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Time domain nuclear magnetic resonance to monitor mass transfer mechanisms in apple tissue promoted by osmotic dehydration combined with pulsed electric fields

Nicolò Dellarosa [a,*], Luigi Ragni [a,b], Luca Laghi [a,b], Urszula Tylewicz [a], Pietro Rocculi [a,b], Marco Dalla Rosa [a,b]

[a] Department of Agricultural and Food Sciences, University of Bologna, Cesena, Italy
[b] Interdepartmental Centre for Agri-Food Industrial Research, University of Bologna, Cesena, Italy

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ABSTRACT

Pulsed electric field (PEF) technology is gaining momentum as a pre-treatment to enhance mass transfer of vegetable tissues obtained by further processing. In this study PEF pre-treatment increased osmotic dehydration (OD) effectiveness, in terms of water loss and solid gain in apples, as a function of electric field strength and number of pulses. Mass transfer was particularly high when average electric fields of 250 and 400 V cm^{-1} were applied. Time domain nuclear magnetic resonance (TD-NMR), with the use of a contrast agent, clarified structural changes that drive mass transfer. Treatments at 100 V cm^{-1} redistributed water between vacuole, cytoplasm and extracellular space, while at 250 and 400 V cm^{-1} the membrane breakages caused the loss of cellular compartmentalization. Two non-destructive and fast acquirable parameters, the longest measured relaxation time (T_2) and water self diffusion coefficient (D_w), allowed the separate and accurate observation of PEF treatment and osmotic dehydration effects.

Industrial relevance: The developed non-destructive method, here described, allows the measure of the effects of PEF treatment on apple tissue which can be exploited to have reliable control of the process within minutes. Since mass transfer parameters depend on subcellular water redistribution, the present work provides a tool to boost the development and optimization of agri-food processes on fresh vegetable tissues.

1. Introduction

Pulsed electric field (PEF) is an innovative non-thermal technology which delivers short pulses to food products, placed between two electrodes, generating electric fields, which usually span from 0.1 to 5 kV cm^{-1}. When coupled to extraction techniques, its application leads to an enhancement of mass transfer phenomena, which can be exploited to increase extraction yields from vegetable tissues (Donsì, Ferrari, & Pataro, 2010). In addition, its effectiveness has been demonstrated by combining PEF together with osmotic dehydration (Ade-Omowaye, Angersbach, Taiwo, & Knorr, 2001; Amami, Vorobiev, & Kechaou, 2006; Wiktor, Śledź, Nowacka, Chudoba, & Witrowa-Rajchert, 2014), air drying (Ade-Omowaye, Rastogi, Angersbach, & Knorr, 2003; Wiktor et al., 2013), compression (Bazhal, Lebovka, & Vorobiev, 2001) and thermal treatments (Lebovka, Praporscic, Ghnimi, & Vorobiev, 2005; Parniakov, Lebovka, Bals, & Vorobiev, 2015).

The application of PEF on vegetable tissue acts on the membrane permeability, inducing electroporation of cells (Teissie, Eynard, Gabriel, & Rols, 1999). The mechanism of electroporation includes different steps: polarization of membranes, creation of pores, expansion of pore radii and resealing of pores (Donsì et al., 2010; Vorobiev & Lebovka, 2008). In addition to the type of fruit and vegetable tissue, the extent of electroporation, especially the resealing of pores, which can last from seconds to hours, depends on the applied electric field strength, duration, number and shape of pulses, and interval between pulses. It is of practical importance that the application of electric fields lower than 1 kV cm^{-1}, and a total treatment time in the order of milliseconds, do not significantly contribute to a temperature increase, which would alter membrane permeability caused by heat related damages (Lebovka, Bazhal, & Vorobiev, 2002) and the quality of the obtained products.

In mass transfer applications, PEF effects on vegetable tissues are generally evaluated by the extraction yields, or by the release of some target compounds (Soliva-Fortuny, Balasa, Knorr, & Martín-Belloso, 2009). The measurement of the apparent diffusion coefficient, often compared to untreated and totally destroyed samples, is another index of macroscopic changes. This method has the drawback of being indirect and invasive, leading to inconsistent results due to possible modification of the structure of the tissue (Vorobiev, Jemai, Bouzrara, Lebovka, & Bazhal, 2005). Alternatively, changes of color and texture are also controlled as a side effect of PEF treatment, being even desirable, for instance, when material softening is the objective of the study (Lebovka,

* Corresponding author.
E-mail address: nicolo.dellarosa@unibo.it (N. Dellarosa).

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Praporscic, & Vorobiev, 2004). Direct effects on membrane permeabilization can be qualitatively observed by staining of plant tissues followed by microscope visualization (Fincan & Dejmek, 2002). However, the most commonly applied method to measure cell disintegration is based on changes in electrophysical properties, i.e. the impedance, that gives information on the damage degree of a sample when compared to both an untreated and a totally destroyed sample (Angersbach, Heinz, & Knorr, 1999; Lebovka et al., 2002).

Time domain nuclear magnetic resonance (TD-NMR) is a fast, non-destructive analytical technique that allows to evaluate spatial features in vegetable cellular compartments by the indirect measurement of water distribution inside and outside cells. Recently, the measurement of transverse relaxation time (T_2) curves has been successfully applied to study the subcellular water redistribution upon osmotic dehydration, its combination with ultrasound in kiwifruit (Nowacka, Tylewicz, Laghi, Dalla Rosa, & Witrowa-Rajchert, 2014; Tylewicz et al., 2011) and the addition of calcium and ascorbic salts to the osmotic solution in apple tissue (Mauro et al., 2015). Furthermore, through the evaluation of the water self diffusion coefficient, an overview of water possibility to explore the surrounding space can be achieved. Santagapita et al. (2013) found that water loss and solid gain, during the osmotic treatment of kiwifruit, were in good agreement with the reduction of the water self diffusion coefficient.

