSC and TD-NMR techniques



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### ABSTRACT

This work aimed to study pulsed electric fields (PEF) effect on the water distribution of freeze-dried apple. Apple (var. Cripps Pink) was treated in 15% trehalose and 1% ascorbic acid solution ( $388 \mu\text{S}/\text{cm}$ ) at 3 Hz and various electric field strengths 0.3; 0.6; 0.9 and  $1.2 \text{ kV cm}^{-1}$  for 5, 10 or 15 pulses. The samples were frozen at  $-45^\circ\text{C}$  and freeze-dried. The analyses were performed after rehydration. Differential Scanning Calorimetry (DSC) and Nuclear Magnetic Resonance in the domain of time (TD-NMR) were performed to assess thermal properties of freezable water and water distribution in apple tissue, respectively. PEF changed the integrity and continuity of the cell structure shown by the water redistribution between different compartments. The water in vacuoles and extracellular spaces had higher TD-NMR relaxation times as water molecules can diffuse in larger volumes before relaxing, even if the overall solutes concentration in the tissue increases.

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### 1. Introduction

Freeze-drying is widely used to dry fruit and vegetables. This technique is based on dehydration by sublimation of previously frozen tissues. It offers advantages such as increasing rehydration rate, maintaining the shape and the volume of plant tissues and increasing microbial stability (Ratti, 2001). Due to the formation of ice during the freezing process, the tissue collapse after the rehydration process was reported (Lewicki & Wiczowska, 2007). Since the freeze-dried products are usually consumed after rehydration, understanding the mechanisms of mass transfer (water and soluble solids) occurring during rehydration is of the key importance (Mastrocola, Dalla Rosa, & Massini, 1997). In order to improve qualitative and nutritional characteristics and to reduce the time of freeze-drying process, the application of several pre-treatments before freeze-drying has been studied as for example blanching (Doymaz, 2010), osmotic dehydration (Ciuurzyńska & Lenart, 2010) and infrared heating (Pan, Shih, McHugh, & Hirschberg, 2008).

In the last years the use of pulsed electric fields (PEF) technology for food processing has been studied. PEF treatment leads to electroporation of the cell membrane by applying an external electric field to the cellular tissue (Zimmermann, 1996; Zimmermann, Pilwat, & Riemann, 1974). Electroporation of the cell membranes could promote reversible or irreversible pore formation and cell disintegration, depending on both the intensity of the electric field strength applied and the characteristics of the raw materials (Angersbach, Heinz, & Knorr, 2000; Dymek, Dejmek, & Gómez, 2014; Puc et al., 2004; Toepfl, Siemer, & Heinz, 2014). Cell disintegration can be measured based on changes in the conductivity (Ben Ammar, Lanoisellé, Lebovka, Van Hecke, & Vorobiev, 2011; De Vito, Ferrari, Lebovka, Shynkaryk, & Vorobiev, 2008). PEF processing offers several advantages i.e. to improve extraction process (Barba, Galanakis, Esteve, Frigola, & Vorobiev, 2015; Luengo, Álvarez, & Raso, 2013), to enhance mass transport phenomena (Donsi, Ferrari, & Pataro, 2010; Vorobiev & Lebovka, 2010; Wiktor et al., 2013), and to inactivate enzymes (Elez-Martínez, Suárez-Recio, & Martín-Belloso, 2007) and microorganisms (Saldaña et al., 2009; Timmermans et al., 2014).

PEF treatment prior to drying process seems to have an effect on rehydration capacity of the resulting dried samples. Some authors reported an increase in rehydration rate (Eshtiagi, Stute, & Knorr, 1994), but

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some authors reported an inverse phenomenon, i.e. a decrease in rehydration rate (Ade-Omowaye, Angersbach, Taiwo, & Knorr, 2001; Taiwo, Angersbach, & Knorr, 2002) or no change in the constant rate of rehydration in both control and PEF treated samples (Gachovska, Simpson, Ngadi, & Raghavan, 2009). So far, the aforementioned conflicting phenomena are not well understood and they require further investigation using advanced methodology to investigate the water distribution and movement in heterogeneous matrix such as plant materials after PEF pretreatment.

Foods are proton-rich due to the presence of water, fat, carbohydrates or proteins.  $^1\text{H}$  Nuclear Magnetic Resonance ( $^1\text{H}$  NMR) in the time domain (TD-NMR) is a powerful non-invasive and non-destructive technique already commonly used to investigate the composition and internal structure of foods. TD-NMR can also monitor the compositional and structural modifications when foods undergo natural or artificial processes. Hence this technique can be used to investigate internal variations in the water content, as well as changes in the water interaction with cellular tissues (Butz, Hofmann, & Tauscher, 2005). During TD-NMR measurement, these variations are examined by studying the relaxation times parameters, i.e.  $T_1$  (spin-lattice or longitudinal relaxation) and  $T_2$  (spin-spin or transverse relaxation). In general,  $T_1$  and  $T_2$  are complex distributions of relaxation times and are commonly used to monitor the structural changes in foods during processing and storage (Marcone et al., 2013).