The present work evaluated the effect of PEF on apple tissue as preliminary treatment to osmotic dehydration, at three different electric field strengths (100, 250 and 400 V cm^{-1}) and total number of pulses (20 and 60 train series). Besides the control of the mass transfer parameters water loss and solid gain, a subcellular level observation was applied by means of TD-NMR to understand, in-depth, the PEF-induced mechanisms that affect mass balances. Differently from previous works, the transverse relaxation time (T_2) of the osmotic solution was selectively dropped by the addition of a contrast agent. This eased the discrimination of three characteristic cellular compartments, namely vacuole, cytoplasm and extracellular space, respectively delimited by plasma membrane and tonoplast. Moreover, once the membrane permeability was altered due to electroporation, the contrast agent was a key element to observe the external solution diffusing through the inner compartments of apples. In addition, the average water self diffusion coefficient (D_w) of water contained in apple tissue was evaluated as a non-destructive control tool for the osmotic dehydration process.

2. Material and methods

2.1. Material

Apples (*Malus domestica*) of the Cripps Pink variety, also known by the brand name Pink Lady®, were purchased at a local market and stored at 2 ± 1 °C for no longer than a month, within which experiments were run. Average moisture and soluble solid contents were, respectively, 83.5 ± 0.5 g and 14.0 ± 0.5 g per 100 g of fresh product (g_{fw}). The apples were cut with a manual cork borer and cutter to obtain cylinders of 8 mm diameter and a length of 10 mm.

2.2. Pulsed electric field (PEF) treatment

Pulsed electric field (PEF) treatments were applied to apple cylinders using an in-house developed pulse generator equipment based on MOSFET technology and on capacitors as energy tank. The PEF generator provides monopolar pulses of near-rectangular shape at different voltages, adjustable repetition time between pulses and variable total treatment duration which lead to a variable number of delivered pulses. Treatments were run at 20 °C in a 30 × 20 × 20 mm (length × width × height) chamber equipped with two stainless steel electrodes (active contact surface = 20 × 20 mm^2) with a distance between them fixed at 30 mm. For each treatment 12 apple cylinders (approximately 5 g) were inserted into the chamber with the two circle sides parallel to the electrodes (Fig. 1). The chamber was filled up with tap water, with an electrical conductivity of 328 ± 4 μS cm^{-1} at 25 °C, with product-to-water ratio around 1:1 (v/v).Table 1 shows the experimented pulse series and the average applied electric field strengths in the chamber of trials conducted at fixed pulse width (100 ± 2 μS) and repetition time (10.0 ± 0.1 ms) with a voltage of 300 V, 750 V, and 1200 V to the electrodes. The current and voltage values were registered by using a digital oscilloscope (PicoScope 2204a, Pico Technology, UK) connected to a personal computer.

2.3. Osmotic dehydration (OD) and mass transfer control

Immediately after PEF application, the treated apple cylinders were removed from the PEF treatment solution and placed into 7 different beakers containing a continuously stirred 30% (w/w) sucrose osmotic solution, in a product-to-solution ratio of approximately 1:20 (w/w), to avoid changes in the concentration of the solution during the treatment. The rotational speed was experimentally determined to assure negligible resistance to mass transfer. Besides, control samples were prepared by directly placing the apple cylinders into the osmotic solution without PEF pre-treatment. Iron (III) chloride (Sigma-Aldrich — Steinheim, Germany) was employed as a contrast agent for NMR analysis and added to the osmotic solution to obtain a final concentration of 0.01 M. Samples were collected 0 (fresh control), 15, 30, 60 and 120 min after the immersion, blotted with absorbing paper, weighted and analyzed. The moisture content of 3 apple cylinders (weighing approximately 1.5 g) of fresh and treated samples was determined gravimetrically by drying at 70 °C until a constant weight was achieved, as recommended for fruit products by AOAC International (2002). In parallel, the same experimental plan (Table 1) was run by replacing the osmotic treatment with an isotonic solution, to gain insight of mass transfer phenomena caused by PEF only, without an external osmotic driven force.

Mass transfer was evaluated by calculating the mass balances, in terms of mass variation, water loss and solid gain. The total mass variation (ΔM) in relation to the initial mass during osmotic dehydration was calculated from experimental data according to Eq. (1):

$$\Delta M = \frac{(m - m_0)}{m_0} \quad (1)$$

where m = mass and m_0 = mass at initial time (t = 0).

Water loss (ΔM_w) and solid gain (ΔM_s) were calculated in relation to the initial mass according to Eqs. (2) and (3), respectively:

$$\Delta M_w = \frac{(w \cdot m - w_0 \cdot m_0)}{m_0} \quad (2)$$

$$\Delta M_s = \Delta M - \Delta M_w \quad (3)$$

where w = water content and w_0 = water content at initial time (t = 0).

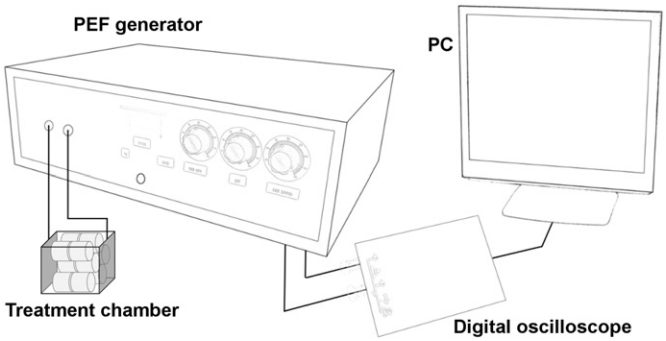

Fig. 1. Layout of the experimental setup.