Recently TD-NMR has been employed to investigate the cell walls and membrane disruption and water mobility in the plant tissue subjected to PEF treatment (Aguiló-Aguayo et al., 2014; Ersus, Oztop, McCarthy, & Barrett, 2010). Ersus et al. (2010) were able to follow the changes of  $T_2$  relaxation times from two different cell compartments namely cytoplasm and vacuoles. They showed that the  $T_2$  values were strongly correlated with ion leakage from the onion tissue. Aguiló-Aguayo et al. (2014) investigated the water distribution in carrot slices after PEF treatment, showing the alteration of cell membrane and vacuoles which caused changes in  $T_2$  as a consequence of water redistribution between cellular structures. TD-NMR technique has been successfully used to explore the effect of osmotic dehydration on water mobility in terms of signal intensity and the  $T_2$  relaxation of protons separately for vacuoles, cytoplasm/extracellular spaces and cell wall of different fruits (Cheng, Zhang, Adhikari, & Islam, 2014; Dalla Rosa et al., 2011; Marigheto, Venturi, & Hills, 2008; Panarese et al., 2012; Santagapita et al., 2013; Tylewicz et al., 2011). In addition, Magnetic Resonance Imaging can be used for non-destructive evaluation of the internal structure of different food systems (Bortolotti et al., 2010; Defraeye et al., 2013) on the basis of gas, water and solutes exchange. In general, most studies use  $T_2$  as a parameter because it is easily measured however  $T_1$  is a robust parameter that can well describe foods properties and structural modifications.

The aim of this work was to investigate the effects induced by PEF pre-treatment at different electric field strengths on the water distribution in freeze-dried apple tissue using both DSC and TD-NMR. In this work, TD-NMR  $T_1$  analysis was applied on rehydrated apple tissue to detect variations in the water state of the cellular tissue treated with PEF processing.  $T_2$  analysis was additionally performed to evaluate potential water diffusion effects. To investigate the water–solid exchange followed by different food processing, Differential Scanning Calorimetry (DSC) was used. Different authors investigated the changes in freezable water content by DSC technique during fruit ripening (Goñi, Muñoz, Ruiz-Cabello, Escribano, & Merodio, 2007), osmotic dehydration (Cheng et al., 2014; Tylewicz et al., 2011) and freeze-drying (Zhao, Liu, Wen, Xiao, & Ni, 2015). During rehydration of freeze-dried products several changes take place i.e. water transfer from the liquid phase, where the sample is soaked, into the food and transfer of soluble solids from the food into the liquid phase. To the best of our knowledge so far there is no information about the consequences of the PEF application prior to freeze-drying on the water state of the cellular plant tissue.

## 2. Material and methods

### 2.1. Raw material, handling, and storage

Medium-size apples var. Cripps Pink ( $15 \pm 1$  °Bx) were purchased from the local market in Quakenbrueck (Germany). The apples were stored at  $2 \pm 1$  °C at high relative humidity protected from light until use.

### 2.2. Sample preparation

The apples were manually washed, peeled and cut into half with a sharp scalpel. Two cylindrical samples with a diameter of  $d = 20$  mm, height of  $h = 20$  mm and weight of about  $m = 2.6$  g were cut from the inner part of the parenchyma tissue of each apple half. Immediately after cutting, the samples were weighted and placed in the PEF chamber. The treatment was performed in a solution mixture of 15% trehalose and 1% ascorbic acid having the conductivity of  $388 \mu\text{S}/\text{cm}$ .

### 2.3. Pulsed electric field (PEF) treatment

The PEF treatment was conducted using batch 5 kW generator constructed by DIL (Germany), with a voltage output range of 8–19 kV. The treatment chamber was composed of 2 stainless steel parallel electrodes. The distance between the electrodes was 8 cm with the total area of  $170 \text{ cm}^2$ . The form of the chamber was rectangular prism (length: 17 cm, height: 10 cm; width: 8 cm), where the bottom of the chamber and the side holding the two electrodes were made of polyether ether ketone (PEEK) materials as insulator.

One of the electrode was connected with high voltage current and the other was grounded.

To reach desired electric field strength, output voltage was adjusted according to the size of treatment chamber used (distance between the electrodes) and calculated using Eq. (1):

$$E = \frac{U}{d} \quad (1)$$

where  $E$  is electric field strength ( $\text{V cm}^{-1}$ ),  $U$  is voltage (V) and  $d$  is electrode distance (cm).

Twelve cylindrical samples (total weight of 70 g) were used for each treatment. The treatment chamber was filled with trehalose/ascorbic acid solution (total weight of 650 g). The voltage output was set to achieve 0.3; 0.6; 0.9 and  $1.2 \text{ kV cm}^{-1}$  in a treatment chamber and the number of pulses (with a monopolar exponential decaying pulse shape) used for the treatments was 5, 10 and 15. The pulse width was set to  $60 \mu\text{s}$  and the frequency was fixed to 3 Hz. The parameters used in this study were selected based on the previous study of Ersus et al. (2010), as summarised in Table 1. Total energy input ( $W_{\text{whole}}$ , kJ/kg) was calculated according to Eq. (2).

$$W_{\text{whole}} = \frac{0.5 \cdot U^2 \cdot c}{V \cdot \rho} \cdot \eta \quad (2)$$

where  $U$  is voltage applied (V);  $c$  – capacity of the construction (F);  $V$  – volume of treatment chamber ( $\text{m}^3$ );  $\rho$  – product density ( $\text{kg m}^{-3}$ ) and  $\eta$  – number of pulses used for treating the product. All the treatments were performed at room temperature.

After PEF treatment, the samples were removed from the solution, placed on blotting paper (to remove the surface water) and weighted. Afterwards the untreated and PEF-treated samples were cut lengthways into half, frozen at  $-45$  °C and freeze-dried.

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